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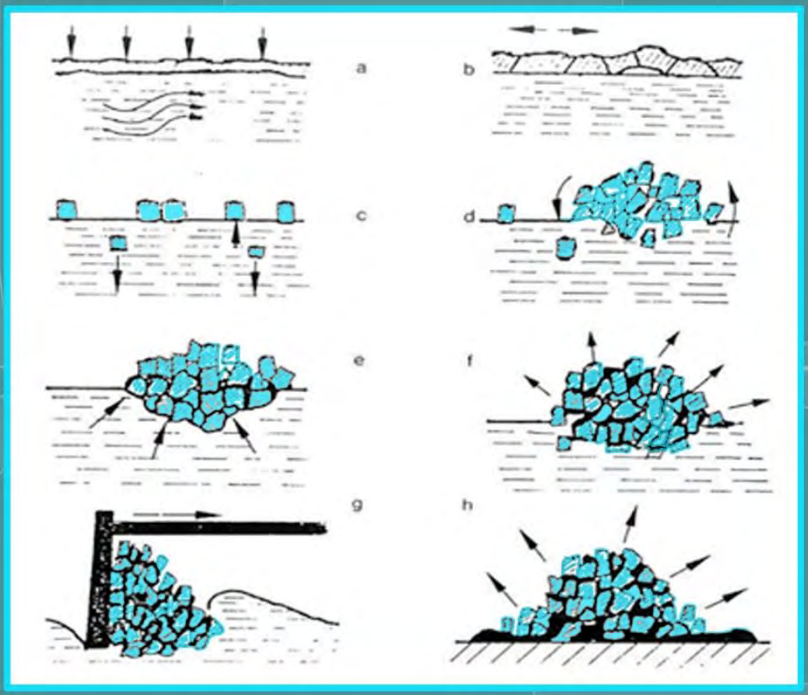
# Secondary Aluminum Processing Waste:

## Salt Cake Characterization and Reactivity




Office of Research and Development
National Risk Management Research Laboratory
Land Remediation and Pollution Control Division

## Notice

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# Foreword

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Cynthia Sonich-Mullin, Director
National Risk Management Research Laboratory

# Executive Summary

Aluminum is a ubiquitous, highly utilized non-ferrous metal, employed in a wide range of products. Unlike other metals, aluminum can be almost completely recycled into new products through a series of processes that re-melt aluminum to produce aluminum ingots at secondary aluminum processing (SAP) facilities. Solid residues generated by the aluminum melting processes are often referred to as aluminum dross, which forms on the surface of molten aluminum through contact with oxygen during melting, holding, refining, and transfer processes. Dross obtained from primary melting operations (also known as "*white dross*") consists primarily of aluminum oxide (with some oxides of other alloying elements, such as magnesium and silicon) and may contain from 15 to 70% recoverable metallic aluminum. Dross from secondary smelting operations (also known as "*black dross*") typically contains a mixture of aluminum/alloy oxides and slag (a partially amorphous solid by-product of the smelting process) and contains approximately 12 to 18% recoverable aluminum.

Commercial smelting of both white and black dross is often completed in a rotary salt furnace. The nonmetallic byproduct residue that results from such furnace slag is frequently termed "salt cake", and contains only 3 to 10% residual metallic aluminum. The formation of salt cake and the amount of salt cake formed depend on several factors such as type and quality of input material (e.g., aluminum scraps), operating conditions, and technology applied. Traditionally, salt cake from SAP facilities is disposed of into municipal solid waste landfills, with an annual disposal quantity of approximately one million tons in the United States (U.S.). Worldwide, the aluminum industry produces nearly five million tons of furnace waste (salt cake and baghouse dust) each year and this figure is tied to overall aluminum consumption. There is a need to treat and dispose of the waste from aluminum production as well as to reduce the generation and disposal of by-products from aluminum recycling in both the U.S. and in other countries around the world.

Given the potential concern associated with the reactivity of salt cake in a landfill environment, the U.S. EPA initiated a collaborative research effort with the Aluminum Association and the Environmental Research and Education Foundation to characterize salt cake waste material and investigate its reactivity under controlled conditions. This report presents the first systematic study on the characteristics and reactivity of salt cake derived from operating SAP facilities in the U.S.

The specific objectives of the study were to:

- Identify and characterize the metal constituents in salt cake
- Investigate the leaching behavior of metals from salt cake following its reaction with water
- Identify the dominant crystalline mineral phases in salt cake
- Evaluate factors that impact the reactivity of salt cake
- Evaluate salt cake reactivity with water

Thirty-nine salt cake samples were collected from 10 SAP facilities across the U.S. The facilities were identified by the Aluminum Association to cover a wide range of processes. Results suggest that while the percent metal leached from salt cake was relatively low, the leachable metal content may still pose a contamination concern and potential human and ecological exposure if uncontrollably released to the environment. As a result, salt cake should always be managed at facilities that utilize synthetic liner systems with leachate collection (the salt content of the leachate will increase the hydraulic conductivity of clay liners within a few years of installation). The mineral phase analysis showed that various species of aluminum are present in the salt cake samples with a large degree of variability. The relative abundance of various aluminum species was evaluated but it is noted that the method used is a semi-quantitative method and as a result there is a limitation for the data use. The analysis only showed a few aluminum species present in salt cake which does not exclude the presence of other crystalline species, especially in light of the variability observed in the samples.

Results presented in this document are of particular importance when trying to understand concerns associated with the disposal of salt cake in municipal solid waste (MSW) landfills. From the end-of-life management perspective, data presented here suggest that salt cake should not be size reduced before disposal. Also, care should be taken not to size reduce it after disposal by compacting it with heavy bulldozers. The MSW decomposition process is exothermic and as a result MSW landfill temperatures are typically greater than 37°C and may reach 50°C or greater. The elevated temperatures present in most MSW landfills can be conducive and could initiate salt cake reactions that otherwise may

not take place. Furthermore, elevated temperatures can increase the rate of the salt cake reaction which further propagates the salt cake reaction. Even outside of MSW landfills, once a salt cake reaction starts, it may self-propagate and increase the rate at which the reaction occurs. While the heat generation potential per gram of salt cake sample is a sample specific parameter, the results presented herein suggest that higher environmental temperatures tend to intensify the salt cake reaction. This means that with increasing temperature the rate of salt cake reaction will also increase. As stated earlier, this is of particular significance for the disposal of salt cake. Depending on their design and capacity, MSW landfills tend to be well insulated and operate at elevated temperatures. When these conditions exist, the reaction of salt cake with liquids tends to become more problematic since the elevated temperatures in MSW landfills may initiate the salt cake reaction and the insulating properties of the MSW may further intensify the reaction. Furthermore, the propagation effect that has been stated earlier can also magnify the impact of the reaction.

At elevated temperatures encountered in MSW landfills, elevated levels of hydrogen gas may be generated as a result of salt cake reactivity and will be of concern. Hydrogen is an explosive gas and can potentially cause fires. Hydrogen is even of greater concern if generated in MSW landfills. Apart from being a fire hazard when mixed with methane, landfill gas collection systems are not designed to handle hydrogen since MSW landfill gas generally consists of methane and carbon dioxide at ~ 50/50 ratio. Like hydrogen, methane is an explosive gas and care must be taken if salt cake reacts with liquids. But unlike hydrogen, in an MSW landfill, the gas collection system is designed to handle methane gas which is a greenhouse gas generated during the anaerobic decomposition of MSW. Since the full combustion of methane needs more oxygen than that of hydrogen, this unstable composition of landfill gas can make trouble for its application and management. From a human health perspective, the concentration of ammonia gas produced from the salt cake reaction was relatively high and can potentially be of concern. However, the level of ammonium resulting from the salt cake reaction is significantly lower compared to the levels found in landfills and as a result, the ammonia generated from salt cake might not have a detrimental impact on the performance of MSW landfills.

The samples of salt cake analyzed in this study represented a range of processes that produce these materials at SAP facilities. Testing was conducted under a controlled set of laboratory conditions and represent a broad yet detailed characterization of different salt cakes including, resultant products following reaction with water. The data generated during this effort will help to support future, further characterization efforts which may ultimately be used to better predict the ultimate impacts of disposing salt cake from SAP facilities in landfills. This may possibly help identify operational techniques and procedures to minimize these impacts.

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## Acronyms and Abbreviations

$\Delta T$ = difference in temperature
$\Sigma\Delta T$ = sum of temperature change
AA = atomic absorption
AES = atomic emission spectroscopy
Al = aluminum
ANOVA = analysis of variance
ASTM =American Society for Testing and Materials
CHL = Center Hill Laboratory
DIW = deionized water
EC = electrical conductivity
ECD = electron capture detector
EPA = Environmental Protection Agency
EREF = Environmental Research and Education Foundation
GC = gas chromatograph
GFAA = graphite furnace atomic absorption
ICDD = International Centre for Diffraction Data
ICP = inductively coupled plasma
L/S = liquid to solid
MC = moisture content
MDL = method detection limit
MRL = method reporting limit
MSW = municipal solid waste
PDF = powder diffraction file
PVDF = polyvinylidene fluoride
RCRA = Resource Conservation and Recovery Act
SC = salt cake
SAP = secondary aluminum processing
TCD = thermal conductivity detector
TCLP = toxicity characteristics leaching procedure
UCL = upper confidence limit
U.S. = United States
VOA = volatile organic analyte
WHC = water holding capacity

# 1. INTRODUCTION

Aluminum is one of the most commonly used non-ferrous metals. Unlike other metals and recyclable commodities, aluminum can be almost completely recycled into new aluminum products [1-4]. Secondary aluminum processing (SAP) involves the recycling and re-melting of scrap aluminum to reproduce aluminum alloys, which can produce various types of waste, referred to as SAP wastes. The process of treating and disposing the waste from aluminum production as well as the presence of byproducts of the aluminum recycling process is becoming not only a problem in the U.S. [5-13], but globally [4, 14-20]. The main solid waste produced during aluminum processing in the U.S. is referred to as salt cake (SC), and the SC from SAP is the subject of this report.

Aluminum is utilized in a large number of products, both alone or as a component of an alloy. The total amount of aluminum production in the world was approximately 28 million tons (with over 8 million tons recycled from scrap) in 1990 and near 56 million tons (with close to 18 million tons recycled from scrap) in 2008. By 2020, aluminum metal demand is projected to increase to around 97 million tons (including approximately 31 million tons recycled from scrap) [4]. The total North American aluminum industry supply was 11.5 million tons in 2013, 41% of which came from recycled aluminum [21]. The used beverage aluminum container recycling rate was 50% (52 billion cans) in 2006, and increased to 66.7% (60.2 billion cans) by 2013 [22]. Almost 90% of automotive aluminum, including versatile aluminum alloys, is reclaimed and recycled [3]. But Americans still discarded about 2.9 million tons of aluminum in the municipal solid waste (MSW) stream after recycling and recovery in 2012, totaling 1.4 percent of total MSW discards [23]. Recycling scrap aluminum requires only 5% of the energy used to produce aluminum from raw materials (i.e., ore), and avoids approximately 95% of the emissions associated with producing new aluminum from ore [24-27]. The technologies used for SAP vary from one plant to another depending on the scrap type, oxide content, impurities present, and other factors [3, 27, 28]. In the U.S., one popular method of producing secondary aluminum is through the use of rotary furnaces with the addition of salt fluxes to improve recovery and reduce oxidation of the aluminum metal [29 ]. Salt flux is a mixture of sodium chloride (NaCl) and potassium chloride (KCl) with lower amounts of other compounds, such as calcium fluoride ($CaF_2$) [14, 30-33]. Salt flux melts at a slightly lower temperature than aluminum, and therefore coats the metal before it melts in the furnace and reduces aluminum metal loss through oxidation. Additives in salt flux also penetrate and break down previously-formed aluminum oxide layers and improve separation between aluminum metal and nonmetal material in the furnace [32-34].

Aluminum dross is a residue from aluminium melting activities. It is generated when impurities and oxidation layers are skimmed from the top of molten metal during the melting, holding, and casting operations. The formation of dross and the amount of dross formed depends on different factors, such as the type and quality of input material (e.g., aluminium scraps in secondary industry). Drosses obtained from primary melting operations (known as white drosses) consist primarily of aluminum oxide (with some oxides of other alloying elements such as magnesium and silicon) and may contain 15 to 70% recoverable metallic aluminum [3, 27, 29, 35, 36]. Drosses from secondary smelting operations (black drosses) typically contain a mixture of aluminum/alloy oxides and slag, and typically contain recoverable aluminum contents ranging from 12 to 18% [3, 27, 29, 35, 36]. Commercial smelting of both white and black dross is often done in a rotary salt furnace. The nonmetallic byproduct residue, which results from such dross smelting operations (SC or salt slag in Europe) contains 3 to 10% residual metallic aluminum [3, 27, 29, 35, 36] (Table 1-1).

**Table 1-1: Approximate composition of Aluminum by-products [36]**

| Material | % Metallic aluminum | % Aluminum Oxide | % Salt |
|---|---|---|---|
| White Dross | 15-80 | 20-85 | 0-1 |
| Black Dross | 7-35 | 30-50 | 30-50 |
| Salt Cake | 3-10 | 20-60 | 20-80 |

The formation of SC and the amount of SC formed depends on different factors such as type and quality of input material (e.g., aluminium scraps in secondary industry), operating conditions, type of technology, and furnace applied [3, 27, 29, 35, 36]. It is believed that its formation involves the following steps: (a) surface oxidation, (b) crushing of oxidic skin, (c) sinking and floating of oxide particles, (d) conglomeration of oxidic particles, (e) the filling of interspatial areas by metallic aluminum, (f) inside oxidation of dispersed aluminium, (g) skimming of dross from melt

bath surface, and (h) metallic aluminum drip off and follow-up oxidation of solids during dross cooling, suggested by Antrekowitsch [37] (Figure 1-1). Aluminum nitrides, carbides, and sulfides may also be present, as well as metal oxides derived from the molten alloy [3, 27, 35, 36, 38].

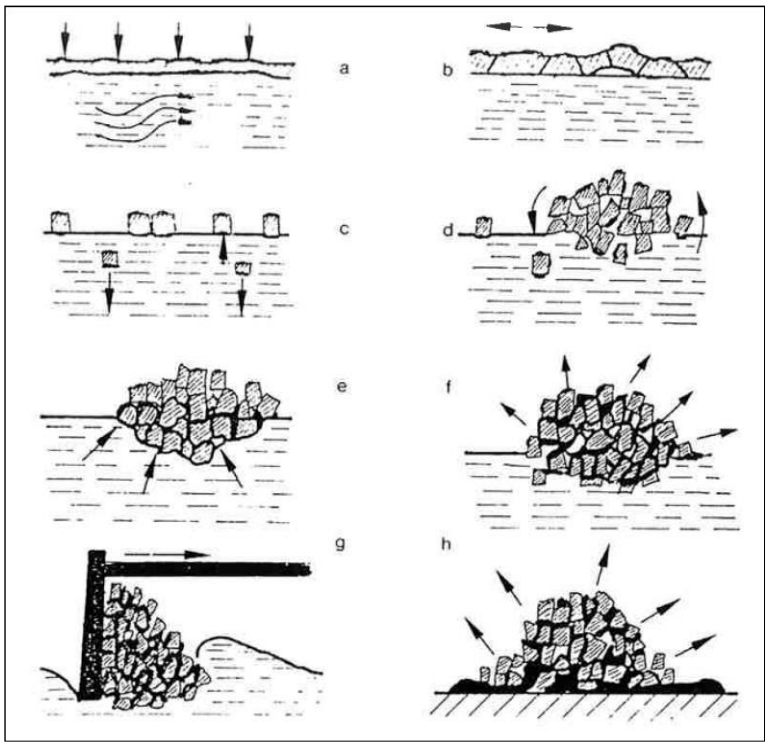



**Figure 1-1: The formation of salt cake [37]**

a) Surface oxidation
b) Crushing of oxidic skin
c) Sinking and floating of oxidic particles
d) Conglomeration of oxidic particles
e) The filling of interspatial areas by metallic aluminum
f) Inside oxidation of dispersed aluminum
g) Skimming of dross from melt bath surface
h) Metallic aluminum drip off and follow-up oxidation of solids during dross cooling.

Traditionally, SC has been disposed of in landfills. Almost one million tons of SC are placed in MSW landfills annually in the U.S. [39]. Worldwide, the aluminum industry produces nearly five million tons of furnace waste (e.g., SC and baghouse dust) each year [28], and this number continues to grow with the increase in aluminum consumption, especially from the SAP [40]. SC is recognized as a hazardous waste in European Union countries [41] because it is considered to be "highly flammable" (Category H3-A) and an "irritant" (Category H4**)** [42] . When SC comes in contact with water or damp air, highly flammable gases form. These gases can be explosive as well as act as irritants to skin and mucous membranes**.** Furthermore, the SC particles have been found to be harmful if inhaled or ingested **(**Category H5) [42]. SC is also in the category of substances that are capable, after disposal (landfill or other), of potentially yielding another substance (e.g., leachate) which can possess any of the characteristics associated with the solid SC or gaseous products (Category H13) [42]. Therefore, landfill disposal of SC can be costly and attempts in Europe to landfill this type of waste are dwindling due to the lack of landfill space [43].

Because of aluminum's amphoteric properties (meaning it dissolves in both acid and alkaline solutions, generating heat and gas), the disposal of large quantities of aluminum-containing SC can be problematic. The many forms of aluminum (i.e., metallic aluminum (Al), aluminum nitride (AlN), aluminum carbide ($Al_4C_3$), aluminium sulfide ($Al_2S_3$), and aluminum oxynitride ($Al_5O_6N$)) that may be contained in SC can undergo a wide range of potential

reactions after interacting with water during disposal. These reactions are listed in Equations 1-1 through 1-10:

$$\mathbf{2Al + 2OH^- + 6H_2O \rightarrow 2(Al(OH)_4)^- + 3H_{2(g)} \uparrow + heat} \quad (1\text{-}1)$$

Note that reaction 1-1 could only occur in alkaline solutions above a pH of 8. Metallic aluminum could also react under acidic solutions to produce complex aluminum-containing ions and hydrogen gas in the following manner:

$$\mathbf{2Al + 6H_3O^+ + 6H_2O \rightarrow 2(Al(OH)_6)^{3+} + 9H_{2(g)} \uparrow + heat} \quad (1\text{-}2)$$

The presence of aluminum nitride and carbide in the SC is due to the reactions of molten aluminum with the different gases during the melting process. These reactions include:

$$\mathbf{2Al + N_{2(g)} \rightarrow 2AlN} \quad (1\text{-}3)$$

$$\mathbf{6Al + 3CO_{2(g)} \rightarrow Al_4C_3 + Al_2O_3} \quad (1\text{-}4)$$

$$\mathbf{4Al + 3SO_{2(g)} \rightarrow Al_2S_3 + Al_2O_3} \quad (1\text{-}5)$$

The formation of aluminum nitride (AlN) in SC is a function of the melting duration, casting, and temperature. The influence of temperature on the AlN composition in SC has been described [44]. When SC is generated at a temperature of 700°C, the AlN content in SC is approximately 2% by weight. This concentration rises to around 18% when formation occurs at approximately 1,000°C. Aluminum nitride and its oxide in SC have often been documented in reviews and reports [3, 11, 16, 27, 36, 38, 45-49]. The formation of aluminum carbide is believed to occur at above 900°C [44], especially in the white dross form [36]. Aluminum carbide in SC has also been observed by some investigators [3, 16, 49-51].

The reaction of aluminum nitride is also pH-dependent. Under alkaline conditions, the reaction products are aluminum hydroxide and ammonium hydroxide (1-6):

$$\mathbf{AlN + 4H_2O \rightarrow Al(OH)_3 + NH_4OH} \quad (1\text{-}6)$$

Under acidic conditions, below a pH of 8, the reaction proceeds to produce ammonia gas:

$$\mathbf{AlN + 3H_2O \rightarrow Al(OH)_3 + NH_{3(g)} \uparrow} \quad (1\text{-}7)$$

Aluminum oxide nitride can also react with water to yield aluminum oxide and ammonia gas:

$$\mathbf{2Al_5O_6N + 3H_2O \rightarrow 5Al_2O_3 + 2NH_{3(g)} \uparrow} \quad (1\text{-}8)$$

Aluminum carbide can react with water to produce methane [52]:.

$$\mathbf{Al_4C_3 + 12H_2O \rightarrow 4Al(OH)_3 + 3CH_{4(g)} \uparrow} \quad (1\text{-}9)$$

Aluminum sulfide hydrolysis can also generate hydrogen sulfide [53]:

$$\mathbf{Al_2S_3 + 6H_2O \rightarrow 2Al(OH)_3 + 3H_2S_{(g)} \uparrow} \quad (1\text{-}10)$$

As outlined earlier, various forms of aluminum may be present in SC waste, which can react with water to generate flammable gases (e.g., hydrogen and methane). There is no current policy or guidance from the U.S. EPA for a specific test to determine if SC exhibits the reactivity characteristic for the purpose of hazardous waste classification. If there is a decision to test for reactivity, one type of testing in the U.S. that could be used as guidance for potential testing protocol is the Department of Transportation's "Hazardous When Wet" test (CFR 173.124-Class 4) [54]. Hazardous When Wet material (Division 4.3) means a material that, when it contacts water, is liable to become spontaneously flammable or to give off flammable or toxic gas at a rate greater than 1 L per kilogram of the material per hour, when tested in accordance with the United Nations Manual of Tests and Criteria at an ambient temperature (20°C) [54, 55]. Meanwhile, there is a requirement for generators to use "generator knowledge" in making the reactivity characteristic determination. It is noted that the U.S. EPA does not classify aluminum processing waste as ignitable, corrosive, reactive, or toxic (the four hazardous characteristic categories) at the present time. Therefore, U.S. EPA regulations do not exclude aluminum processing waste from disposal in MSW landfills.

While, secondary aluminum processing waste may not meet the U.S. Federal definition of a hazardous waste, reactions with leachate in MSW landfills have been documented and are of potential concern to engineered protective

components of the landfill (e.g., bottom liners that capture leachate that percolates through the waste). In MSW landfills, heat is generated as a result of biochemical processes and the decomposition of organic components in waste [56]. Temperature is considered to affect waste decomposition in two ways: short-term effects on reaction rates and long-term effects on the microbial population balance are expected [57]. In laboratory studies, optimum temperature ranges for the growth of mesophilic and thermophilic bacteria involved in waste decomposition were identified to range from 35 to 40°C and 50 to 60°C, respectively [58, 59]. There is no specific landfill design for the handling of SC in the U.S.; most of the generated SC is still currently disposed of in MSW landfills (Subtitle D municipal and industrial, non-hazardous waste), which are usually designed with liners to protect groundwater from contamination. The liners are expected to provide this protection for the entire period of time that a landfill may contain leachable chemicals, thus damage could result in a negative impact on the environment. The potential for groundwater contamination by a landfill may continue to be a concern for many decades after the landfill has ceased waste disposal operations. Current liner designs include a geomembrane and either a geosynthetic clay liner or a compacted clay liner. As long as the geomembrane component of the composite liner retains its structural integrity, this barrier successfully limits landfill leachate and gases from impacting groundwater and the surrounding environment. A geosynthetic clay or compacted clay layer incorporated into a MSW landfill composite liner system may also lose this integrity when exposed to a thermal gradient, which can induce desiccation and cracking of the clay layer. The potential for desiccation of the clay layer results from thermal gradients, which can arise between the liner and the cooler ground under the liner [60]. This becomes especially problematic if the geomembrane above the clay liner develops defects such as cracks or holes as it ages because the clay liner then acts as the primary barrier to leachate. Benchtop laboratory experiments have determined that the initial moisture content of the foundational soil below the clay liner is a critical factor affecting the potential for clay liner desiccation, with lower initial subsoil water content resulting in greater desiccation potential. In addition, the higher the temperature gradient a geosynthetic clay liner experiences, the greater the potential for desiccation induced cracking. As seen with the geomembrane, a temperature of 60ºC applied to the top of a test cell will create a temperature gradient that increases the risk of desiccation [61]. There is currently information available that suggests that engineered landfill components designed to protect the environment from contamination by MSW landfills can develop significant problems over time due to exothermic biological activity and exothermic reactions associated with other waste [62-64].

Some MSW landfills in the U.S. have reported operational challenges that may be attributable to reactions from SC disposal, including:

- High subsurface temperatures
- Elevated gas flow, and hydrogen and ammonia concentration
- Nuisance odors with potential hazardous gaseous emissions
- Increased leachate volume with potential heavy metal contaminants
- Subsurface oxidation events
- Outbreaks of leachate from side slopes
- High rates of landfill settlement/subsidence
- Reduction in landfill gas collection efficiency
- Impacts to groundwater quality

As an example, aluminum processing waste disposed in an unlined landfill by the Recycled Aluminum Metals Company (RAMCO) resulted in a host of issues. SC and a smaller amount of baghouse dust were placed in an unlined landfill near Dallesport, WA, between 1982 and 1989. A Site Hazard Assessment conducted by the Washington State Department of Ecology showed elevated concentrations of salt in the groundwater and borings beneath the landfill surface showed elevated temperatures and the presence of ammonia gas. Measurements indicated that the composition of the landfill included up to 29% aluminum, 8% sodium, 2.8% magnesium, 2.1% calcium, and 1.5% potassium, with lesser amounts of metals such as chromium, manganese, iron, copper, nickel, and zinc present. Groundwater impacts from nitrate, sodium, chloride, and total dissolved solids were also observed. Ultimately, the waste was removed from the site and transported to a landfill in compliance with the liner and monitoring requirements of Resource Conservation and Recovery Act subtitle D [6].

Another example is Brantley Landfill (Island, McLean County, Kentucky). Over a two-year period from 1978–1980, a total of 250,306 tons of SC fines were deposited in the landfill (previously the site of a coal strip mine). Site investigations found contamination in air, ground water, sediment, soil, and surface water that could potentially harm

people in the area. Contaminants of concern included chromium, copper, titanium, vanadium, aluminum, magnesium, and sodium, which react with water to form several gases, including ammonia, acetylene, methane, hydrogen, and hydrogen sulfide. A number of options were evaluated as remedial treatments, including the removal of the waste from the site. However, since the landfilled waste was 60 feet thick at its deepest point, this option would have required special equipment and excavation techniques. It was determined that the waste would remain at the fenced in, isolated location. A new landfill cap and an alkaline recharge trench were constructed, surface and groundwater were monitored, and leachate was collected, treated and disposed off-site. The total cost of the remedial action was projected to exceed $5,000,000, with an additional annual cost of $57,000 to $223,000 allotted for site monitoring [5].

SAP waste, placed in an MSW landfill, may result in an exothermic reaction upon contact with water, which can initiate incomplete combustion or pyrolysis of other waste materials [11, 65, 66]. Aside from the potential impacts on engineered systems relating to leachate collection, gas collection, and liner systems, there are potentially significant environmental and community health impacts associated with air emissions and surface water/groundwater releases from these reactions. For example, between 1993 and 2006, the Countywide Recycling and Disposal Facility in East Sparta, OH, accepted approximately 600,000 tons of aluminum processing waste, primarily aluminum dross and SC. In 1998, Countywide began re-circulating leachate, and in 2001, elevated temperatures were detected in gas wells in two cells of the landfill. Odor complaints were made by citizens in 2004, and so an odor suppression system was installed in 2005. By the end of 2005, hundreds of odor complaints were again being filed and landfill gas wells were showing elevated temperatures. In 2006, well temperatures were still increasing, leachate outbreaks were occurring, accelerated settlement was observed and odor problems increased [7]. Numerous actions were taken during this period to alleviate the problems. Leachate recirculation was halted; aluminum processing wastes were no longer accepted and site monitoring actions were increased. Despite these and other efforts, the Ohio EPA concluded that a fire was occurring in the landfill which involved the SAP wastes and the MSW. Although additional actions recommended or required by the Ohio EPA and the U.S. EPA were implemented, evidence of a continuing subsurface reaction involving the aluminum processing waste and water was observed in 2008, which may also have caused pyrolysis of landfilled MSW. Acetylene, hydrogen, and carbon monoxide were also detected at the site along with methane. Mixtures of these gases may cause an explosive atmosphere near the surface of the landfill [67]. In June 2009, a remedial strategy involving the removal of the problematic areas within the landfill excavation was completed, resulting in the extraction of approximately 400,000 cubic yards of waste. A liner was installed to cover the excavated area to reduce contact by moisture and atmospheric oxygen [7].

In light of the issues that have been documented regarding SC disposal at unauthorized landfills and at Subtitle D landfills, there is a need to more fully understand the conditions that lead to reaction of SC and the effect those reactions have on landfill operation and landfill engineered components. The U.S. EPA initiated a collaborative research effort with the Aluminum Association and the Environmental Research and Education Foundation (EREF) to examine safe management practices for SC waste. The U.S. EPA is seeking to better understand SC waste material, its potential risk, and strategies for safe management of municipal landfills. This research report presents a systematic study on the characteristics of SC and evaluation of SC reactivity when exposed to liquid water. The specific study objectives were as follows:

- Evaluate the physical and chemical properties of SC
- Evaluate the factors impacting SC reactivity
- Evaluate SC reactivity with liquids

# 2. METHODOLOGY

## 2.1 Sample Collection and Preparation

Thirty nine SC samples were collected from 10 secondary aluminum processing facilities (designated as B, C, D, E, F, H, J, L, M and N; Table 2-1). The facilities were identified in collaboration with the Aluminum Association and EREF to cover a wide range of SAP processes.

**Table 2-1: SAP waste (salt cake) facility and sample identification list**

| Sample # | Facility | Sample ID | Date Received | Sample # | Facility | Sample ID | Date Received |
|---|---|---|---|---|---|---|---|
| 1 | B | 2452 | 2/24/2010 | 21 | H | 2046 | 3/12/2010 |
| 2 | B | 2468 | 2/24/2010 | 22 | H | 2568 | 2/12/2010 |
| 3 | B | 2478 | 2/24/2010 | 23 | J | 2512 | 11/20/2009 |
| 4 | B | 2454 | 3/19/2010 | 24 | J | 2515 | 12/2/2009 |
| 5 | C | 2433 | 11/25/2009 | 25 | J | 2517 | 1/8/2010 |
| 6 | C | 2435 | 12/28/2009 | 26 | J | 2519 | 2/5/2010 |
| 7 | D | 2532 | 11/5/2009 | 27 | L | 2482 | 12/2/2009 |
| 8 | D | 2536 | 1/15/2010 | 28 | L | 2484 | 12/28/2009 |
| 9 | D | 2538 | 2/24/2010 | 29 | L | 2488 | 2/2/2010 |
| 10 | D | 2540 | 3/19/2010 | 30 | L | 2486 | 2/2/2010 |
| 11 | E | 2437 | 2/25/2010 | 31 | L | 2490 | 4/6/2010 |
| 12 | E | 2439 | 2/24/2010 | 32 | M | 2601 | 2/24/2010 |
| 13 | F | 2502 | 11/20/2009 | 33 | M | 2605 | 2/24/2010 |
| 14 | F | 2504 | 12/28/2009 | 34 | M | 2609 | 3/8/2010 |
| 15 | F | 2506 | 12/28/2009 | 35 | M | 2613 | 5/14/2010 |
| 16 | F | 2508 | 2/24/2010 | 36 | N | 2492 | 12/28/2009 |
| 17 | F | 2510 | 4/6/2010 | 37 | N | 2494 | 2/2/2010 |
| 18 | H | 2544 | 11/13/2009 | 38 | N | 2496 | 3/8/2010 |
| 19 | H | 2555 | 12/14/2009 | 39 | N | 2498 | 4/6/2010 |
| 20 | H | 2559 | 1/11/2010 | | | | |

### 2.1.1 Field Sampling

SC samples were collected by a designated third party at a storage area from each facility approximately once a month for four months. After cooling (72 to 120 h), the stored materials were size reduced following ASTM Method C702 - 98: "Standard practice for reducing samples of aggregate to testing size" [68]. Each SC sample was collected using pre-cleaned sampling equipment and divided into two fractions. The first fraction, approximately 1 kg sample, was delivered to U.S. EPA's Center Hill Laboratory (CHL) Research Facility in a shipping container. The second sample fraction (10 to 20 kg) was stored at the test site for further analysis, if needed, based on current findings.

### 2.1.2 Salt Cake Sample Processing

Upon receipt at CHL, samples were logged and stored in a storage unit. Within two weeks, individual samples were processed by placing them into a stainless steel pan and crushing them to a size less than 9 mm by sieve. After thorough mixing, approximately 500 g of the 9 mm sieved materials was further size reduced down to a size of 2 mm

using a grinding machine. About 50 g of the 2 mm size SC sample was further reduced to a size of 0.05 mm using a sealed stainless steel jar mill equipped with zirconia grinding beads. Any sample that was not used in the experiment was returned to its original labeled container and placed in storage.

## 2.2 Salt Cake Characterization

### 2.2.1 Physical Properties

#### 2.2.1.1 Moisture Content (MC)

MC of the SC samples was measured after the sample size was reduced to less than 2 mm. The samples were then placed in an oven and moisture content was determined following ASTM Method D-2216 [69].

#### 2.2.1.2 Water Holding Capacity (WHC)

WHC of the SC samples was measured using a modified ASTM Method F1815-06 [70]. Approximately 10 g of sample (size < 2 mm) was placed into a cylinder (see Figure 2-1), and cold water (Temperature <12°C) was added to saturate the sample. The saturated sample within the cylinder was placed on top of an absorbent membrane until the excess water was drawn away by gravity. Once equilibrium was reached (usually in approximately 16 h), the WHC was calculated based on the weight of the water held in the sample vs. the sample's dry weight. Note that the WHC data must be used with caution because of the reactions that can occur when the SC is mixed with water.

**Figure 2-1: Water holding capacity method illustration**

#### 2.2.1.3 pH and Electrical Conductivity (EC)

The pH and EC of the SC samples (< 2 mm) were measured after the sample reacted with deionized water (DIW) at 50°C for 5d of incubation in a closed system with a gas capture apparatus. The liquid to solid ratio was 20 to 1 (100 ml DIW/5g). After filtering by using a 0.45-µm polyvinylidene fluoride (PVDF) media, pH and EC were measured directly using a hydrogen ion selective electrode and conductivity probe following Standard Methods for the Examination of Water and Wastewater (Standard Method 4500-H pH Value, 2510 Conductivity) [71], respectively. Both the pH and conductivity meters were calibrated before analysis.

### 2.2.2 Chemical Properties of Salt cake

#### 2.2.2.1 Total Elemental Analysis

The SC samples (< 0.05 mm) were acid digested following EPA Method 3051A [72]. Because of the need for aluminum quantification, a mixture of hydrochloric and nitric acid (1 part HCl and 3 parts $HNO_3$) was employed. Furthermore, the microwave temperature was set at 185°C for 30 min rather than the 175°C for 10 min that is specified in the method to increase the recovery of aluminum. The weight of the SC samples was approximately 0.1 g instead of the 0.5 g called for by Method 3051A. After acid digestion, major metal composition (Al, Ca, Cu, Fe, K, Mn, Na, S, and Zn) was measured via EPA Method 6010C using a Thermo inductively coupled plasma atomic

emission spectroscopy (ICP-AES) [73]. Analysis of As, Cd, Cr, Pb, and Se was conducted using a Perkin-Elmer graphite furnace atomic absorption spectrometry (GFAA) (EPA Method 7010) [74]. The method detection limits (MDL) for metals in aqueous phase and the method reporting limit (MRL) for metals in solid phase for ICP are presented in Appendix A (Table A-1). Standard reference material 1633b (Coal Fly Ash), blank spikes and SC samples spikes were also digested during each batch for quality control. The average recovery of spiked Al, K, Na, and Mg in the blank, 1633b, and SC samples (2452-B, 2433-C, 2502-F, 2046-H, 2512-J, 2490-L, and 2613-M) are presented in Table 2-2.

**Table 2-2: Spike recovery for metals**

| Metal | Recovery (%) | | |
|---|---|---|---|
| | Blank | Reference material (1633b) | Salt cake samples (n=7) |
| Al | 104 | 103 | 96±14 |
| K | 93 | 94 | 98±11 |
| Na | 97 | 105 | 85±4.0 |
| Mg | 102 | 103 | 100±4.5 |

#### 2.2.2.2 Leachate Elements Analysis

Leachable elements (Al, Fe, Mg, Cu, Zn, K, Na, Ca, Mn, Pb, Cr, As, Se, Cl and F), following the mixing of SC with water during 5d-long incubation at 50°C (5 g SC to 100 ml DIW in a 500 ml bottle), were analyzed using Thermo ICP-AES using EPA Method 6010C [73] for the macro metals, Perkin-Elmer graphite furnace AA for trace metals by EPA Method 7010 [74], and IC for anions using EPA Method 300.1 [75]. The leachate was filtered using 0.45-µm PVDF media. The MDL and MRL for the metal analysis using GFAA are presented in Appendix A (Table A-2). Due to the high quantity and variation of sodium and aluminum in the leachate, a standard addition method for GFAA analysis was employed for leachate As, Cr, Pb and Se [76, 77].

#### 2.2.2.3 Toxicity Characteristic Leaching Procedure (TCLP)

The toxicity characteristics leaching procedure (TCLP) was conducted as prescribed in U.S. EPA Method 1311 [78]. Two different extraction fluids were employed based on the buffering capacity of each sample. The TCLP extracts were digested following EPA Method 3015A after filtration and acidification (pH < 2.0) and the concentrations of metals were analyzed using Thermo ICP-AES [73]. Of the eight Resource Conservation and Recovery Act (RCRA) toxicity characteristic metals, mercury was not analyzed.

#### 2.2.2.4 X-ray Diffraction (XRD) Analysis

The crystalline mineral phases of SC were investigated from 5 to 110 degrees 2θ on a Philips X'Pert Pro Diffractometer using copper Kα radiation. The SC sample was further powdered (< 0.05 mm) by a sealed stainless-steel jar mill with zirconia grinding beads. The powder diffraction file (PDF) patterns database from the International Centre for Diffraction Data (ICDD) was employed for the search, match, and identification steps. A subset of reference patterns was built for all SC samples. The semi-quantitative phase analysis was performed by the X'Pert HighScore Plus software, based on the CHUNG Normalized RIR Method [79]. The relative intensity of each phase is given by the scale factor, which is determined by least squares fit through all matching reference pattern lines in X'Pert HighScore. The concentration X of phase α is calculated using:

$$X_{\propto} = \frac{I_{(hkl)_{\propto}}}{RIR_{\propto} I^{rel}_{(hkl)_{\propto}}} \left[ \frac{1}{\sum_{j=1}^{n} \left( I_{(hkl)j} RIR_j I^{rel}_{(hkl)_j} \right)} \right] \quad (2\text{-}1)$$

where $RIR_{\propto}$ is Reference Intensity Ratio, based on the relative net peak height ratio of the strongest line ($I^{rel}$ = 100%)

of the phase and of the strongest line of corundum, measured with copper Kα radiation in a mixture of equal weight percentages and $I_{(hkl)_\alpha}$ is Intensity of reflection of hkl in phase α. hkl are the reflection indices.

The normalization used in this method assumes that the sum of all identified phases is 100%. This means that there are no unidentified crystalline phases or amorphous phases assumed to be present in the sample. Only under these conditions can meaningful, semi-quantitative results be obtained.

## 2.3 Reactivity of Salt cake

The chemical reaction of aluminum species with water typically generates heat and gas. Therefore, the reactivity of SC as a result of SC reaction with liquid was evaluated using three indicators: 1) temperature increase as well as time to reach the maximum temperature; 2) heat generation potential; and 3) gas generation potential. It is noted that the SC samples in this work were from 10 different facilities, which might contribute some variability of the reactivity of SC. However, this report will focus on the general patterns of the reactivity of SC.

### 2.3.1 Apparatus for Testing Salt Cake Reactivity

The apparatus presented in Figure 2-2 was utilized to evaluate the increase in temperature caused by the reaction of SC with liquid. The target SC sample was placed in a 60 ml volatile organic analyte (VOA) vial and purged with argon for five minutes. The vial was then sealed and connected to a 1 L Tedlar bag for pressure release. Pre-heated DIW was then added to the sealed vial to achieve certain liquid to solid ratio. The change in temperature as a result of the SC reaction with liquid was monitored using a thermocouple wire placed in the sample. Sand samples were utilized as control under the same experimental condition (e.g. liquid to solid ratio). When the average temperature difference between the sample and control reached less than 0.5°C, the experiment was stopped. The overall experimental duration typically ranged from 72 to 120 h. It is noted that the sensitivity of the thermocouple is 0.5°C and as a result this value was utilized to determine the end of the experiment. Furthermore, once the difference between the control and the SC sample reached the 0.5°C, the experiment was kept for 10 h to ensure the stability of the temperature difference at this level.

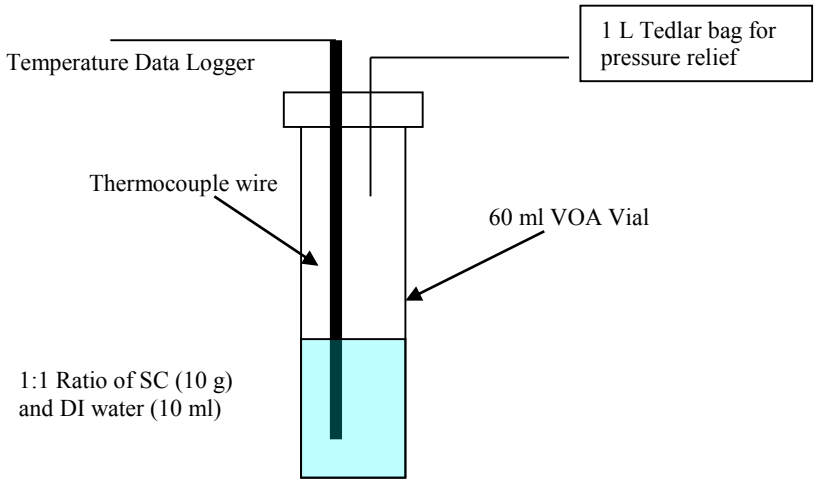



**Figure 2-2: Temperature increase apparatus**

The heat generation potential was evaluated using a custom-made reaction calorimeter presented in Figure 2-3. The calorimeter is composed of double glass vessels, an inner-vessel (40 ml VOA vial) and an outer-vessel (250 ml vial with a three-port Teflon cap). The inner-vessel is immersed in the outer-vessel containing 90 ml ethylene glycol as a thermal buffer since it has a low specific heat capacity. The calorimeter is equipped with three thermocouple wires immersed in the ethylene glycol solution and connected to a data logger. The inner-vessel is connected to a 1 L Tedlar bag to relieve any pressure resulting from the reaction of SC with liquid.

The gas generation potential of SC was evaluated under anaerobic conditions (to simulate MSW landfill environment) using the test apparatus presented in Figure 2-4. The target SC sample was placed into a 500 ml lined media bottle sealed with a lyophilization stopper and a cap. The media bottle was then purged with argon for 10 minutes. An aliquot of oxygen-free, argon-purged, and pre-heated water was then added to the sample to achieve a certain liquid to solid ratio. A 1 L Tedlar bag was connected to the bottle to collect the gas generated from the reaction. The media bottles were then placed in a 50°C incubator (Fischer Scientific Isotemp Dual $CO_2$ Incubator) for 5 d, after which the Tedlar bag was disconnected and gas quantity and composition were measured. It is noted that the sample bottles were allowed to reach room temperature before performing the gas volume and composition measurements.

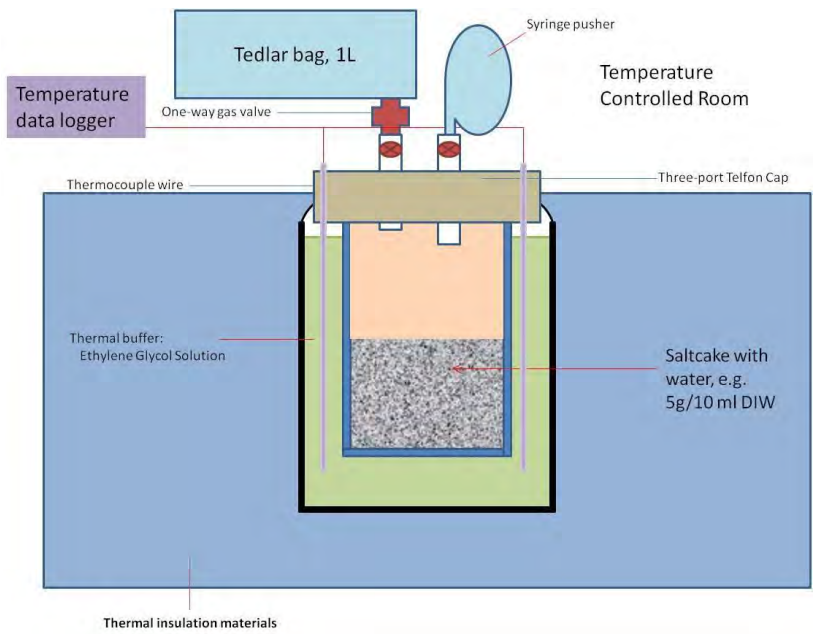



**Figure 2-3: Heat measurement apparatus**

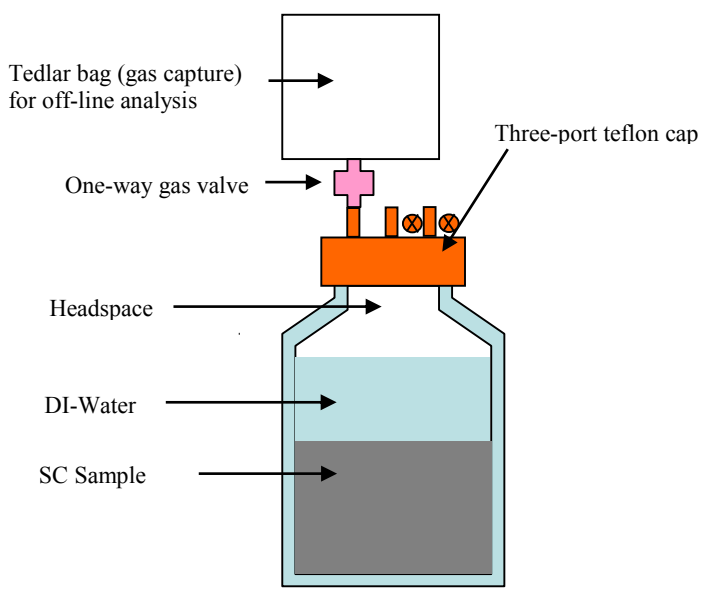



**Figure 2-4: Reactivity test apparatus for gas generation measurement**

## 2.3.2 Factors Impacting Salt Cake Reactivity

Several factors may impact the SC reactivity and include environmental temperature, SC particle size, liquid addition rate, liquid to solid ratio, chemical composition of the liquid, and chemical composition of SC. The impact of these parameters on SC reactivity was evaluated and the results were utilized to select the experimental conditions for the temperature increase and gas generation experiments.

### 2.3.2.1 Effect of Environmental Temperature

Randomly selected SC samples (2439-E, 2510-F, 2482-L, 2488-L, and 2601-M) were employed to investigate the impact of environmental temperature (20, 37, and 50°C) on the temperature change upon exposure to water. Samples with particle size < 2 mm (size was achieved as described in section 2.1.2) were mixed with pre-heated DIW (pH 6.2) to achieve a liquid to solid ratio of 1 to1 (10 ml DIW to 10 g sample) and the temperature increase was measured using the apparatus described in section 2.3.1.

To evaluate the impact of environmental temperature (37 and 50°C) on the heat generation potential of SC, samples (2502-F, 2046-H, 2490-L, and 2613-M) with size < 0.05 mm were utilized. A 10 ml pre-heated DIW (pH 6.2) was added to the samples to achieve a liquid to solid ratio of 2 to1 (10 ml DIW to 5 g sample). The heat generation potential was measured using the apparatus described in section 2.3.1.

To evaluate the impact of environmental temperature (4, 20, 37, and 50°C) on the gas generation of SC, samples (2439-E, 2046-H and 2512-J) with size < 2 mm were utilized. A 20 ml of pre-heated DIW to the target temperature (pH 6.2) was added to the samples to achieve a liquid to solid ratio of 20 to1 (20 ml DIW to 1 g sample, duplicated). The hydrogen and methane productivity were measured after five days reaction in 60 ml vials, which were similar to the apparatus described in section 2.3.1.

### 2.3.2.2 Effect of Salt Cake Particle Size

Randomly selected SC samples (2502-F, 2512-J, and 2046-H) were tested at 37°C. Three different particle sizes were compared, < 0.05, < 2, and < 9 mm. Ten ml pre-heated DIW (37°C) with a pH 6.2 was added to 10 g SC sample to achieve 1:1 liquid to solid ratio. The increase in temperature as a function of particle size was measured using the apparatus described in section 2.3.1.

To evaluate the impact of particle size (< 0.05 and < 2 mm) on the heat generation potential of SC, samples (2502-F, 2512-J, 2046-H, and 2490-L) were utilized. Ten ml pre-heated DIW (pH 6.2) was added to the samples to achieve a liquid to solid ratio of 2 to1 (10 ml liquid to 5 g sample). The heat generation potential was measured using the apparatus described in section 2.3.1.

To evaluate the impact of particle size (< 0.05, < 2, and < 9 mm) on the gas generation potential of SC, samples (2512-J, 2046-H, and 2613-M) were utilized. Twenty ml pre-heated DIW (pH 6.2) was added to the samples to achieve a liquid to solid ratio of 20 to1 (20 ml liquid to 1 g sample, duplicated) at 37°C environmental temperature. The hydrogen and methane productivity was measured after five days reaction in 60 ml vials, which were similar to the apparatus described in section 2.3.1.

### 2.3.2.3 Effect of Liquid Addition Rate

Ten g of SC sample 2069M with particle size < 2 mm was exposed to two treatments to investigate the impact of liquid addition rate on the reactivity of SC. The first treatment received 10 ml DIW at once and acted as a control. The second treatment received 10 ml DIW intermittently over a period of 72 h. Both treatments were conducted at 37°C. The increase in temperature as a function of hydration/dehydration cycle was measured using the apparatus described in section 2.3.1.

#### 2.3.2.4 Effect of Liquid Chemical Composition

The effect of chemical composition of liquid on SC reactivity was evaluated by measuring the temperature increase, heat generation and gas generation from samples exposed to liquids with different chemistry.

The apparatus described in section 2.3.1 was utilized for conducting this test. Randomly selected SC samples (Table 2-3) with particle size < 2 mm (for temperature increase and gas generation) and < 0.05 mm (for heat generation and gas generation) were employed. The ratio of liquid to solid and the various liquids utilized are presented in Table 2-3. All liquids used were pre-heated to 37 or 50°C, the temperatures at which the experiments were carried out.

#### 2.3.2.5 Effect of Liquid to Solid Ratio and Mass of Salt Cake

Randomly selected SC samples were employed to investigate the effect of liquid to solid ratio and mass of SC on SC reactivity. Different treatments were used where the liquid to solid ratio or the mass of SC were varied as presented in Table 2-4. Pre-heated DIW (pH 6.2) was used as the liquid. The effect of liquid to solid ratio was evaluated at 50°C and the effect of mass of SC was evaluated at 37°C. The reactivity of SC in each case was evaluated by measuring the temperature increase, heat generated or gas generated for the samples using the apparatus described in section 2.3.1.

**Table 2-3: Treatments for testing the effect of liquid composition on salt cake reactivity**

| Reactivity Indicator | Liquid to Solid Ratio | Solution chemical composition | pH | Sample ID |
|---|---|---|---|---|
| Temperature Change | 1:1 (10 ml liquid to 10 g SC, < 2 mm, 37°C) | DIW | 6.2 | 2502-F, 2555-H, 2046-H, and 2613-M |
| | | 0.1 M HCl | 1.0 | |
| | | 0.1 M NaOH | 13 | |
| | | 1% salt of humic acid* | 8.3 | |
| | | VFA** | 5.3 | |
| | | Landfill Leachate*** | 8.1 | |
| Heat Generation | 2:1 (10 ml liquid to 5 g SC, < 0.05 mm, 50°C) | DIW | 6.2 | 2502-F, 2555-H, and 2613-M |
| | | 0.1 M HCl | 1.0 | |
| | | 0.1 M NaOH | 13 | |
| | | VFA | 5.3 | |
| | | Landfill Leachate | 8.1 | |
| Gas Generation | 20:1 (20 ml liquid to 1 g SC, < 0.05 mm, 50°C) | DIW | 6.2 | 2512-J, 2489-L, 2046-H, 2555-H, and 2613-M |
| | | 0.1 M HCl | 1.0 | |
| | | 0.1 M NaOH | 13 | |
| | | Landfill Leachate | 8.1 | |
| | | 1% salt of humic acid | 8.3 | |
| | | VFA | 5.3 | |

*Salt of humic acid, Sigma-Aldrich (CAS # 68131-04-4).
** VFA composition: 1,795 mg $L^{-1}$ acetic acid; 1,296 mg $L^{-1}$ propionic acid; and 1,284 mg $L^{-1}$ n-butyric acid;
***Landfill leachate: DOC: 20,000 mg $L^{-1}$

#### 2.3.2.6 Effect of Aluminum Species in Salt Cake Simulators

To understand the mechanism of temperature increase in SC and the effect of SC composition on the magnitude of temperature change, commercial chemicals NaCl, KCl, $Al_2O_3$, metallic aluminum, AlN, and $Al_4C_3$ were used in varying mixtures to simulate the SC chemical composition. The reason for synthesizing SC for the purpose of this test over using the actual SC samples is to enable the understanding of the role of each individual aluminum species in SC reactivity. The particle sizes of $Al_2O_3$, $Al_4C_3$, metallic aluminum, and AlN in the mixture were 0.05, 10, 20, and 44 µm, respectively. The particle sizes of added salt (NaCl and KCl) were less than 1 mm. The liquid to solid ratio was 1 to 1 (10 ml DIW to 10 g solid sample). The experiments were conducted at 37°C using the apparatus described in section 2.3.1 to measure the temperature change for each scenario. The basic mixture (Mixture I) was composed of 5% KCl, 10% NaCl, and 85% $Al_2O_3$. The details of the chemical composition in these SC simulators are presented in Table 2-5. It is noted that a control sample (sand) was employed with each set of experiments.

### 2.3.3 Salt Cake Reactivity Evaluation

The results of the experiments conducted in section 2.3.2 to evaluate the impact of various parameters on SC reactivity were the basis for selecting the experimental conditions for evaluating the reactivity of all the 39 SC samples investigated in the current study. The reactivity was measured by the temperature increase, heat generation and gas generation after SC reaction with water.

#### 2.3.3.1 Temperature Increase

The apparatus presented in Figure 2-2 was utilized to measure the temperature increase as a result of SC reaction with DIW. A 10 g SC sample of < 2 mm size was placed into a 60 ml VOA vial. After purging the vial with argon for 5 min, the vial was sealed and 10 ml pre-heated DIW was then added to the vial to achieve 1:1 liquid to solid ratio. The experiments were conducted at two different temperature conditions (37 and 50°C). The temperature conditions were selected based on reported ideal temperatures for mesophilic (35 to 40°C) and thermophilic (50 to 60°C) bacteria that are normally present in MSW landfills [58, 59] and temperatures consistent with reported landfill temperatures [80-82]. The temperature conditions were achieved by placing the experimental apparatus in an incubator heated to the desired temperature (Fischer Scientific Isotemp Dual $CO_2$ Incubator). Usually, six to 12 sample vials and one control sample vial were grouped as one set arranged in an insulated box inside the incubator (Figure 2-5). Insulation materials were used to isolate the heat transfer among the vials and to decrease the influence among the samples. The increase in temperature as a function of time was measured as previously described in section 2.3.1.

**Table 2-4: Treatments for testing the effect of liquid to solid ratio and mass of salt cake on the reactivity**

| Reactivity Indicator | Liquid to Solid Ratio | Liquid Volume (ml) | Solid Mass (g) | Particle Size (mm) | Sample ID |
|---|---|---|---|---|---|
| Temperature Increase | Variable | 3 | 10 | 2 | 2478-B |
| | | 5 | 10 | 2 | |
| | | 10 | 10 | 2 | |
| | | 15 | 10 | 2 | |
| | Variable | 3 | 5 | 2 | 2478-B |
| | | 3 | 10 | 2 | |
| | | 3 | 15 | 2 | |
| | Fixed (3:10) | 1.5 | 5 | 2 | 2478-B |
| | | 3 | 10 | 2 | |
| | | 4.5 | 15 | 2 | |
| | | 6 | 20 | 2 | |
| | Fixed (1:2) | 2.5 | 5 | 2 | 2601-M |
| | | 5 | 10 | 2 | |
| | | 7.5 | 15 | 2 | |
| | Fixed (1:1) | 5 | 5 | 2 | 2482-L, 2506-F, and 2439-E |
| | | 10 | 10 | 2 | |
| | | 15 | 15 | 2 | |
| | | 20 | 20 | 2 | |
| Heat Generation | Variable | 10 | 1 | 0.05 | 2613-M |
| | | 10 | 2 | 0.05 | |
| | Fixed (2:1) | 6 | 3 | 0.05 | 2046-H, 2490-L and 2502-F |
| | | 10 | 5 | 0.05 | |
| | | 15 | 7.5 | 0.05 | |
| | | 20 | 10 | 0.05 | |
| Gas Generation | Variable | 2.5 | 5 | 2 | |
| | | 5 | 5 | 2 | |

| | | | | |
|---|---|---|---|---|
| | 25 | 5 | 2 | 2433-C, 2512-J, and 2613-M |
| | 100 | 5 | 2 | |

### 2.3.3.2 Heat Generation Potential

The heat generation potential was evaluated using the calorimeter described in section 2.3.1. A 5 g SC sample (size < 0.05 mm) was added to the 40 ml inner-vessel which was then capped and placed in the 250 ml outer vessel containing 90 ml pre-heated ethylene glycol. The experiments were conducted at two different temperature conditions, 37 and 50°C, which were achieved by placing the calorimeter in an incubator heated to the desired temperature (Fischer Scientific Isotemp Dual $CO_2$ Incubator). Ten ml pre-heated liquid was injected to the inner-vessel using a syringe to achieve a liquid to solid ratio of 2:1. The temperature of the ethylene glycol solution was monitored over time (one reading every two min recorded using a data logger) until no temperature change was observed from the reaction (at least 72 h). With each batch (4 double vessels in an insulated box) sand samples were employed as controls under the same experimental conditions.

**Table 2-5: Composition of the SC stimulators for testing the impact aluminum species on the reactivity**

| Experiment | Composition | Al (%) | AlN (%) | $Al_4C_3$ (%) | Other (%) |
|---|---|---|---|---|---|
| I | 1 | 0 | 0 | 0 | 100- Mixture I* |
| | 2 | 5 | 0 | 0 | 95- Mixture I |
| | 3 | 10 | 0 | 0 | 90- Mixture I |
| | 4 | 20 | 0 | 0 | 80- Mixture I |
| II | 1 | 0 | 2 | 0 | 98- Mixture I |
| | 2 | 0 | 5 | 0 | 95- Mixture I |
| | 3 | 0 | 10 | 0 | 90- Mixture I |
| | 4 | 10 | 2 | 0 | 88- Mixture I |
| | 5 | 10 | 10 | 0 | 80- Mixture I |
| III | 1 | 5 | 1 | 0 | 94- Mixture I |
| | 2 | 20 | 1 | 0 | 79- Mixture I |
| | 3 | 5 | 2 | 0 | 93- Mixture I |
| | 4 | 20 | 2 | 0 | 78- Mixture I |
| IV | 1 | 0 | 0 | 5 | 95- Mixture I |
| | 2 | 5 | 0 | 5 | 90- Mixture I |
| | 3 | 10 | 0 | 5 | 85- Mixture I |
| | 4 | 20 | 0 | 5 | 75- Mixture I |
| V | 1 | 20 | 0 | 0 | 54% NaCl and 26% KCl |
| | 2 | 20 | 5 | 0 | 50% NaCl and 25% KCl |
| | 3 | 20 | 10 | 0 | 47% NaCl and 23% KCl |

*Mixture I is composed of 5% KCl, 10% NaCl, and 85% $Al_2O_3$.

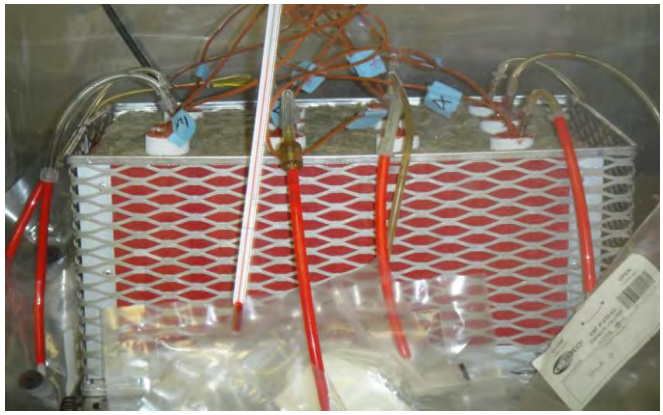


**Figure 2-5: Sample arrangement in an insulation box for temperature change experiment**

For every recorded reading, the difference in temperature (ΔT) between the SC sample and control was calculated. It is noted that each temperature reading is the average of the three thermocouple readings. The sum of temperature change as compared to the control (ΣΔT) was converted to heat using a calibration curve. The calibration curve was constructed between the ΣΔT and the heat generated by a fixed resistance (R) when applying varying electric current (I) with time. The power (P) is calculated using the following relationship:

$$P = VI \qquad (2-1)$$

Where P is the power, I is the current, and V is the voltage (V = I × R). The generated heat (Q) from a resistance is related to the power and time as follows:

$$Q = PT \qquad (2-2)$$

The calibration curve is constructed by plotting the ΣΔT and Q. Examples of detailed calculations and calibration curve are presented in Table 2-6 and Figure 2-6, respectively. The following relationship was obtained from the calibration curve under laboratory setting:

$$Q = 0.006\Sigma\Delta T \qquad (2-3)$$

**Table 2-6: Calculations of heat generation from a fixed resistance and ΣΔT**

| Resistance (Ω) | Current (mA) | Time Energized (min) | Power Applied (W) | Voltage (V) | Energy (kJ) | ΣΔT (°C per 2min) |
|---|---|---|---|---|---|---|
| 45.8 | 135 | 60 | 0.83 | 6.2 | 3 | 480 |
| | 174 | 60 | 1.4 | 8.0 | 5 | 800 |
| | 191 | 60 | 1.7 | 8.7 | 6 | 1,100 |
| | 246 | 60 | 2.8 | 11 | 10 | 1,700 |
| | 348 | 60 | 5.6 | 16 | 20 | 3,100 |
| | 427 | 60 | 8.3 | 20 | 30 | 5,000 |
| | 174 | 120 | 1.4 | 8.0 | 10 | 1,700 |
| | 246 | 120 | 2.8 | 11 | 20 | 3,000 |

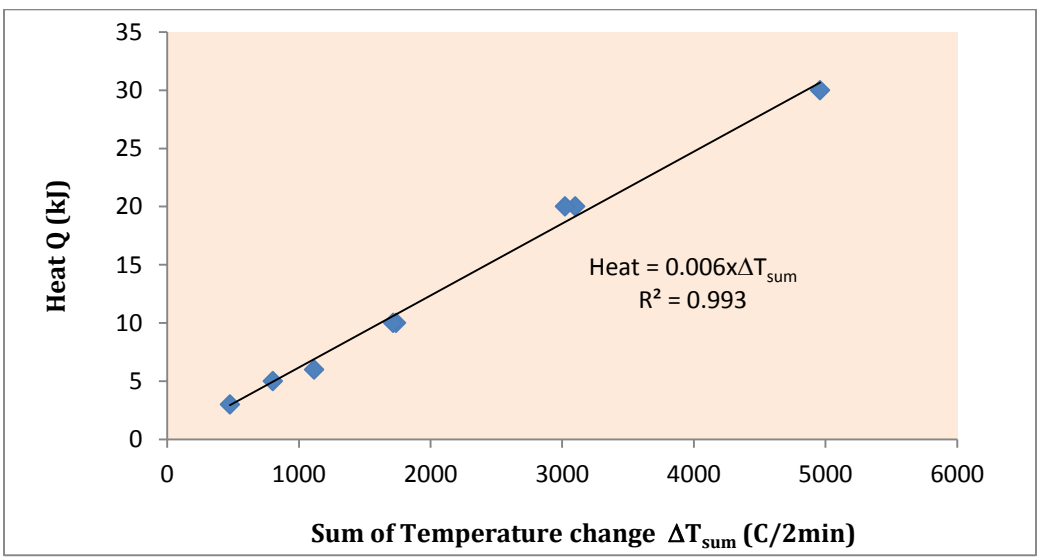



**Figure 2-6: Example of calibration curve for heat calculation**

### 2.3.3.3 Gas Generation Potential

The apparatus presented in Figure 2-4 was utilized for evaluating the gas generation potential as a result of SC reaction

with water. A 5 g SC sample with size < 2 mm was placed into a 500 ml lined media bottle sealed with a lyophilization stopper and cap. The media bottle was then purged with argon for 10 mins. One hundred ml of oxygen-free, argon-purged, pre-heated (50°C) water was then added to the sample to achieve a 20:1 liquid to solid ratio. The volume and composition of the gas generated were measured after 5d reaction as presented below.

#### *2.3.3.3.1 Gas Volume*

The gas volume was measured after 5d reaction using 60 ml gas-tight syringe. The gas volume generated was composed of two portions, one is the gas present in the bottle's headspace and the other is the gas collected in the Tedlar bag. Disconnected the Tedlar bag, and placed both bottle and Tedlar bag under a chemical fume hood at room temperature (25 $^{o}$C) for 4 hours. The headspace gas fraction was measured by attaching a 20 gauge needle to the syringe and then inserting the needle through the bottle septum allowing the syringe plunger to move freely. The gas volume fraction collected in the Tedlar bag was extracted using the syringe. The total gas volume was calculated by the sum of the two fractions.

#### *2.3.3.3.2 Gas Composition*

*Hydrogen ($H_2$) and Methane ($CH_4$)*

The concentrations of hydrogen ($H_2$) and methane ($CH_4$) gas content were determined via analysis of a 100 µL headspace sample using an Agilent 6980 Gas Chromatograph equipped with a thermal conductivity detector (GC/TCD). The gas components were separated by the two analytical columns (PorapakN packed column, and Molesieve 5A packed column), controlled by an automated valve switch. A five-point calibration curve was prepared to quantify the gas composition. The calibration range was 1 to 100% for $H_2$ and 1.67 to 100% for $CH_4$. The MRL was 0.14% for $H_2$ and 0.2% for $CH_4$ (Appendix A, Table A-3). A standard gas mixture (15% $CO_2$, 15% $CH_4$, 4% $H_2$, and 66% $N_2$) purchased from Matheson TRI-GAS Inc. was used as a standard check before and after gas analysis.

*Nitrous Oxide ($N_2O$)*

Nitrous oxide ($N_2O$) concentration was determined via analysis of a 100 µl headspace sample using an Agilent 5975 Gas Chromatograph equipped with a split/splitless injection port operated in the split mode and Electron Capture Detector (GC/ECD). The gas components were separated using a J&W GS-Gaspro column, 60 meters in length and 0.32 mm in diameter, then detected by a µ–ECD detector. A six-point calibration curve was prepared to quantify the $N_2O$ composition. The retention time of $N_2O$ for the specified conditions was 4.94 minutes, and the linear concentration ranged from 20 to 2000 parts per billion in volume (ppbv). The MRL for $N_2O$ was 20 ppbv (Appendix A, Table A-3).

*Hydrogen Sulfide ($H_2S$)*

Hydrogen sulfide ($H_2S$) gas content was measured using a Jerome meter (JEROME® 631-X, Arizona Instrument LLC) according to the method presented in the instrument manual [83]. The detection range of the Jerome meter for $H_2S$ ranges between 3 ppb to 50 ppm [83] with a large flow rate (150 ml min$^{-1}$).

*Ammonia (gaseous $NH_3$) and Ammonium (aqueous $NH_4^+$)*

After the SC reacts with water, the generated ammonia can be present in the gaseous form as $NH_3$ or in aqueous form as hydrated ions ($NH_4^+$) and dissolved gas. The equilibrium between these two forms is mainly dependent on solution pH. To measure $NH_3$ content in the gaseous form, a 20 ml headspace gas sample was injected into 20 ml of 0.1 M $H_2SO_4$ acid solution to transfer all the gaseous form of ammonia into $NH_4^+$. The concentration of $NH_4^+$ in the solution was measured using an ammonia electrode (Standard Methods for the Examination of Water and Wastewater, 4500-$NH_3$ D, E) [71]. To measure the $NH_4^+$ concentration in the liquid form, approximately 2 to 5 ml of liquid in the media bottle (Figure 2-4) was collected (without filtration) and injected using a syringe into a 50 ml volume of 0.05M $H_2SO_4$ acid solution. The concentration of $NH_4^+$ was determined as previously described [71].

#### *2.3.3.3.3 Gas Productivity*

The $H_2$, $CH_4$ and $N_2O$ gas productivity (mole gas $g^{-1}$ SC sample) was calculated as follows:

- Multiply the total gas volume (measured using syringe) by the gas concentration (measured using GC) to get the volume (L) of each individual gas.
- To convert each individual gas volume to moles of gas, divide the volume by 24.45 L. One mole of an ideal gas at STP occupies 22.4 liters but under the current experimental condition (25°C) the corrected volume for one mole of gas is 24.45 L.
- The productivity (mole $g^{-1}$) of each gas is then calculated by normalizing the moles of gas produced to the SC sample weight used.

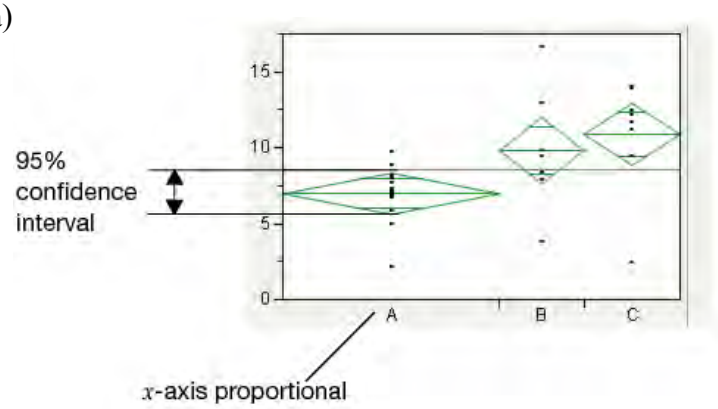
The $NH_3$ gas productivity (mole gas $g^{-1}$ SC sample) was calculated as follows:

- The $NH_3$ concentration measured in the acid solution in mg $L^{-1}$ (section 2.3.2.2.4) was converted to molar concentration (M).
- The moles of $NH_3$ (moles $NH_3$) is calculated by multiplying the acid solution volume (20 ml) by the M concentration of $NH_3$.
- The total moles of $NH_3$ in the gas was calculated by multiplying the moles $NH_3$ by the total gas volume generated and dividing this quantity by 20 (the volume of headspace gas sample analyzed for ammonia).
- The ammonia gas productivity (mole $g^{-1}$) was calculated by normalizing the total moles of gas produced to the SC sample weight used.

## 2.4 Statistical Analysis

Statistical analysis and graphical representation of the data was performed using Microsoft Excel 2007, JMP 9.0, and SigmaPlot 11.0 [84, 85]. The statistical analysis technique was chosen based on the properties of parameters. The mean, standard error, minimum and maximum values as well as $UCL_{95}$ (95% upper confidence limit) were used to summarize the content of elements, mineral phases, and the temperature change, as well as the gas composition and gas production. UC $L_{95}$ was used to describe the potential risk or hazards in USEPA [86]. It is noted that the $UCL_{95}$ is a probability statement: it means "I am 95% confident that the true distribution of the sampling data has a population mean less than or equal to my calculated UCL".

a)



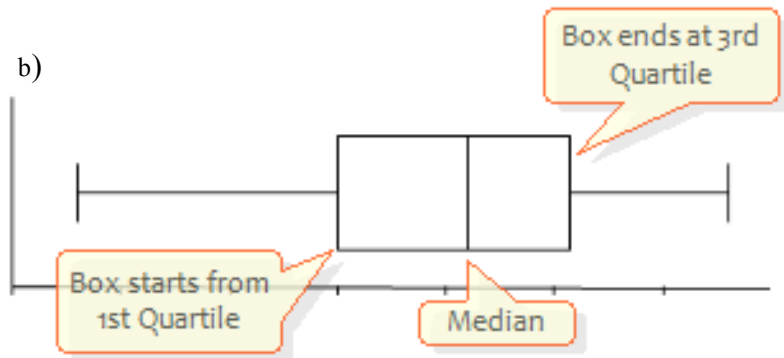



**Figure 2-7: Examples of mean diamonds and box plot**

Box plots with mean diamonds were employed to graphically depict groups of numerical data through their summaries (minimum, lower quartile, median, mean, upper quartile, and maximum). A mean diamond illustrates a sample mean and confidence interval. Figure 2-7 is an example of Mean Diamonds and Box plot. The top and bottom of each diamond (Fig. 2-7a) represent the 95% confidence interval for each group. The confidence interval computation assumes that the variances are equal across observations. Therefore, the height of the diamond is proportional to the reciprocal of the square root of the number of observations in the group. The mean line across the middle of each diamond represents the group mean. Overlap marks appear as lines above and below the group mean. For groups with equal sample sizes, overlapping marks indicate that the two group means are not significantly different at the given confidence level. Basic box plots show a simple rectangular box-plot, from the first to the third quartile, with the median marked in the center (Fig. 2-7b).

One-way ANOVA test was employed to judge the differences of these parameters among facility, including the interaction of the random facility and the tested conditions (e.g. environmental temperature). If there were differences, LS Means Difference Tukey HSD were further tested the differences among factors.

Correlations between the temperature change and mineral phases, or relation between trace metal content and aluminum content, were analyzed using the Pearson product-moment correlation and Spearman's rank correlation to calculate the p-level. The Pearson product-moment correlation coefficient is a measure of the correlation (linear dependence) between two variables X and Y, giving a value between +1 and −1, inclusive. Spearman's rank correlation coefficient is a non-parametric measure of statistical dependence between two variables. It assesses how well the relationship between two variables can be described using a monotonic function. If there are no repeated data values, a perfect Spearman correlation of +1 or −1 occurs when each of the variables is a perfect monotone function of the other. If $p < 0.05$, it was assumed that the relation was significant; and if $p < 0.01$, the correlation was considered very significant. If $p > 0.05$, it was assumed that there was no significant correlation. The linear correlations were used for the calibrations and relations between the properties of parameters (e.g. $\Delta T_{max}$ vs heat). The correlations of the properties of parameters, ANOVA are also performed to test the significance of facility. The bivariate normal confidence ellipse region were also presented ($p = 0.95$). The density ellipsoid is computed from the bivariate normal distribution fit to the X and Y variables. The bivariate normal density is a function of the means and standard deviations of the X and Y variables and the correlation between them. These ellipses are both density contours and confidence curves. As confidence curves, they show where a given percentage of the data is expected to lie, assuming the bivariate normal distribution. The density ellipsoid is a good graphical indicator of the correlation between two variables. The ellipsoid collapses diagonally as the correlation between the two variables approaches either +1 or -1. The ellipsoid is more circular (less diagonally oriented) if the two variables are less correlated.

## 2.5 Quality Metrics

The accuracy checks, precision, calibration of instrumentation, and the determination of detection limits are used to ensure the quality control and the confidence level of the obtained results. Precise, documented and valid data are

needed for the ultimate decisions to be made. To ensure the quality of the data, all instruments are regularly calibrated. QA/QC checks were done to ensure the precision and accuracy of the data. Table 2-7 summarizes the QA/QC checks for each monitoring parameter in this report.

**Table 2-7: Summaries of QA/QC Checks**

| Parameter | QC Check | Method | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| Moisture Content | Initial Calibration | 1 point calibration | At the beginning | Within ± 5% of the full range of the probe | 1. Re-calibrate |
| Water holding capacity | Initial Calibration | 1 point calibration | At the beginning | Within ± 5% of the full range of the probe | 1. Re-calibrate |
| pH | Initial Calibration | 3 point calibration | At the beginning | Within ± 0.1 pH unit of the correct value | 1. Re-calibrate |
| Conductivity | Initial Calibration | 1 point calibration | At the beginning | Within ± 2% of the full range of the probe | 1. Re-calibrate |
| Total metal | Calibration | 5 point calibration | Before each run | $R^2 \geq 0.99$ | 1. Re-calibrate |
| | Precision | Sample duplicate | For every 12 samples | ≤ 25% RPD | 1. Re-run<br>2. Prepare new standards<br>2. Check calculations |
| | Accuracy | Standard check | With every run | 75-125% recovery | 1. Re-calibrate |
| Ammonia ($NH_4$) | Initial calibration | 4 point calibration | At the beginning | $R^2 \geq 0.995$ | 1. Re-calibrate |
| | Continuing calibration | Run mid-point standard | Every 12 samples | ±20% of the actual concentration | 1. Re-calibrate |
| | Precision | Sample duplicate | Every 6 samples | ≤ 20% RPD | 1. Redo triplicate<br>2. Investigate the problem |
| | Blank | Laboratory blank | Every 6 samples | ≤ Reported detection limit | 1. Investigate problem<br>2. Remove contamination<br>3. Check other blanks |
| | Accuracy | Matrix Spike | Every 6 samples | 100 ± 20% recovery | 1. Re-run spike<br>2. Check calculations<br>3. Re-run samples as required |
| Metals (leachate) | Calibration | 4 point calibration | Before each run | $R^2 \geq 0.995$ | 1. Re-calibrate |
| | Precision | Sample Duplicate | Every 12 samples | ≤ 15% RPD for macro-elements (Al, Fe, Ca, K, Na, S and Mg) and ≤ 25% for trace metals (Ag, Ba, Cu, Mn, Zn, Cd, As, Se, Cr and Pb). | 1. Re-analyze<br>2. Investigate the problem |
| | Blank | Laboratory blank | Every 12 samples | ≤ Reported detection limit | 1. Investigate problem<br>2. Remove contamination<br>3. Check other blanks |
| | Accuracy | Check Standard | Every 12 samples | 100 ± 15% recovery for major metals and 100 ± 25% for trace metals | 1. Re-calibrate<br>2- Re-run samples as required |
| Temperature | Initial Calibration | 1 point calibration | At the beginning, and once a year | Within ± 2% of the full range of the probe | 1. Use new thermocouple (within 1 year) |
| Heat measurement | Initial Calibration | 1 point calibration | Every 6 months | Within ± 15% | 1. Re-do sample<br>2. Investigate the problem-Flag data |
| Crystallinity (XRD) | Calibration | Reference standard | Prior to sample analysis | | 1. Re-calibrate |
| Gas volume | Initial Calibration | 1 point calibration | At the beginning | Within ± 5% of the full range of the probe | 1. Re-calibrate |
| Gas Composition | Initial Calibration | 6 point calibration | Monthly | $R^2 \geq 0.990$ | 1. Re-calibrate |
| | Precision | Sample Duplicate | Every 12 samples | ≤ 20% RPD | 1. Re-do sample<br>2. Investigate the problem-Flag data |
| | Accuracy | Standard Check | Twice a Week | 100 ± 20% recovery | 1. Re-calibrate |

# 3. PHYSICAL AND CHEMICAL PROPERTIES

## 3.1 Moisture Content (MC)

The average MC of the investigated SC was 0.8%, with a range of 0.01 to 2% as presented in Table 3-1. The variability of MC by facility and the overall distribution are presented in Figure 3-1. The detailed MC results for each sample are listed in Appendix B (Table B-1). No evidence was shown that there were statistically significant differences from the various facilities, which may be due to the limited sample sizes.

**Table 3-1: Salt cake moisture content**

| Facility | Sample number | Mean | $UCL_{95}$* | Range |
|---|---|---|---|---|
| B | 4 | 0.7±0.4 | 1 | 0.4-1 |
| C | 2 | 0.9±0.6 | 6 | 0.5-1 |
| D | 4 | 1±0.9 | 2 | 0.1-2 |
| E | 2 | 0.8±0.02 | 0.9 | 0.7-0.8 |
| F | 5 | 0.8±0.4 | 1 | 0.04-1 |
| H | 5 | 1±0.2 | 1 | 0.8-1 |
| J | 4 | 0.6±0.4 | 1 | 0.3-1 |
| L | 5 | 0.9±0.7 | 2 | 0.3-2 |
| M | 4 | 0.9±0.9 | 2 | 0.01-2 |
| N | 4 | 0.4±0.3 | 0.9 | 0.1-0.8 |
| All Facilities | 39 | 0.8±0.5 | 1 | 0.01-2 |

*Upper 95% confidence level on the mean

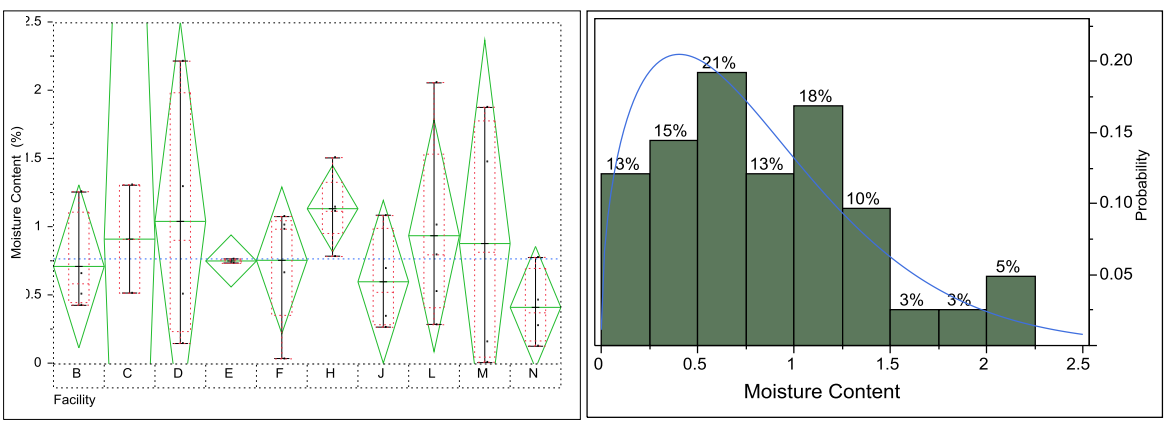



**Figure 3-1: Salt cake moisture content and its distribution**

## 3.2 Water Holding Capacity (WHC)

Water holding capacity (WHC) is an important physical characteristic of materials. The overall average WHC of SC was 25%, ranged from 1.4 to 70% (Table 3-2), and followed a normal distribution (Figure 3-2). Statistically, there were significant differences in WHC between facilities. SC samples from Facility M had the highest WHC, which was more than 20 times the average WHC of Facility B, the facility which had the SC with the lowest WHC. The detailed WHC results for each sample are listed in Appendix B (Table B-1).

The measured WHC values should be viewed with some caution – as described, some degree of reaction of the SC with water may occur, which could result in a mass loss and ultimately skew the WHC measurements. Mass loss would result in an under reported WHC value.

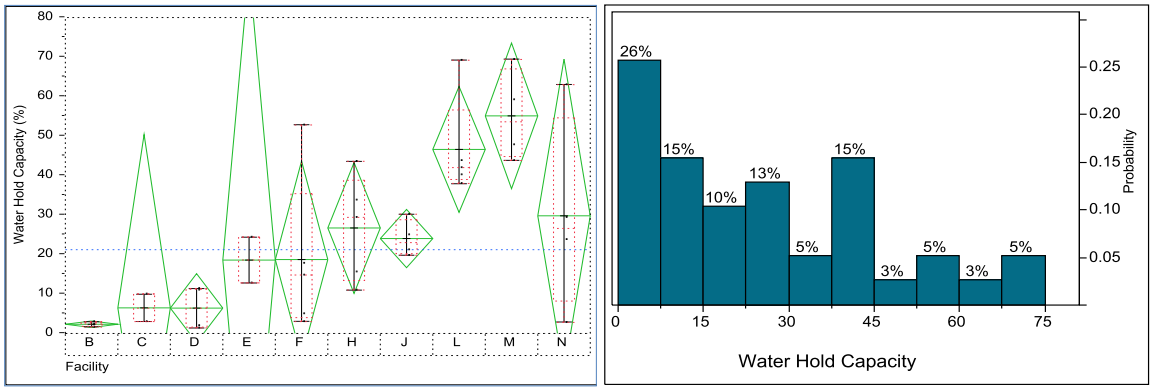



**Figure 3-2: Salt cake water holding capacity and its distribution**

**Table 3-2: Salt cake water holding capacity (%)**

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| B | 4 | 2.3±0.5 | 3.2 | 1.9-3.0 |
| C | 2 | 6.5±4.9 | 50 | 3.0-10 |
| D | 4 | 6.4±5.5 | 15 | 1.4-11 |
| E | 2 | 19±8 | 92 | 13-24 |
| F | 5 | 19±20 | 44 | 3.1-53 |
| H | 5 | 27±13 | 43 | 11-44 |
| J | 4 | 24±4.6 | 31 | 20-30 |
| L | 5 | 47±13 | 63 | 38-70 |
| M | 4 | 55±12 | 74 | 43-70 |
| N | 4 | 30±25 | 70 | 2.9-63 |
| All Facilities | 39 | 25±20 | 32 | 1.4-70 |

## 3.3 pH and Electrical Conductivity (EC)

Both the pH and EC of SC were measured after SC reacted with water for five d at 50°C in the reaction device (Figure 2-4, Section 2.3.1). The liquid to solid ratio was 20 to 1. The pH of SC was alkaline, and ranged from 9.6 to 11.3 with an average of 10.5 (Table 3-3). Sample 2506 from Facility F had the highest pH (11.3), whereas sample 2515 from Facility J exhibited the lowest pH (9.6). The detailed pH results for all SC samples are listed in Appendix B (Table B-1). No evidence was shown that there were statistically significant differences among samples, and the distribution of pH is presented in Figure 3-3.

**Table 3-3: Salt cake pH**

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| B | 4 | 10.8±0.3 | 11.3 | 10.5-11.3 |
| C | 2 | 10.0±0.01 | 10.1 | 10-10 |
| D | 4 | 10.5±0.3 | 11.0 | 10.3-10.9 |
| E | 2 | 10.5±0.2 | 11.9 | 10.4-10.6 |
| F | 5 | 10.8±0.3 | 11.2 | 10.5-11.2 |
| H | 5 | 10.7±0.1 | 10.8 | 10.5-10.9 |
| J | 4 | 10.2±0.6 | 11.1 | 9.6-11.0 |
| L | 5 | 10.8±0.1 | 10.9 | 10.7-11.0 |
| M | 4 | 10.2±0.2 | 10.6 | 10.1-10.5 |
| N | 4 | 10.4±0.2 | 10.8 | 10.2-10.7 |

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| All Facilities | 39 | 10.5±0.4 | 10.7 | 9.6-11.3 |

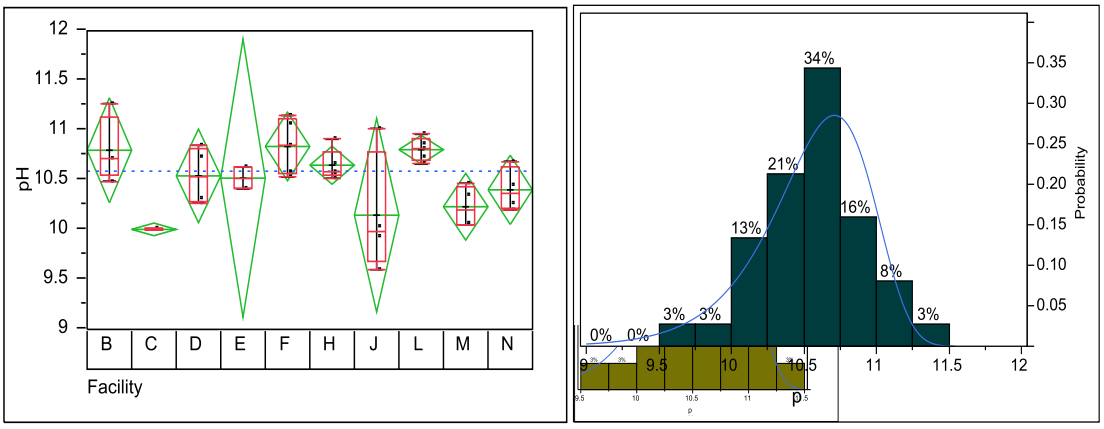



**Figure 3-3: Salt cake pH and its distribution**

The EC of salt cake was relatively high with large variation (Figure 3-4), and largely depended on the sources of sample. The average EC of all samples was 30 mS $cm^{-1}$ (Table 3-4). Sample 2605 from Facility M had the lowest measured EC (18 mS $cm^{-1}$) and Sample 2532 from Facility D had the highest measured EC (47 mS $cm^{-1}$). This is expected because of the addition of salts to the SAP process. The detailed EC results for each sample are listed in Appendix B (Table B-1).

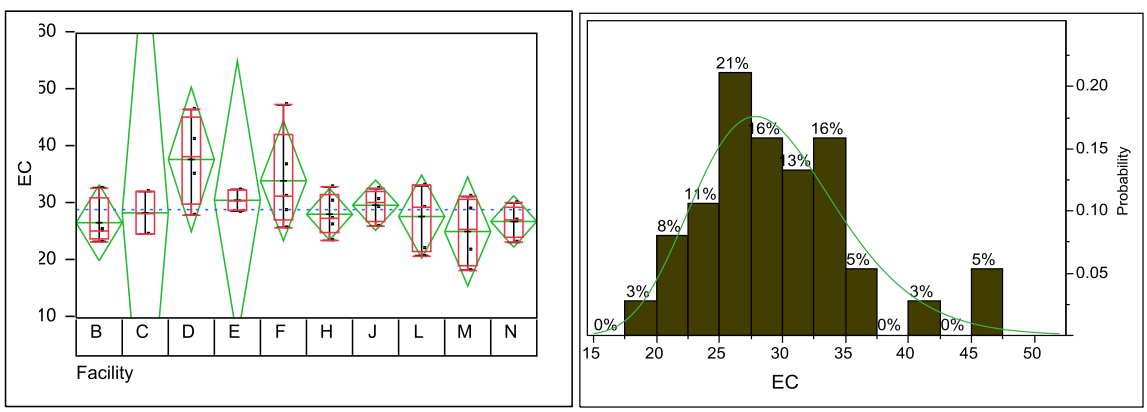



**Figure 3-4: Salt cake EC and its distribution**

**Table 3-4: Salt cake electrical conductivity (EC) (mS $cm^{-1}$)**

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| B | 4 | 27±4.2 | 33 | 23-33 |
| C | 2 | 28±5.3 | 76 | 25-32 |
| D | 4 | 38±8.0 | 51 | 28-47 |
| E | 2 | 31±2.7 | 55 | 29-33 |
| F | 5 | 34±8.5 | 46 | 26-47 |
| H | 5 | 28±3.7 | 33 | 24-33 |

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| J | 4 | 30±2.8 | 34 | 26-33 |
| L | 5 | 28±5.9 | 35 | 21-33 |
| M | 4 | 25±6.0 | 35 | 18-31 |
| N | 4 | 27±4.2 | 33 | 23-31 |
| All Facilities | 39 | 30±6.2 | 32 | 18-47 |

## 3.4 Total Extractable Metals

The major total extractable metal content of each SC sample investigated is listed in Appendix B (Table B-2) and the trace metals content is presented in Appendix B (Table B-3). For the purpose of this section, the percent composition is calculated based on wet weight of the samples.

### 3.4.1 Aluminum (Al) Content

The average content of total Al in the SC was approximately 14%, and ranged from 6.7 to 42% as presented in Table 3-5, which was lower than the previously reported (26 to 47%) [16, 45-47, 87]. The overall distribution of % total Al for the SC samples is presented in Appendix B (Figure B-1). Sample 2609 from Facility M had the highest Al content at 42%, while Sample 2492 from Facility N had the lowest at 6.7% (Table 3-5). Overall, the samples from Facility M had a significantly higher Al content compared to the rest of the sites. Excluding the samples from Facility M, the average total Al content in SC was around 12% lower than the range reported in the literature [16, 20, 46, 87, 88].

**Table 3-5: Extractable Al content in salt cake (%)**

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| B | 4 | 11±2.2 | 14 | 8.5-14 |
| C | 2 | 12±0.93 | 20 | 12-13 |
| D | 4 | 10±1.1 | 12 | 9.2-12 |
| E | 2 | 12±0.21 | 14 | 11-12 |
| F | 5 | 13±2.5 | 16 | 9.2-16 |
| H | 5 | 17±3.1 | 20 | 13-21 |
| J | 4 | 14±3.0 | 19 | 10-17 |
| L | 5 | 13±1.9 | 16 | 12-16 |
| M | 4 | 29±9.7 | 44 | 21-42 |
| N | 4 | 7.8±1.3 | 9.9 | 6.7-9.3 |
| All Facilities | 39 | 14±6.6 | 16 | 6.7-42 |

### 3.4.2 Major Metals Content

In addition to Al, several other metals were detected in SC samples in relatively high concentrations and were denoted "major metals" in the current study. These metals are calcium (Ca), magnesium (Mg), iron (Fe), potassium (K), and sodium (Na) and their concentrations are presented in Table 3-6.

**Table 3-6: Major metals content in salt cake (%)**

| Metal | Mean | Median | $UCL_{95}$ | Range |
|---|---|---|---|---|
| Ca | 1.0±0.53 | 1.0 | 1.2 | 0.32-2.4 |
| Mg | 2.0±1.2 | 1.8 | 2.4 | 0.05-4.1 |
| Fe | 0.58±0.43 | 0.44 | 0.72 | 0.15-2.4 |
| K | 5.8±2.4 | 6.6 | 11 | 2.7-13 |
| Na | 10±2.1 | 10 | 11 | 5.6-17 |

The average calcium content in the sampled SC was approximately 1.0%, and ranged from 0.32 to 2.40% (Table 3-6). The overall distribution of % Ca for the SC samples is presented in Appendix B (Figure B-2). Facilities D and N exhibited low Ca content, while Facilities E and C showed higher values (Appendix B, Tables B-4). Previous research reported Ca content in SC within a range from 0.63 to 1.23% [16, 20, 45-47, 87].

The magnesium content in SC ranged from 0.05 to 4.1%, with an average of 2.0% (Table 3-6). The overall distribution of % Mg for the SC samples is presented in Appendix B (Figure B-3). The Mg content of samples from Facilities N and H was significantly lower than those from most other facilities (Appendix B, Table B-5). In previously-published research, the total Mg content in SC ranged from 0.7 to 7.6% [16, 20, 45-47, 87].

The average measured iron content in SC was 0.58% and ranged from 0.15 to 2.4% (Table 3-6). The overall distribution of the percent Fe for the SC samples is presented in Appendix B (Figure B-4) and the Fe content from all samples analyzed is presented in Appendix B (Table B-6). With the exception of sites J and N, the Fe content in the SC samples had low levels of variability. In previously-published research, the total Fe content in SC ranged from 0.23 to 5.85% [16, 20, 45-47, 87].

The content of potassium in the studied SC ranged from 2.7 to 12.8%, with an average of 5.8% (Table 3-6). The overall distribution of % K for the SC samples is presented in Appendix B (Figure B-5). Approximately 10% of the SC samples contained K at concentrations greater than 9% and samples from Facility D contained the highest K content while samples from Facility N had the lowest K content (Appendix B, Table B-7). In previously-published research, the total K content in SC ranged from 0.48 to 3.74% [16, 20, 45-47, 87].

The average sodium content was 10% (Table 3-6), following a normal distribution (Appendix B, Figure B-6). SC from Facility M had significantly lower Na content than SC from any other facility (Appendix B, Table B-8). In previously-published documents, the total Na content in the SC ranged from 0.5 to 8.5 % [16, 20, 45-47, 87].

### 3.4.3 Trace Metals Content

The metals found in trace concentrations in SC samples were Arsenic (As), Cadmium (Cd), Chromium (Cr), Copper (Cu), Manganese (Mn), Lead (Pb), Selenium (Se), and Zinc (Zn). The concentrations of the trace metals detected in SC samples are presented in Table 3-7.

**Table 3-7: Trace metal content in salt cake (mg kg$^{-1}$)**

| Metal | Mean | Median | $UCL_{95}$ | Range |
|---|---|---|---|---|
| As | 14±8.8 | 11 | 17 | 2.8-41 |
| Cd | 0.33±0.3 | 0.25 | 0.43 | 0.02-1.1 |
| Cr | 330±190 | 280 | 390 | 78-840 |
| Cu | 880±1,400 | 380 | 1,300 | 35-6,200 |
| Mn | 1,400±1,000 | 1,200 | 1,700 | 130-5,300 |
| Pb | 43±58 | 21 | 62 | 4.6-310 |
| Se | 9.9±7.1 | 8.5 | 12 | 1.3-30 |
| Zn | 750±620 | 550 | 950 | 110-2,700 |

Arsenic was detected in all 39 SC samples analyzed. On average, the SC samples contained 14 mg kg$^{-1}$ As, with a range of 2.8 to 41 mg kg$^{-1}$, as presented in Table 3-7. Approximately 80% of the SC samples contained As at concentrations less than 20 mg kg$^{-1}$, as presented in Appendix B (Figure B-7). Samples from Facility C and Facility M had the highest As content (22 and 30 mg kg$^{-1}$, respectively), as presented in Appendix B (Table B-9). It has been reported that As can be used as a component of Al alloys [89].

Every SC sample analyzed had cadmium content greater than the MRL. On average, the SC samples contained Cd at 0.33 mg kg$^{-1}$ (Table 3-7). There was a high level of variability in the samples with the Cd concentration ranging from

0.02 to 1.1 mg $kg^{-1}$ (Appendix B, Figure B-8 and Table B-10). Approximately 80% of the SC samples contained Cd at concentrations less than 600 μg $kg^{-1}$. It is known that Cd is used in some Al alloys [89].

Chromium was detected in all SC samples, with an average value of 330 mg $kg^{-1}$ (Table 3-7). There was a measurable level of variability in the samples, with concentrations ranging from 78 to 840 mg $kg^{-1}$ (Appendix B, Figure B-9 and Table B-11). Approximately 85% of the SC samples contained Cr at concentrations less than 500 mg $kg^{-1}$. Cr is known to be a constituent in some Al alloys [89].

The average copper content measured in SC was 880 mg $kg^{-1}$, with a range of 35 to 6,200 mg $kg^{-1}$ (Table 3-7). Twenty percent of the SC samples contained Cu at concentrations greater than 1,000 mg $kg^{-1}$ (Appendix B, Figure B-10). The total Cu content in samples from Facility J was significantly higher than those from all other facilities (Appendix B, Table B-12). In previously-published research, the total Cu content in SC ranged from 50 to 600 mg $kg^{-1}$ [16, 20, 45-47, 87]. Cu also can be a constituent in Al alloys [89].

Manganese was detected in all SC samples, with an average concentration of 1,400 mg $kg^{-1}$ as presented in Table 3-7. Measured concentrations ranged from 130 to 5,300 mg $kg^{-1}$ (Table 3-7). Approximately 75% of the SC samples contained Mn at concentrations less than 2,000 mg $kg^{-1}$ (Appendix B, Figure B-11). Only the samples from Facility N had significantly lower concentration of Mn (Appendix B, Table B-13). Previously-published research shows that the total Mn content in the SC samples ranged from 47 to 1,400 mg $kg^{-1}$ [16, 20, 45-47, 87].

Lead was detected in all SC samples analyzed. The average Pb content measured was 43 mg $kg^{-1}$ and ranged from 4.6 to 310 mg $kg^{-1}$ (Table 3-7). Approximately 10% of the SC samples contained Pb at concentrations greater than 100 mg $kg^{-1}$; most of these samples were from Facility J (Appendix B, Figure B-12 and Table B-14). In previously-published research, the total Pb content in SC ranged from 190 to 1,000 mg $kg^{-1}$ [16, 20, 45-47, 87]. Pb can be a constituent in Al alloys [89].

Each SC sample had detectable levels of Se, with an average of 9.9 mg $kg^{-1}$ and a range of 1.3 to 30 mg $kg^{-1}$ (Table 3-7). Around 80% of the SC samples contained Se at concentrations less than 15 mg $kg^{-1}$ (Appendix B, Figure B-13). The concentrations of Se in samples of each facility are presented in Appendix B (Table B-15). Hagelstein reported the observation of Se content in SC from 0.18 up to 156 mg $kg^{-1}$ [90]. Se can be used a constituent in Al alloys [89].

Zinc was detected in all SC samples. The average Zn content measured was 750 mg $kg^{-1}$ with a range of 110 to 2,700 mg $kg^{-1}$ (Table 3-7). Approximately 80% of the SC samples contained Zn at concentrations less than 1,000 mg $kg^{-1}$ (Appendix B, Figure B-14). Similar to Cu and Pb, the content of Zn from Facility J was much higher than that from all other facilities (Appendix B, Table B-16). In previously published research, the total Zn content in the SC ranged from 120 to 3,000 mg $kg^{-1}$ [16, 20, 45-47, 87]. Zn can be used a constituent in Al alloys [89].

Although the trace metals content had a high degree of variability, there were correlations between some metals and the Al content (Appendix B, Table B-17). For example, the As content showed positive correlation with the total Al content in the SC samples analyzed. Both correlation coefficients of Pearson product moment and Spearman rank order were statistically significant ($p < 0.01$). Similar observations can be made for the other positively correlated metals (Cd, Cr, Cu, Pb, Mn, Se and Zn) as presented in Appendix B (Table B-17). This may indicate that the source of these metals in the SC is most likely Al alloys and not as impurities with the recycling.

## 3.5 Leachable Elements from Salt Cake

Leachable element content was measured following the reaction of SC with water (Liquid to Solid (L/S) ratio was 20:1). After filtration through a 0.45-μm PVDF filter, the anions ($F^-$, $Cl^-$, $SO_4^{2-}$) were analyzed using IC with appropriate dilution (no acidification) [75], and acidified samples were analyzed for Al, Ca, Cr, Cu, Fe, Mg, Mn, K, Se, Na, and Zn by ICP-AES [73] and trace metals (As, Cr, Pb, and Se) were measured by AA [74]. Because of its low total concentration, Cd was not analyzed for. For the purpose of this section, the leachability of an element is defined as the percentage of the total leachable content divided by the total concentration of the element in the SC.

$$\text{Metal Leachability} = \frac{\text{Leachable metal concentration}}{\text{Total metal concentration}} \times 100\% \quad (3\text{-}1)$$

### 3.5.1 Leachable Aluminum (Al)

Al was detected in all leachate samples analyzed (MRL = 0.095 mg $L^{-1}$). The average concentration of leachable Al was 41 mg $L^{-1}$, and ranged from 2.2 to 150 mg $L^{-1}$ as presented in Table 3-8.

Approximately 75% of the SC samples exhibited a leachable Al concentration greater than 25 mg $L^{-1}$ as shown in Appendix B (Figure B-15). The overall leachability of Al was 0.6% with approximately 80% of all SC samples leaching less than 1% of the total Al in the sample. A significantly positive correlation between leachable Al and pH of the SC was observed and the coefficient of Pearson product moment and Spearman rank order were 0.784 and 0.813 ($p < 0.001$), respectively (Appendix B, Figure B-16).

### 3.5.2 Leachable Major Metals

Ca was detected in all 39 leachate samples analyzed. The average leachable Ca concentration was 65 mg $L^{-1}$, and ranged from 0.33 to 400 mg $L^{-1}$ (Table 3-9). Most of the leachate samples had a Ca concentration less than 80 mg $L^{-1}$ (Appendix B, Figure B-18). A high level of variability of Ca leachability in the SC was found, with a range from 0.13 to 67% (Appendix B, Table B-18).

**Table 3-8: Leachable aluminum (mg $L^{-1}$)**

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| B | 4 | 68±57 | 160 | 25-150 |
| C | 2 | 14±2.5 | 37 | 13-16 |
| D | 4 | 24±11 | 41 | 13-35 |
| E | 2 | 31±14 | 150 | 22-41 |
| F | 5 | 66±36 | 110 | 27-110 |
| H | 5 | 48±9.3 | 65 | 38-61 |
| J | 4 | 32±45 | 100 | 2.2-99 |
| L | 5 | 42±8.3 | 52 | 32-52 |
| M | 4 | 23±9.3 | 46 | 13-31 |
| N | 4 | 29±14 | 51 | 20-49 |
| All Facilities | 39 | 41±30 | 51 | 2.2-150 |

**Table 3-9: Major metal leachability from salt cake**

| Metal | Concentration mg $L^{-1}$ | | Percent Leached (%) | | | |
|---|---|---|---|---|---|---|
| | Mean | Range | Mean | Median | $UCL_{95}$ | Range |
| Ca | 65±100 | 0.33-400 | 11±15 | 2.5 | 16 | 0.13-67 |
| Mg | 0.10±0.33 | 0.001-2 | 0.01±0.03 | 0.002 | 0.02 | 0-0.14 |
| K | 2,500±1,000 | 1,100-5,600 | 87±6.5 | 87 | 89 | 70-100 |
| Na | 2,700±400 | 1,700-3,500 | 54±7.4 | 53 | 56 | 42-82 |

Eleven of the 39 leachate samples analyzed contained Mg at a level above the MRL. The average leachable Mg concentration was 100 µg $L^{-1}$, with a range between 10 and 2,000 µg $L^{-1}$ (Table 3-9). The concentrations of Mg in the leachate of SC from each facility are presented in Appendix B (Table B-19). Though the variability of Mg concentration in leachate was quite high, the leachability of Mg was fairly uniform compared with the other elements – all samples had low leachability (0 to 0.14%) (Table 3-9). Approximately 90% of the samples had leachability of less than 0.03% (Appendix B, Figure B-19), suggesting that Mg minerals present in SC were not readily soluble in water.

Only one leachate sample from Facility N was found to leach detectable levels of Fe, at 0.28 µg $L^{-1}$. Therefore, the

leachability of Fe was very low (less than 0.04%). Detectable concentrations of K were measured in all 39 leachate samples. The average leachable K concentration was 2,500 mg $L^{-1}$, with a minimum of 1,100 mg $L^{-1}$ and a maximum of 5,600 mg $L^{-1}$ (Table 3-9). The concentration of K in the leachate of SC from each facility is presented in Appendix B (Table B-20). Unlike Mg and Al, the leachability of K from SC was relatively high (from 70 to 100%) and uniform across all samples (Table 3-9 and Appendix B, Figure B-19).

Similar to K, detectable concentrations of Na were measured in all 39 leachate samples. The average leachable Na concentration was 2,700 mg $L^{-1}$, with a range from 1,700 to 3,500 mg $L^{-1}$ (Table 3-9). The concentration of Na in the leachate of SC from each facility is presented in Appendix B (Table B-21). The average leachability of Na in SC was 54% and ranged from 42 to 82% (Appendix B, Figure B-20).

### 3.5.3 Leachable Trace Metals

The average leachable As concentration was 2.0 μg $L^{-1}$, and ranged between 1.8 and 3.6 μg $L^{-1}$ (Table 3-10) (concentrations < MRL = MRL or 1.8 μg $L^{-1}$). A total of seven of the 39 leachate samples analyzed had As levels greater than the MRL, which were from Facilities B, E, H and J (Appendix B, Table B-22). The overall leachability of As in SC was low, 0.4%, with a range of 0.09 to 1.3% (Appendix B, Figure B-21 and Table 3-10).

Eleven of the 39 leachate samples analyzed were above the MRL for Cr. The average leachable Cr concentration was 1.5 μg $L^{-1}$, with a range between 0.75 and 4.6 μg $L^{-1}$ (Table 3-10). The concentration of Cr in the leachate of SC from each facility is presented in Appendix B (Table B-23). The mean overall leachability of Cr in SC was very low, 0.01%, with a range from 0.002 to 0.09% (Table 3-10 and Appendix B, Figure B-22).

**Table 3-10: Trace metal concentration in leachate of salt cake (μg $L^{-1}$)**

| Metal | Leached Concentration (μg $L^{-1}$) | | | | Percent Leached (%) | | | |
|---|---|---|---|---|---|---|---|---|
| | Mean | Median | $UCL_{95}$ | Range | Mean | Median | $UCL_{95}$ | Range |
| As | 2.0±0.5 | 1.8 | 2.2 | 1.8-3.6 | 0.41±0.27 | 0.4 | 0.5 | 0.09-1.3 |
| Cr | 1.5±2.3 | 0.75 | 2.2 | 0.75-4.6 | 0.01±0.02 | 0.01 | 0.02 | 0.002-0.09 |
| Cu | 73±220 | 7.1 | 150 | 7.1-1000 | 0.18±0.26 | 0.07 | 0.3 | 0.01-1.2 |
| Mn | 19±1.6 | 19 | 20 | 16-25 | 0.06±0.07 | 0.03 | 0.08 | 0.01-0.32 |
| Pb | 3.3±2.0 | 2.5 | 3.9 | 2.5-10 | 0.50±0.42 | 0.4 | 0.6 | 0.02-1.6 |
| Se | 24±40 | 10 | 38 | 10-190 | 5.3±4.8 | 4 | 7 | 0.8-21 |
| Zn | 26±43 | 5.2 | 40 | 5.2-200 | 0.08±0.13 | 0.03 | 0.1 | 0.001-0.8 |

Sixteen of the 39 leachate samples analyzed were above the MRL for Cu, and some detected concentrations were elevated, up to 1,000 μg $L^{-1}$. The average leachable Cu concentration was 73 μg $L^{-1}$, with a range from 7.1 to 1000 μg $L^{-1}$ (Table 3-10). The concentration of Cu in the leachate of SC from each facility is presented in Appendix B (Table B-24). The overall leachability of Cu in SC was 0.18%, ranging from 0.01 to 1.2% (Appendix B, Table B-24 and Figure B-23).

Mn was detected in all 39 leachate samples analyzed (MRL = 14 μg $L^{-1}$). The concentration of Mn in leachate fell in a relatively narrow range, from 16 to 25 μg $L^{-1}$ (Table 3-10). The concentration of Mn in the leachate of SC from each facility is presented in Appendix B (Table B-25). The average leachability of Mn in SC was also relatively low (0.06%) (Table 3-10) and 70% of the samples had a leachability of less than 0.05% (Appendix B, Figure B-25).

Seven of the 39 leachate samples analyzed were above the MRL for Pb. The average leachable Pb concentration was 3.3 μg $L^{-1}$, with a range between 2.5 and 10 μg $L^{-1}$ (Table 3-10). The concentration of Pb in the leachate of SC from each facility is presented in Appendix B (Table B-26). The overall leachability of Pb in SC was low, 0.5%, with a range from 0.02 to 1.6% (Table 3-10). Detectable concentrations of Pb were found in samples from a relatively low number of facilities (Appendix B, Table B-26).

Only 11 of the 39 leachate samples analyzed were above the MRL for Se, but detected concentrations were as high as 190 μg $L^{-1}$. The average leachable Se concentration was 24 μg $L^{-1}$ (Table 3-10). The corresponding leachability ranged

from 0.8 to 21%, with an average of 5.3% (Table 3-10). Approximately 10% of the samples had greater than 12% leachability (Appendix B, Figure B-26), which was high compared with the other trace metals. The concentration of Se in leachate and its overall leachability was facility-specific. For example, no Se was detected from Facilities C, D, E, M, and N, whereas high leachability of Se was found for Facility B (37-190 μg $L^{-1}$) (Appendix B, Table B-27).

Eighteen of the 39 leachate samples analyzed contained Zn at a level above the MRL. The average leachable Zn concentration was 26 μg $L^{-1}$, with a range between 5.2 and 200 μg $L^{-1}$ (Table 3-10). The concentration of Zn in the leachate of SC from each facility is presented in Appendix B (Table B-28). Similar to Mn, the leachability of Zn in SC was low (from 0 to 0.8%) (Table 3-10). Approximately 70% of the leachable Zn in the SC comprised less than 0.1% of the total Zn mass (Appendix B, Figure B-27).

## 3.6 Results of TCLP

The toxicity characteristic leaching procedure (TCLP) was also carried out on the SC samples. Of the 39 samples, 14 (35%) were extracted using extraction fluid #2 (1.14% glacial $CH_3CH_2OOH$, pH 2.88) due to their high buffer capacity, whereas the other 25 samples were leached using extraction fluid #1 (1.14% glacial $CH_3CH_2OOH$-NaOH, pH 4.95). In addition to Al, the concentration of seven out of the eight toxicity (TC) metals were measured (As, Se, Ag, Ba, Cr, Cd, and Pb) as presented in Table 3-11. The concentrations of all seven metals (Ag, As, Ba, Cr, Cd, Pb and Se) were consistently below their corresponding TC limit which indicates that SC from SAP are not characteristically hazardous waste in the U.S. Facility specific results are presented in Appendix B (Table B-28 to Table B-36). Although DIW and TCLP extraction tests were performed at the same L/S ratio (20:1), there were no significant correlations between the results obtained from these two methods. The differences between the results obtained by both methods may be attributed to the differences in the extraction pH. In addition, the TCLP utilizes an organic acid (acetic acid) that tends to chelate metals.

**Table 3-11: Metal concentration in the TCLP solution (mg $L^{-1}$)**

| Metal | Mean | Median | $UCL_{95}$ | Range | TC Limit |
|---|---|---|---|---|---|
| Al* | 90±139 | 16 | 135 | 0.95-566 | NA |
| Ag | <MRL | -- | -- | -- | 5 |
| As | 0.03±0.01 | 0.02 | 0.03 | 0.02-0.08 | 5 |
| Ba | 6.8±8.5 | 3.9 | 9.6 | 0.57-45 | 100 |
| Cd | 0.01±0.01 | 0.004 | 0.01 | 0.004- | 1 |
| Cr | 0.06±0.08 | 0.02 | 0.09 | 0.02-0.46 | 5 |
| Pb | 0.32±0.77 | 0.02 | 0.57 | 0.01-3.3 | 5 |
| Se | 0.32±0.77 | 0.04 | 0.10 | 0.02-0.72 | 1 |

*Al is not among the TCLP metals; NA: not applicable.

## 3.7 Crystalline Mineral Phases of Salt Cake

The mineral phases of SC were characterized using XRD. As expected, halite (sodium chloride, NaCl) and sylvite (potassium chloride, KCl) were present in all SC samples. Fluorite ($CaF_2$) was also found in most of the investigated samples. Metallic aluminum (Al), aluminum nitride (AlN) and its oxides ($Al_xO_yN_z$), spinel (magnesium aluminum oxide, $Al_2MgO_4$), elpasolite ($K_2NaAlF_6$), and different forms of aluminum oxides were the dominant Al minerals. However, both aluminum carbide ($Al_3C_4$) and aluminum sulfide ($Al_2S_3$) were not detected. Periclase (MgO), calcium sulfate ($CaSO_4$), calcium carbonate ($CaCO_3$), and quartz ($SiO_2$) were also found in some samples. The details of the mineral phases of each sample, including the peak and pattern lists, are listed in Appendix C. The semi-quantitative results for each mineral are listed in Appendix B (Table B-37 and Table B-38).

The nature of the analytical technique to assess the mineral phase in this study is semi-quantitative – a built-in assumption is that all phases add to 100%. Thus, the results output by the software will not indicate unidentified crystalline or amorphous phases. It is noted that not all minerals in the SC were present in the crystalline phase. The

variability of each mineral and its distribution were further summarized and organized by facility, using JMP 9.0.

### 3.7.1 Non-Aluminum Mineral Phases

KCl is applied in the SAP process [3, 27, 35, 36], and was detected in all the investigated SC samples. The variability of KCl by facility and the overall distribution are presented in Appendix B (Figure B-28); the average KCl abundance by weight was 8.9% with a range from 3 to 19% as presented in Table 3-12. The abundance of KCl in SC by facility is presented in Appendix B (Table B-39). Peterson and Revet reported that the KCl content in two sample drosses were 16.3% and 25.0% [91], while Schmitz reported a typical KCl content of 18 - 20% for a salt slag recycled by special plants in Germany [27].

Significant linear correlations were found between the total K by KCl and $K_2NaAlF_6$ , quantified from XRD vs. total K from acid digestion ($r^2 = 0.80$, n = 39, $p < 0.01$); and the K content by KCl-XRD vs. leachable K content from leaching experiments ($r^2 = 0.77$, n = 38, $p < 0.01$) (Figure 3-5). This further supports the reliability of quantitative results of mineral phase analysis in this work.

NaCl, typically added during the SAP process [3, 27, 35, 36], was detected in all of the investigated SC samples. The overall average abundance of NaCl by weight was 18% with a range from 11 to 30%, as presented in Table 3-12. The abundance of NaCl in SC by facility and the overall distribution and variability of NaCl in SC are presented in Appendix B (Table B-40 and Figure B-29). Peterson and Revet reported that the NaCl content in two investigated sample drosses were 19.1% and 24.4% [91]. Schmitz reported that the content of NaCl was approximately 40 to 50% for typical salt slag recycled by special plants in Germany [27].

**Table 3-12: Non-aluminum mineral phases (%) in salt cake**

| Mineral | Mean±Stdev | Median | $UCL_{95}$ | Range | % detected |
|---|---|---|---|---|---|
| KCl | 8.9±4.0 | 9.0 | 10 | 3-19 | 100 |
| NaCl | 18±3.8 | 18 | 19 | 11-30 | 100 |
| $CaF_2$ | 5.8±2.9 | 6.0 | 6.1 | ND*-12 | 90 |
| $CaSO_4$ | 5.5±1.0 | 5.0 | 5.9 | ND-8.0 | 85 |
| $CaCO_3$ | 1.0±0.4 | 1.0 | 1.3 | ND-2.0 | 31 |
| $SiO_2$ | 1.6±0.9 | 1.0 | 3.6 | ND-4.0 | 85 |
| MgO | 1.6±1.1 | 1.0 | 2 | ND-5.0 | 64 |

*ND: Not detected.

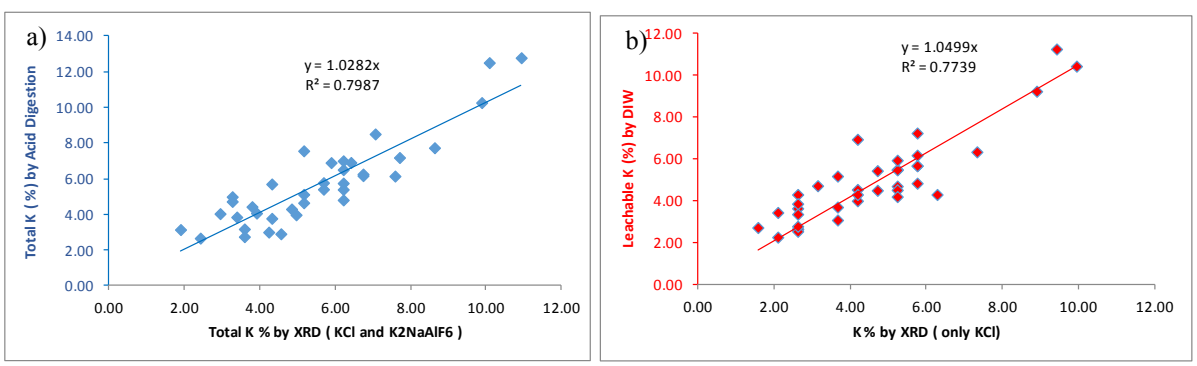



**Figure 3-5: Correlation of potassium by different methods**
(a) Total K [blue]; (b) Leachable K [red]

$CaF_2$ is also traditionally used in the SAP process [3, 27, 35, 36], and was detected in approximately 90% of the

studied SC samples. The average $CaF_2$ abundance by weight was 5.8%, with samples ranging from undetectable to 12%, as presented in Table 3-12. The variability of $CaF_2$ by facility and the overall distribution are presented in Appendix B (Figure B-30). The abundance of fluorite in SC from Facilities D, E, and H were significantly higher than that of Facilities F, M and N (Appendix B, Table B-41). These differences might be related to the technology used for the SAP [33].

In fact, the total salt content (flux), instead of the content of NaCl or KCl, was frequently reported in the literature for SC and was observed to have a fairly wide range, from 20 to 80% [3, 35, 36]. Based on the sum of the NaCl, KCl, and fluorite obtained in this study, the salt content in SC from SAP was about one third of the total weight (31%), with a range from 18 to 50% as presented in Appendix B (Table B-42). The variability of total salt by facility and the overall distribution is presented in Appendix B (Figure B-31).

$CaSO_4$, MgO, $SiO_2$, and $CaCO_3$ were four minor minerals identified in SC (Table 3-12). Not all of these minerals were detected in every SC sample and the detected percentage of these minerals in the samples was 85, 85, 64 and 31%, respectively. The average abundance of $CaSO_4$, MgO, $SiO_2$, and $CaCO_3$ in SC were 5.5, 1.6, 1.6 and 1.0%, respectively, considering only the samples where these minerals were detected. More than half of these samples had less than 2% $SiO_2$ (74%), MgO (65%), and $CaCO_3$ (97%) and less than 6% $CaSO_4$. Previous research reported a MgO content ranging from 0 to 50% with a typical range of 5 to 10%, and an $SiO_2$ content of 0 to 10%, with a typical range of 1 to 2% [38].

## 3.7.2 Aluminum Crystalline Phases

### 3.7.2.1 Metallic Aluminum

Metallic aluminum (Al) is generated from SAP. All SC samples had detectable abundance of metallic aluminum with an average of 3.2% and a range from 1 to 18% (Table 3-13). It is noted that there were significant variations between facilities. The samples from Facility M had a much higher abundance of metallic aluminum than all of the others (Appendix B, Table B-43). In fact, there were also significant differences within samples collected from the same facility. The variability of metallic aluminum by facility and the overall distribution is presented in Appendix B (Figure B-32). Approximately 60% of the samples had a range of 2 to 4% metallic aluminum, 18% of the samples had less than 2% of metallic aluminum, whereas another 18% of the samples contained a range of 4 to 8% metallic aluminum. Only 6% of the samples contained more than 10% metallic aluminum.

**Table 3-13: Aluminum crystalline phases (%) in salt cake**

| Crystalline Phase | Mean | Median | $UCL_{95}$ | Range | % Samples Detected |
|---|---|---|---|---|---|
| Al | 3.2±3.3 | 2.0 | 4.2 | 1.0-18 | 100 |
| Total AlN | 8.0±2.0 | 8.0 | 8.6 | 3.8-13 | 100 |
| AlN | 4.8±2.1 | 4.0 | 5.4 | 1.0-10 | 100 |
| $Al_{2.85}O_{3.45}N_{0.55}$ | 11±2.6 | 11 | 12 | 6.0-17 | 100 |
| $Al_{2.81}O_{3.56}N_{0.44}$ | 7.4±2.0 | 7.0 | 8.0 | 4.0-13 | 100 |
| $Al_{2.78}O_{3.65}N_{0.35}$ | 5.9±1.5 | 6.0 | 6.3 | 3.0-10 | 100 |
| Total $Al_2O_3$ | 42±9.7 | 40 | 45 | 25-68 | 100 |
| $Al_2O_3$ | 7.7±6.0 | 6.0 | 9.4 | ND*-33 | 97 |
| $Al_{2.67}O_4$ | 10±2.7 | 10 | 11 | 5.0-17 | 100 |
| Mg $Al_2O_4$ | 8.6±3.9 | 8.0 | 9.9 | 2.0-23 | 100 |
| $K_2Na$ $AlF_6$ | 3.1±1.3 | 3.0 | 3.5 | 1.0-6.0 | 100 |

*ND: Not detected.

In previously published research, the content of metallic aluminum in the SC was from 0 up to 10% [36, 38], typically 3 to 5% in North America [38]. Peterson and Revet reported that the average metallic aluminum content was 7.8% wet weight for four SC (salty dross) samples [91]. However, crystalline metallic aluminum content has been reported to be relatively low (1.2 to 2.8% using XRD) [45, 46, 92] in samples collected from other countries, e.g. Australia, Brazil, and Russia. There were no correlations between the salt content and metallic aluminum in SC samples (Appendix B, Figure B-33). It is known that both salt content and the ratio of metallic aluminum and salt content play a significant role in the recovery of metallic aluminum in SAP [33, 93].

#### 3.7.2.2 Aluminum Nitride and its Oxides

AlN and its oxides are one type of newly formed minerals resulting from SAP processing, and it also can originate from the SAP source. Oxidation of AlN occurs under 650 to 1,400°C conditions [94, 95], and the presence of three different aluminum oxide nitrides was confirmed in the investigated SC samples. Negative correlations between AlN and its oxides were observed. The Pearson product moment correlation coefficient between AlN, and $Al_{2.85}O_{3.45}N_{0.55}$, $Al_{2.81}O_{3.56}N_{0.44}$, and $Al_{2.78}O_{3.65}N_{0.35}$ was -0.42, -0.44, and -0.44 ($p < 0.01$), respectively. The corresponding Spearman rank order correlation coefficients were -0.45 ($p < 0.01$), -0.36, and -0.35 ($p < 0.05$), respectively.

The average abundance of AlN was 4.8%, with a range from 1 to 10% (Table 3-13). The average abundances of $Al_{2.85}O_{3.45}N_{0.55}$, $Al_{2.81}O_{3.56}N_{0.44}$, and $Al_{2.78}O_{3.65}N_{0.35}$ were 11, 7.4, and 5.9% (Table 3-13), which were equivalent to 1.8, 1.0, and 0.6% of AlN, respectively. The average percentages of AlN, $Al_{2.85}O_{3.45}N_{0.55}$, $Al_{2.81}O_{3.56}N_{0.44}$, and $Al_{2.78}O_{3.65}N_{0.35}$ in total AlN were 58, 22, 12, and 8%, respectively. The AlN oxide ($Al_{2.85}O_{3.45}N_{0.55}$, $Al_{2.81}O_{3.56}N_{0.44}$, and $Al_{2.78}O_{3.65}N_{0.35}$) content in SC by facility and their overall distributions are presented in Appendix B (Tables B-44 to B-47 and Figure B-34). The total AlN abundance in SC by facility and the overall distribution are presented in Appendix B (Table B-48 and Figure B-35). The total AlN abundance in SC was 8.0% with a range of 3.8 to 13% (Table 3-13). Facility H has the highest abundance of total AlN (8.8 to 13%), while Facilities D and M had the lowest abundances (3.5 to 8.4%). In previously-published documents, the AlN content in SC also had high variability, from 0 to 30%, and a typical range of 15 to 30 % [38]. Kashcheev et al. reported an AlN content of 2.11% based on six samples from Russia using XRD technology [92]; Yoshimura et al. reported an AlN content by semi-quantitative XRD as high as 28% [45], and Bruckard and Woodcock reported that AlN content of SC ranged from 1.26 to 4.8%, based on nitrogen content [46].

The relations between AlN and the salt content in SC are complex (Appendix B, Figure B-36). It appears that KCl and fluorite had negative effects on the formation of AlN oxides. The Pearson product moment correlation coefficients of KCl to $Al_{2.85}O_{3.45}N_{0.55}$, $Al_{2.81}O_{3.56}N_{0.44}$, $Al_{2.78}O_{3.65}N_{0.35}$ and total AlN were - 0.38, -0.40, -0.36, and -0.37 ($p < 0.05$), respectively. The corresponding Spearman rank order correlation coefficients were -0.38, -0.41, -0.38 and -0.35 ($p < 0.05$), respectively. The Pearson product moment correlation coefficients between $CaF_2$ to $Al_{2.85}O_{3.45}N_{0.55}$, $Al_{2.81}O_{3.56}N_{0.44}$, and $Al_{2.78}O_{3.65}N_{0.35}$ were -0.42, -0.42, and -0.39 ($p < 0.01$), respectively; while the corresponding Spearman rank order correlation coefficients were -0.41, -0.37, and -0.33 ($p < 0.05$), respectively.

#### 3.7.2.3 Aluminum Oxides

Aluminum oxides are another main type of Al minerals found in SC. It was confirmed that at least $Al_2O_3$ and $Al_{2.67}O_4$ were present in most SC samples. The other aluminum oxides included three forms of AlN oxides. Usually, one or more aluminum oxides were detected in the same SC sample. The average alumina ($Al_2O_3$), $Al_{2.67}O_4$, and total $Al_2O_3$ abundances in SC samples were 7.5%, 10%, and 42%, respectively (Table 3-13). The corresponding variability of aluminum and total aluminum oxide by facility and within the overall distribution is presented in Appendix B (Figures B-37 to B-39 and Tables B-49 to B-51).

Significant variations in quantities of aluminum oxides in SC were found, not only among different facilities, but also among samples within the same facility. For example, alumina content in samples from Facility N was significantly higher than that of the other facilities, whereas samples from Facility D and Facility M contained significantly lower quantities of alumina than that of any other facilities. Previously published research also shows a wide range of aluminum oxide in SC (20 to 60%) [27, 29, 38, 91]. Peterson and Revet also reported that the average aluminum oxide content was 46.10% in four SC (salty dross) samples by weight [91]. Kashcheev et al. reported that the content of

aluminum oxide in SC was 44% based on six samples from Russia, analyzed using XRD [92].

As expected, negative correlations between the aluminum oxides and metallic aluminum were found. The Pearson product moment and Spearman rank order correlation coefficients between Al and total aluminum oxides were -0.46 ($p < 0.05$) and -0.37 ($p < 0.05$), respectively. Meanwhile, negative correlations between the salt content and the aluminum oxides in SC samples were observed (Appendix B, Figures B-40 to B-42). For example, the Pearson product moment and Spearman rank order correlation coefficients between KCl and alumina were -0.37 and -0.39 ($p < 0.05$), respectively. The Pearson product moment and Spearman rank order correlation coefficients of $CaF_2$ and total aluminum oxides were -0.60 and 0.57 ($p < 0.01$), respectively. However, no correlations were found between NaCl and any aluminum oxides. It should be mentioned that there was a positive correlation between KCl and $CaF_2$ in SC samples.

#### 3.7.2.4 Elpasolite and Spinel

Both elpasolite ($K_2NaAlF_6$) and spinel ($MgAl_2O_4$) are byproducts of secondary Al processes, and their presence in Al dross is widely reported in the literature [16, 96, 97]. The average elpasolite abundance in this study was 3.1% with a range from 1 to 6% and the average spinel abundance was 8.6% with a range from 2 to 23%, as presented in Table 3-13. The abundance of $K_2NaAlF_6$ and $MgAl_2O_4$ by facility and the overall distribution are presented in Appendix B (Tables B-52 and B-53 and Figures B-43 and B-44).

Significant positive correlations between the metallic aluminum and elpasolite were found in the SC samples (Appendix B, Figure B-45). The Pearson product moment and Spearman rank order correlation coefficients were 0.85 and 0.74 ($p < 0.01$), respectively. Spinel was also found to be positively correlated with elpasolite. The corresponding Pearson product moment and Spearman rank order correlation coefficients were 0.54 and 0.61 ($p < 0.01$), respectively. However, no significant correlation was found between spinel and metallic aluminum. This may be attributed to the presence of aluminum oxides.

## 3.8 Conclusions

A total of 39 SC samples were evaluated for MC, WHC, pH, EC, total metals, leachable metals, and crystalline phases. Overall, SC samples were highly alkaline with a large degree of inter- and intra- sample variability which could be attributed to variability of the Al feed stock, conditions at the processing facilities, sample processing and analysis. There was a positive correlation between most of the trace metals concentrations and the Al content of the samples which may be attributed to the presence of some of these metals in various Al alloys. Overall, SC samples contained elevated total metal content but none of the SC samples exceeded the toxicity limit for seven of the eight RCRA metals (Hg was not analyzed). While the percent metal leached was relatively low, the leachable metal content may still pose a contamination concern and potential human and ecological exposure if uncontrollably released to the environment. As a result, SC should always be managed at facilities that utilize synthetic liner systems with leachate collection (the salt content of the leachate will increase the hydraulic conductivity of clay liners within a few years of installation). The mineral phase analysis showed that various species of Al are present in the SC samples with a large degree of variability. The relative abundance of various aluminum species was evaluated but it is noted that the method used is a semi-quantitative method and as a result there is a limitation for the data use. The analysis only showed a few aluminum species present in SC which does not exclude the presence of other crystalline species, especially in light of the variability observed in the samples.

# 4. SALT CAKE REACTIVITY

Three key indices were employed to evaluate the impact of reacting SC with water, particularly with regards to the temperature profile of the reaction (the maximum temperature change, $\Delta T_{max}$ (°C) and the time to reach the highest post-reaction temperature, $t_{max}$ (h)), the heat generation, and the volume of gas generation. SC reactivity may be impacted by various factors including but not limited to: 1) environmental temperature, 2) particle size, 3) liquid introduction rate, 4) liquid composition, 5) L/S ratio, and 6) Al speciation in the SC sample. This chapter outlines the impact of these factors on SC reactivity and presents the three key indices for reactivity measured for the 39 SC samples.

## 4.1 Factors Impacting Salt Cake Reactivity

### 4.1.1 Environmental Temperature

Aluminum based reactions are often influenced by environmental temperatures. Research suggests that metallic aluminum reaction with water requires activation [98], whereas aluminum carbide or AlN can react with water and water vapor at room temperatures [53, 99, 100]. Temperature increase as a result of DIW addition to SC samples was examined as a function of time under 20, 37, and 50°C environmental temperatures. As presented in Figure 4-1 (a), for sample 2488-L, the temperature increase was minimal when DIW was added at room temperature indicating low reactivity of the SC sample. However, when the environmental temperature was raised to 37 and 50°C, a significant temperature increase was observed ($\Delta T_{max}$ ~ 30°C and 40°C, respectively) indicating higher degree of sample reactivity. SC sample 2510-F exhibited different temperature profiles as demonstrated in Figure 4-1 (b). This sample demonstrated reactivity at room temperature which resulted in an increase in the intensity of the reaction which resulted in a higher $\Delta T_{max}$ and shorter $t_{max\text{-}T}$.

The differences between the behaviors of the SC samples in response to a change in environmental temperature could be attributed to variation in the speciation of Al present in the SC samples. While the XRD analysis did not detect aluminum carbide in the SC samples above its detection limit, methane presence in the gas generated from the reaction of these samples (as will be presented later) with DIW suggests the presence of some aluminum carbide in sample 2510-M which could react with DIW at room temperature. The increase in temperature provided by the aluminum carbide reaction may then initiate the reaction of other aluminum species present in the sample.

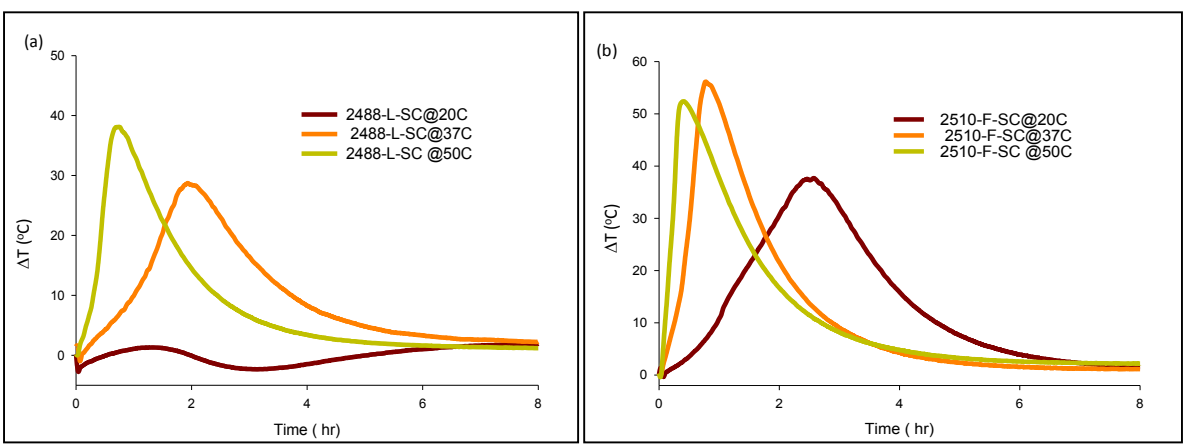



**Figure 4-1: Impact of environmental temperature on maximum temperature change**

While environmental temperature impacts the rate of reaction for SC, it has minimal impact on the heat generation potential (kJ $g^{-1}$). As presented in Table 4-1, the difference in the heat generation potential at two environmental temperatures (37 and 50°C) was not significant. It is noted that there is some variability between heat generation at the

two temperatures, but that could be the result of heterogeneity of the samples as well as the experimental set up and detection.

**Table 4-1: Impact of environmental temperature on heat generation potential**

| Sample ID | Heat (kJ $g^{-1}$) | |
|---|---|---|
| | 37°C | 50°C |
| 2502-F | 1.4 ±0.2 | 1.6±0.3 |
| 2046-H | 1.1±0.2 | 1.2±0.1 |
| 2490-L | 0.81±0.1 | 1.0±0.1 |
| 2613-M | 3.8±0.3 | 4.5±0.6 |

*L/S = 2:1, DIW, 5 g SC, < 0.05 mm, 5 days

Results presented here are of particular importance when trying to understand concerns associated with the disposal of SC in MSW landfills. While the heat generation potential of a given SC sample is not impacted by environmental temperatures, the rate at which the reaction occurs increases with increasing temperatures. MSW decomposition process is exothermic and as a result MSW landfill temperatures are typically greater than 37°C and may reach 50°C or greater [56, 58, 59]. The elevated temperatures present in most MSW landfills can be conducive and could initiate SC reactions that otherwise may not take place. Furthermore, elevated temperatures can increase the rate of the SC reaction which further propagate the reaction as described in Figure 4-2. Even outside of MSW landfills, once SC reaction starts, it may self-propagate and increase the rate at which the reaction occurs.

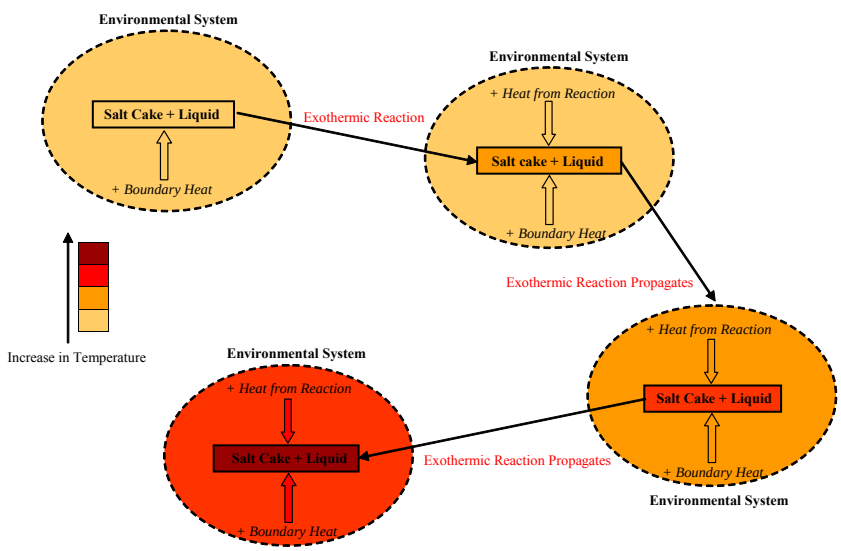



**Figure 4-2: Schematic describing self-propagation of salt cake reaction**

The impact of environmental temperature on gas generation potential was also evaluated. The productivity of $H_2$ and $CH_4$ gases as a function of environmental temperature for randomly selected SC samples is presented in Table 4-2. It is noted that other gases evolve from SC reaction with DIW, but $H_2$ and $CH_4$ are the major ones to be presented in section 4.2.4. The results indicate that, in general, $H_2$ productivity increased with the increase in environmental temperature until 37°C after which no significant impacts were observed. On the other hand, $CH_4$ productivity was minimally impacted by changes in environmental temperatures. The $H_2$ productivity was significantly higher than that of $CH_4$. This may be attributed to the limited amount of aluminum carbide in SC as compared to the relatively large amount of metallic aluminum. Thus, at the elevated temperatures encountered in MSW landfills, elevated levels of $H_2$ gas may be generated as a result of SC reactivity and will be of concern. It is noted that even at 4°C environmental temperature, there was gas productivity from SC despite the minimal increase in $\Delta T_{max}$. This shows that salt can still react, but the reaction rate is so slow that the increase in temperature may not be observed.

### 4.1.2 Particle Size

Particle size of the SC samples may also influence the reactivity of the sample and the temperature change observed. As presented in section 2.3.2.2, the impact of SC particle size on reactivity was evaluated for three sizes. Results presented in Figure 4-3 demonstrated that, in general, the $\Delta T_{max}$ (°C) increases with a decreasing particle size. Furthermore, the time needed to achieve maximum temperature ($t_{max-T}$) decreases with decreasing particle size because of a greater particle reaction surface area (i.e., specific surface area). However, the degree of this effect was sample-specific as presented in Table 4-3. For example, the reaction of SC sample 2512-J resulted in a $\Delta T_{max}$ increase from 9.2°C for the < 2 mm size to 96°C for the fine particle size (< 0.05 mm) as compared to a $\Delta T_{max}$ increase from 51°C for the < 2 mm size to 67°C for the < 0.05 mm for sample 2502-F.

**Table 4-2: Impact of environmental temperature on gas generation potential**

| Sample ID | $H_2$ productivity (ml $g^{-1}$) | | | | $CH_4$ productivity (ml $g^{-1}$) | | | |
|---|---|---|---|---|---|---|---|---|
| | 4°C | 20°C | 37°C | 50°C | 4°C | 20°C | 37°C | 50°C |
| 2512-J | 6.8±0.1 | 12±0.9 | 23 ± 6.6 | 18±0.5 | 1.8±0.1 | 2.3±0.01 | 3.2±0.6 | 2.3±0.02 |
| 2046-H | 0.1±0.0 | 8.0±0.3 | 13 ± 2.5 | 11±0.9 | 0.5±0.08 | 2.5±0.1 | 5.2±1.7 | 4.3±0.02 |
| 2439-E | 24±1.1 | 30±0.7 | 34±1.7 | 32±1.3 | 2.6±0.1 | 3.5±0.2 | 3.9±0.1 | 4.2±0.4 |

*L/S = 20:1, DIW, < 2 mm, 5 days.

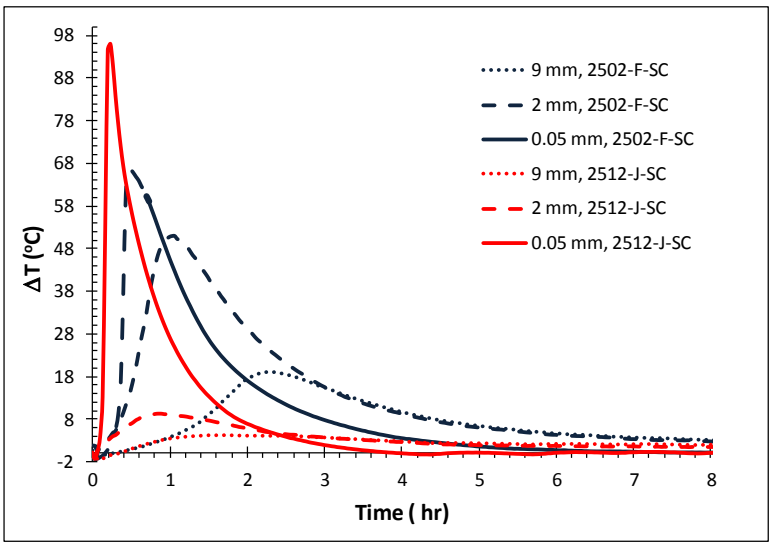



**Figure 4-3: Effect of particle size on salt cake reactivity**

**Table 4-3: Effect of particle size on $\Delta T_{max}$ and $t_{max-T}$**

| Reactivity Indices | Sample ID | Particle size (mm) | | |
|---|---|---|---|---|
| | | 9 | 2 | 0.05 |
| $\Delta T_{max}$ (ºC) | 2502-F | 19 | 51 | 67 |
| | 2512-J | 4.1 | 9.2 | 96 |
| | 2046-H | 12 | 24 | 56 |
| $t_{max}$ | 2502-F | 2.3 | 1.1 | 0.67 |

| | | | |
|---|---|---|---|
| 2512-J | 1.6 | 0.81 | 0.23 |
| 2046-H | 2.2 | 1.3 | 1.6 |

*L/S = 1:1, DIW, 10 g SC, 37 °C, < 2 mm, 5 days.

The decrease in particle size also had an impact on the heat generation and gas generation potentials as presented in Table 4-4 and Table 4-5, respectively. While it is difficult to quantify the sample heterogeneity and its impact on heat and gas generation, there is a clear trend across the samples that SC with smaller particle size generate more heat and more $H_2$ gas (impact on $CH_4$ generation was minimal as presented in section 4.1.1). Larger particles have smaller surface area to volume ratio as compared to smaller particles and thus, higher SC reactivity is expected with the decrease of particle size. From an end-of-life management perspective, data presented here suggest that SC should not be size reduced before disposal. Also, care should be taken not to size reduce it after disposal by compacting it with heavy bulldozers.

**Table 4-4: Effect of particle size on heat generation potential (kJ $g^{-1}$)**

| Sample ID | Particle size (mm) | | |
|---|---|---|---|
| | 9 | 2 | 0.05 |
| 2502-F | NA* | 1.1 | 1.4 |
| 2512-J | NA | 0.31 | 1.5 |
| 2046-H | NA | 0.67 | 1.1 |
| 2490-L | NA | 0.64 | 0.82 |

* L/S = 2:1, 10 ml DIW, 5 g SC, 5 days; NA: Not analyzed due to the instrumentation constraints. The utilized calorimeter does not allow for the 9 mm size.

**Table 4-5: Effect of particle size on salt gas productivity (ml $g^{-1}$)**

| Sample ID* | $H_2$ | | | $CH_4$ | | |
|---|---|---|---|---|---|---|
| | 9 mm | 2 mm | 0.05 mm | 9 mm | 2 mm | 0.05 mm |
| 2046-H | 7.7±3.2 | 13 ± 2.5 | 20 ±1.7 | 6.8±2.0 | 5.2±1.7 | 5.2±2.8 |
| 2512-J | 13±8.2 | 23 ± 6.6 | 42 ±24 | 1.5±1.3 | 3.2±0.6 | 1.4±0.5 |
| 2613-M | 49±10 | 58 ± 9.1 | 62 ±16 | 4.2±3.2 | 1.8±0.5 | 1.0±0.8 |

* L/S = 20:1, DIW, 37°C, 5 days

### 4.1.3 Liquid Addition Rate

In the environment it is likely that liquids would contact SC in an intermittent fashion. To that end, a SC sample (2069-M) received two treatments. The first treatment received 10 ml DIW at once and acted as a control (referred to as 10G-10ML). The second treatment received DIW intermittently over a period of 72 hours and is referred to as 10G-4-3-3ML. The temperature profiles for both treatments (Figure 4-4a) show that within the first hour, the temperature increased by 55°C for both treatments. While the intermittent addition of DIW to the second treatment caused an increase in temperature, that increase in ΔT was minor compared to the initial ΔT increase. It must be noted that while the final liquid to solid ratio for both treatments was the same, the initial ratio was different. The initial liquid to solid ratio of the control treatment was 1:1 while for the intermittent sample it was 1:2.5. Thus, while the temperature increase in both treatments was similar, the heat generated by the control sample (within the first few hours) was higher since the sample contained more liquid.

In a follow up experiment, the final liquid to solid ratio of the reaction was fixed; however, the initial one varied with varying mass of SC (Figure 4-4b). In this case the initial temperature change varied as a function of the mass of SC added. This is most likely the result of increasing mass of potentially reactive aluminum species in the reaction vessel. Again, the largest temperature increase always occurred after the initial liquid addition; the temperature spike due to the secondary watering was relatively minor. Nonetheless, the results of these experiments suggest that while the

largest change in reaction temperature occurs after the first liquid addition, unless enough liquids are added to a SC sample to fully react it, that SC would continue to react every time a new source of liquids becomes available. Since the impact of liquid addition rate was minimal on the temperature generation, no further evaluation of this factor was carried out on heat generation or gas generation potential.

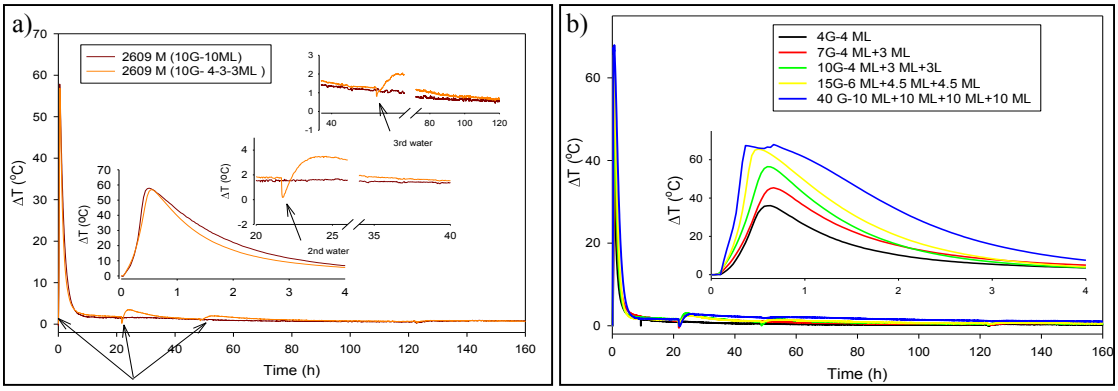



**Figure 4-4: Temperature change as a function of (a) watering schedule and (b) salt cake mass**

### 4.1.4 Liquid Composition

The chemical composition of the liquid that starts the SC reaction may also have an impact on the observed temperature profile. To that end, various solutions were added to the same sample of SC and temperature was measured as a function of time as presented in Table 4-6. It is apparent that the reaction solution had an impact on the magnitude of the reaction. The maximum temperature increase and the faster reactions occurred when SC was reacted with alkaline solutions (0.1M NaOH) or DIW (Table 4-6). On the other hand, reactions of SC with landfill leachate or acidic solutions showed relatively lower temperature increase and slower reactions. For example, a high pH fluid (i.e., 0.1M NaOH) resulted in a $\Delta T_{max}$ of 54°C for sample 2613-M, whereas landfill leachate appeared to diminish the chemical reaction rate and significantly reduce the $\Delta T_{max}$ (13°C) as presented in Figure 4-5. Similar to the impacts on temperature change and reaction intensity, the gas productivity of SC was significantly impacted by the liquid composition (Table 4-7) with highest $H_2$ gas productivity obtained in the cases of alkaline solutions (0.1 M NaOH) and DIW.

**Table 4-6: Impact of liquid composition on $\Delta T_{max}$ and $t_{max\text{-}T}$**

| Liquid | $\Delta T_{max}$ (°C) | | | $t_{max\text{-}T}$ (h) | | |
|---|---|---|---|---|---|---|
| | 2502-F | 2555-H | 2613-M | 2502-F | 2555-H | 2613-M |
| 0.1 M HCl | 32 | 14 | 28 | 2 | 3.6 | 1.5 |
| 0.1 M NaOH | 56 | 20 | 54 | 0.9 | 2.3 | 0.63 |
| DIW | 50 | 19 | 43 | 1.1 | 2.7 | 0.97 |
| Landfill leachate | 12 | 9 | 13 | 3.4 | 3.7 | 2.2 |
| VFA mixture | 5 | 9 | 34 | 7.2 | 4.1 | 1.2 |
| Humic acid salt | 3 | 7 | 15 | 12 | 4.5 | 1.9 |

*L/S = 1:1, 10 ml DIW, 10g SC, < 2 mm, 37°C, 5 days

**Table 4-7: Gas generation potential (ml $g^{-1}$) as a function of liquid composition**

| Liquid | $H_2$ | | | $CH_4$ | | |
|---|---|---|---|---|---|---|
| | 2512-J | 2046-H | 2613-M | 2512-J | 2046-H | 2613-M |
| 0.1 M HCl | 16 | 17 | 79 | 1.7 | 3.5 | 0.8 |
| 0.1 M NaOH | 50 | 19 | 180 | 1.6 | 2.9 | 2.6 |

| | | | | | | |
|---|---|---|---|---|---|---|
| DIW | 39 | 20 | 72 | 1.3 | 3.2 | 1.8 |
| Landfill leachate | 17 | 0.5 | 55 | 2.0 | 1.2 | 0.8 |
| VFA mixture | 38 | 18 | 53 | 1.3 | 3.5 | 0.8 |
| Humic acids salt | 38 | 12 | 59 | 1.7 | 3.5 | 0.9 |

*L/S = 20:1, < 0.05 mm, 50ºC, 5 days

**Table 4-8: Impact of liquid composition on heat generation potential**

| Liquid | Heat (kJ $g^{-1}$) | | |
|---|---|---|---|
| | 2502-F | 2555-H | 2613-M |
| 0.1 M HCl | 1.2 | 1.2 | 2.8 |
| 0.1 M NaOH | 1.4 | 1.6 | 3.8 |
| DIW | 1.6 | 0.95 | 5 |
| Landfill leachate | 1.7 | 0.87 | 5 |
| VFA mixture | 1.2 | 1.1 | 4.5 |

*L/S =2:1, 5 g SC, <0.05 mm, 50ºC, 5 days

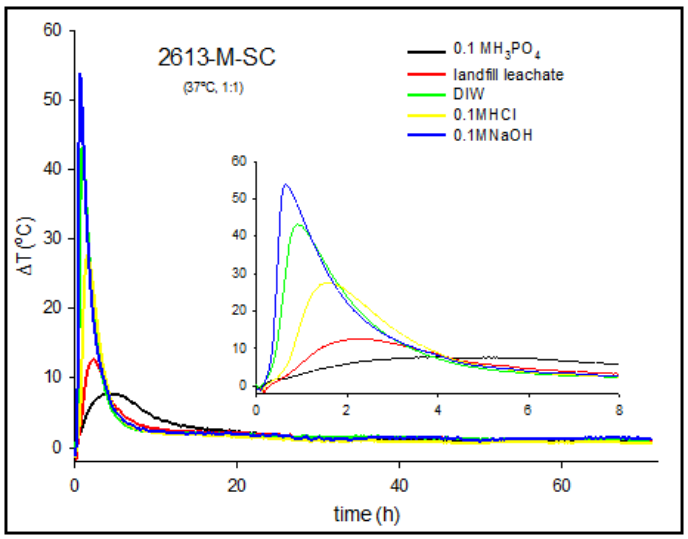



**Figure 4-5: Liquid composition impact on salt cake reactivity**

While the liquids used in this evaluation had varying initial pH values, the final pH for all samples was relatively similar at around 10. Thus, it is unclear to what degree the initial solution pH impacted the temperature increase and the gas productivity. However, the temperature change and gas productivity may be more impacted by the sample composition rather than by the type of liquids added. Furthermore, these results indicate that reactions of SC with landfill leachate would be much more complex than that in the DIW system. Unlike temperature change and gas productivity, the heat generation potential was minimally impacted by changes is solution chemistry as presented in Table 4-8.

### 4.1.5 Liquid to Solid Ratio and Mass of Salt Cake

To evaluate the impact of L/S ratio on the reactivity of SC, various amounts of liquids were added to a SC sample and reacted as presented in section 2.3.2.5. The $\Delta T_{max}$ and $t_{max\text{-}T}$ were measured as a result of varying liquid to solid ratio as presented in Figure 4-6. The decrease observed in the $\Delta T_{max}$ accompanied with an increase in the $t_{max\text{-}T}$ indicates a reduction in the intensity of the reaction. This further implies that at some point the addition of more liquids to the SC

sample would "cool off" the reaction. The relatively small decrease in temperature as the L/S ratio doubled from 0.5 to 1 (as compared to 1:1.5) may indicate that the SC sample did not have enough liquid to completely react at the lower ratios. The drastic decrease in the $\Delta T_{max}$ between the liquid to solid ratio of 1 and 1.5 indicates the "cooling effects" of water on the SC reaction. Assuming a complete reaction, the heat generation potential of a sample should not be impacted by changes to the liquid to solid ratio. Experimental data support such an observation. The heat generation potential for a SC sample at 5:1 L/S ratio (5.2 kJ $g^{-1}$) was similar to that at 10:1 ratio (5.0 kJ $g^{-1}$). Similar results were observed with different liquid compositions as presented in Table 4-9.

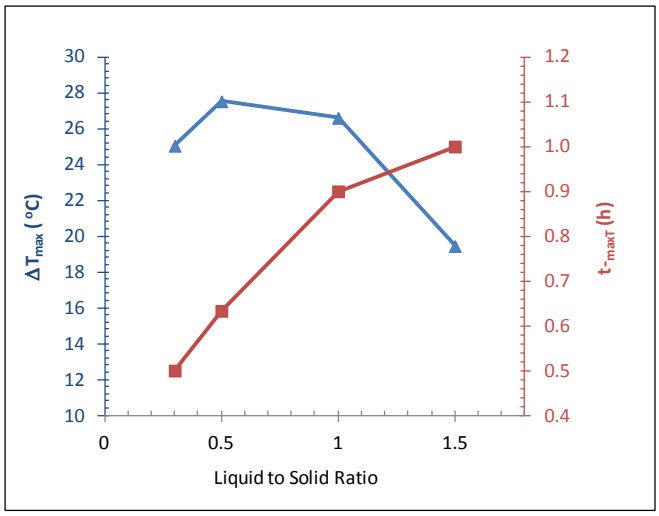



**Figure 4-6: Impact of liquid to solid ratio on temperature change and time to reach maximum temperature**

Varying L/S ratio may also impact gas generation from SC reaction since gases can dissolve in the liquid phase as observed by others [11, 29, 46, 67, 101]. Similar to the temperature profile study, the gas production testing results indicated variability between samples in terms of gas production and gas composition as presented in Figure 4-7 and 4-8, respectively. Liquid to solid ratio impact is mainly driven by the availability of enough liquids to completely react with the SC sample. For example, at an L/S ratio of 5:1, samples 2512-J and 2433-C received enough liquid to completely react and as a result the gas volume or composition did not vary when L/S ratio increased to 20:1. However, for sample 2613-M, the volume of gas continued to increase with increasing L/S ratio. In this case, the volume of liquid added was not large enough to completely react with the SC sample. In general, there were no observed impacts of L/S ratio on the relative abundance of the gas generated by SC samples as presented in Figure 4-8.

The mass of SC is another factor that may impact SC reactivity. At a fixed environmental temperature (50°C) and L/S ratio (1:1), $\Delta T_{max}$ of SC samples 2439-E and 2482-L increased with the increase in SC mass as presented in Figure 4-9a. Furthermore, $\Delta T_{max}$ was a function of SC mass at lower environmental temperature (37°C) or different L/S ratio (1:0.3 and 1:0.5). On the other hand, heat generation potential, expectedly, was minimally impacted by increasing the mass of SC (Figure 4-9b). These results indicate that heat is a better index for evaluating SC reactivity as compared to using temperature change despite the relative simplicity of measuring the latter index. Therefore, the temperature profile in this study, including the two indexes, $\Delta T_{max}$ and $t_{maxT}$, are related to the experimental conditions or "operational definition." The results of the $\Delta T_{max}$ or $t_{maxT}$ only can be comparable under the fixed laboratory conditions, and there are no physical meanings of the absolute values of these indexes if the conditions are unknown. It is not expected that these values or changes in the current laboratory setting are presented in a full-scale landfill after disposal of SC. However, heat is a relatively stable index to describe the reactions of SC in a landfill environment with the relatively complicated procedures in the laboratory.

**Table 4-9: Impact of liquid to solid ratio and leachate composition on heat generation potential**

| | Heat (kJ $g^{-1}$) |
|---|---|

| Liquid | 10:1(10 ml/1g) | 5:1(10 ml/2g) |
|---|---|---|
| 0.1 NaOH | 3.7 | 4.2 |
| DIW | 5 | 5.2 |
| Landfill leachate | 4.5 | 5.3 |
| Volatile fatty acid mixture | 5 | 5.6 |

*2613-M, <0.05 mm, @ 50ºC.

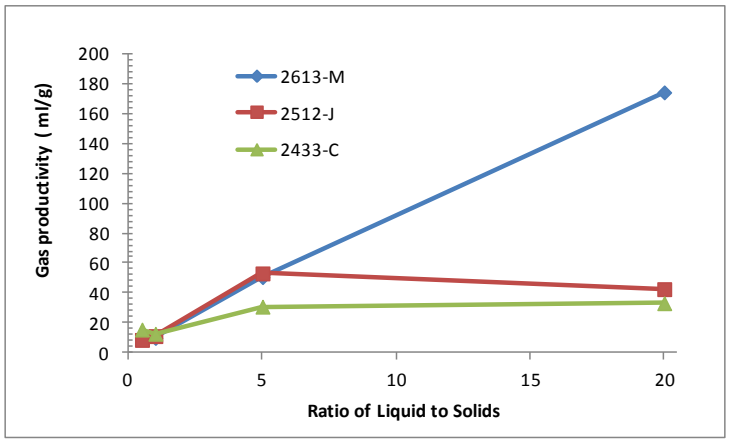



**Figure 4-7: Effect of liquid to solid ratio on gas production of SC reaction**

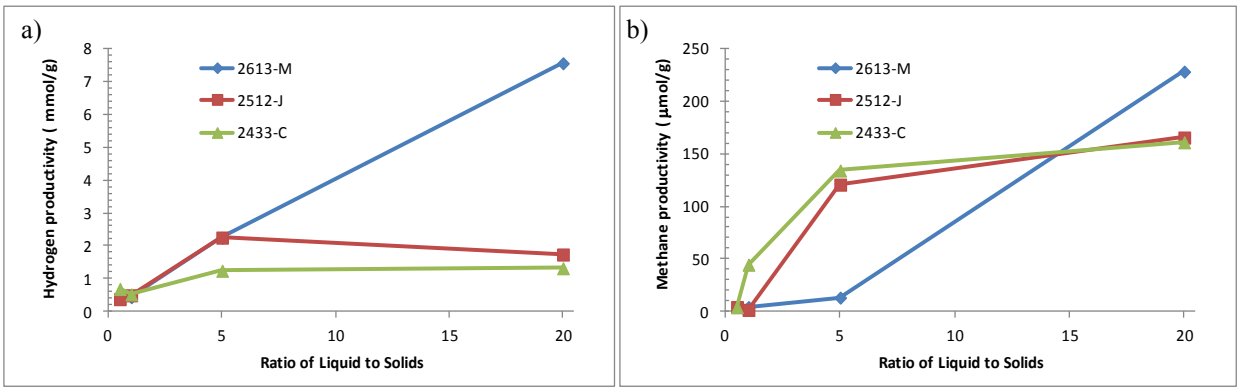



**Figure 4-8: Effect of liquid to solid ratio on gas composition of SC reaction**

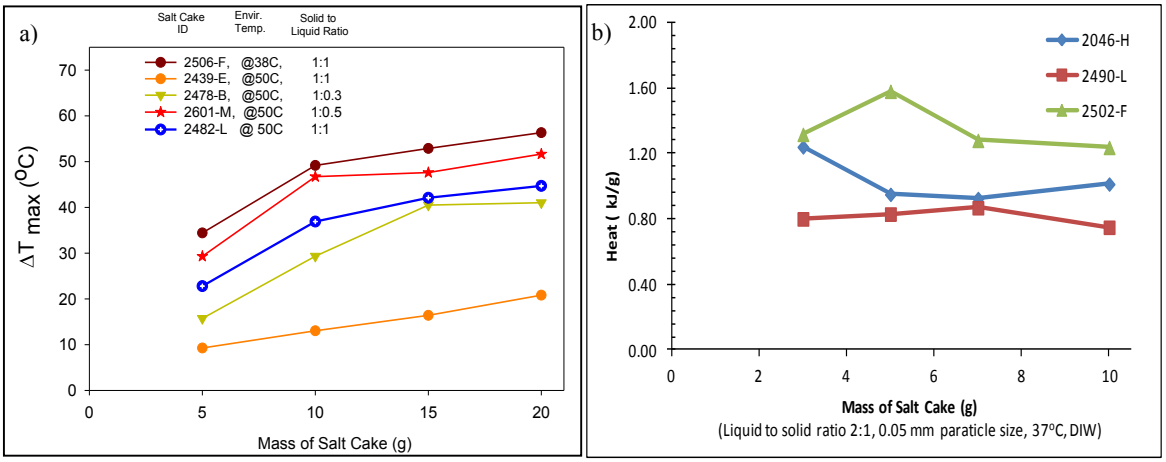



**Figure 4-9: Effect of the mass of salt cake on (a) $\Delta T_{max}$ and (b) heat generation potential**

### 4.1.6 Aluminum Speciation

Since various aluminum species tend to react with liquids at different rates, the relative abundance of various species in SC would impact the temperature generation profile and the reaction intensity. As shown in Table 4-10, the addition of metallic aluminum powder to the theoretical SC mix (mixture I as identified in section 2.3.2.6) had minimal impact on $\Delta T_{max}$ and $t_{max\text{-}T}$. It is noted that some increase in temperature was observed as a result of the addition of metallic aluminum as presented in Figure 4-10a. For example, the temperature of the sample containing 20% metallic aluminum took almost 28 h to return back to background which suggests a low intensity reaction. However, it overall appears that the addition of metallic aluminum powder to the mix has minimal impact on its reactivity with water. Similarly, a temperature increase was observed with the addition of AlN to the theoretical SC and directly correlated with the AlN content (Figure 4-10b). $Al_4C_3$ also reacted with water (in the SC mix) to increase the reaction temperature by approximately 11°C (Figure 4-10c).

The data presented above demonstrates that at concentrations many times higher than those observed in actual SC samples, the individual aluminum species are unable to produce elevated temperatures to explain some of the field data observed at SC disposal sites. Assuming that aluminum species present in the SC are the only species that would react with liquids to generate heat, it is more likely that a mixture of the various species of Al would generate high enough temperatures to explain the field observations. In another words, the reaction temperatures achieved independently by each aluminum species are not additive which further suggests a synergetic effect between aluminum species. For example, higher temperatures due to synergetic effects would be expected by the reactivity of a SC simulator containing 10% metallic aluminum and 10% AlN ($\Delta T_{max}$ = 59°C) or 20% metallic aluminum and 5% $Al_4C_3$ ($\Delta T_{max}$ = 32°C) than by merely adding $\Delta T_{max}$ for each one of those species (3 + 19 = 22°C, or 3 + 11= 14°C, based on the data presented in Table 4-10).

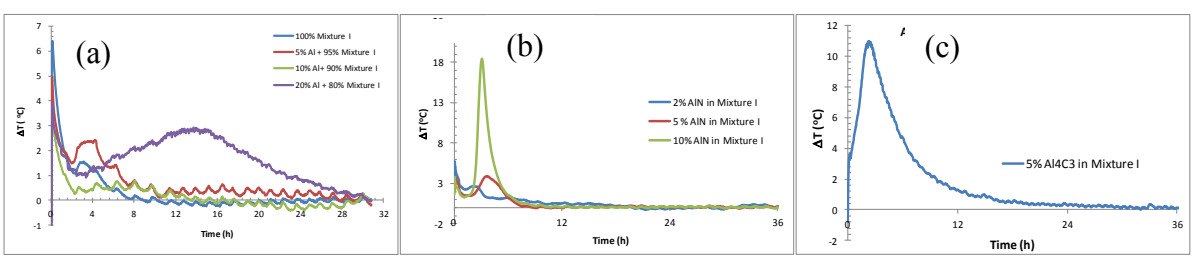



**Figure 4-10: Temperature increase after the addition of (a) Al (b) AlN (c) $Al_4C_3$ in SC stimulators**

**Table 4-10: Impact of aluminum speciation on temperature change in SC simulators**

| Aluminum speciation | Al (%) | AlN (%) | $Al_4C_3$ (%) | Mix (%) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (hrs) |
|---|---|---|---|---|---|---|
| Al | 5 | 0 | 0 | 95 | 2.9 | 0.13 |
| | 10 | 0 | 0 | 90 | 3.0 | 0.13 |
| | 20 | 0 | 0 | 80 | 2.9 | 0.17 |
| AlN | 0 | 2 | 0 | 98 | 3.9 | 0.03 |
| | 0 | 5 | 0 | 95 | 6.3 | 3.9 |
| | 0 | 10 | 0 | 90 | 19 | 3.1 |
| $Al_4C_3$ | 0 | 0 | 5 | 95 | 12 | 2.6 |
| Al/AlN | 5 | 1 | 0 | 94 | 13 | 5.2 |
| | 5 | 2 | 0 | 93 | 15 | 5.1 |
| | 10 | 2 | 0 | 88 | 33 | 2.6 |
| | 10 | 10 | 0 | 80 | 59 | 2.5 |
| | 20 | 1 | 0 | 79 | 30 | 3.2 |
| | 20 | 2 | 0 | 78 | 49 | 1.8 |
| Al/$Al_4C_3$ | 5 | 0 | 5 | 90 | 14 | 2.5 |
| | 10 | 0 | 5 | 85 | 19 | 2.8 |
| | 20 | 0 | 5 | 75 | 32 | 2.5 |

Upon the addition of AlN and metallic aluminum to the mixture I SC simulators, the reaction temperature significantly increased above that observed for similar individual concentrations of AlN and Al (Table 4-10). For example, the $\Delta T_{max}$ measured for a SC simulator containing 10% metallic aluminum and 2% AlN was four times more than that generated by each individual species (Table 4-10). The temperature difference might be attributed to the interaction of

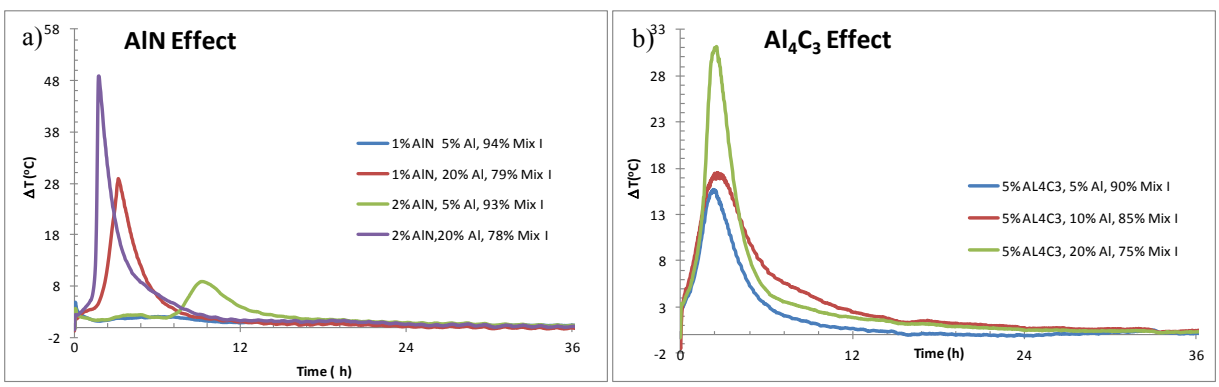



**Figure 4-11: The impact of the addition of AlN and $Al_4C_3$ in SC simulators on the temperature changes**

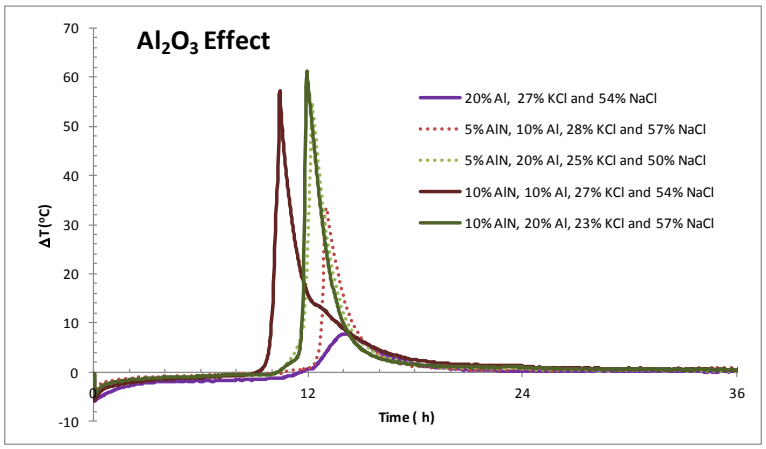



**Figure 4-12: The role of $Al_2O_3$ in SC simulators on the temperature changes**

**Table 4-11: Impact of aluminum speciation in SC simulators on the heat generation**

| Aluminum speciation | Al (g) | AlN (g) | $Al_4C_3$ (g) | Total Heat (kJ) | |
|---|---|---|---|---|---|
| | | | | 37°C | 50°C |
| Al | 1 | 0 | 0 | ND | ND |
| AlN | 0 | 0.5 | 0 | 2.5 | 2.5 |
| | 0 | 1 | 0 | 5.5 | 3.2 |
| | 0 | 2 | 0 | 12 | 9.4 |
| | 0 | 3 | 0 | 17 | 17 |
| $Al_4C_3$ | 0 | 0 | 1.0 | 9.0 | 15.6 |
| Al/AlN | 0.5 | 0.2 | 0 | 8.1 | 11 |
| | 0.5 | 0.5 | 0 | 11 | 14 |
| | 1.0 | 0.2 | 0 | 16 | 20 |
| | 1.0 | 0.5 | 0 | 18 | 24 |
| | 2.0 | 0.2 | 0 | 28 | 37 |
| | 2.0 | 0.5 | 0 | 32 | 39 |
| Al/$Al_4C_3$ | 0.5 | 0 | 0.2 | ND | 9.8 |
| | 1.0 | 0 | 0.2 | ND | 20 |

*ND: not detected

AlN with metallic aluminum. Furthermore, as presented in Figure 4-11, the addition of various species also tends to increase the rate of the reaction by reducing the time it takes to achieve the maximum temperature. A similar synergetic effect on the reaction of aluminum with water was also observed in the presence of aluminum carbide (Figure 4-11).

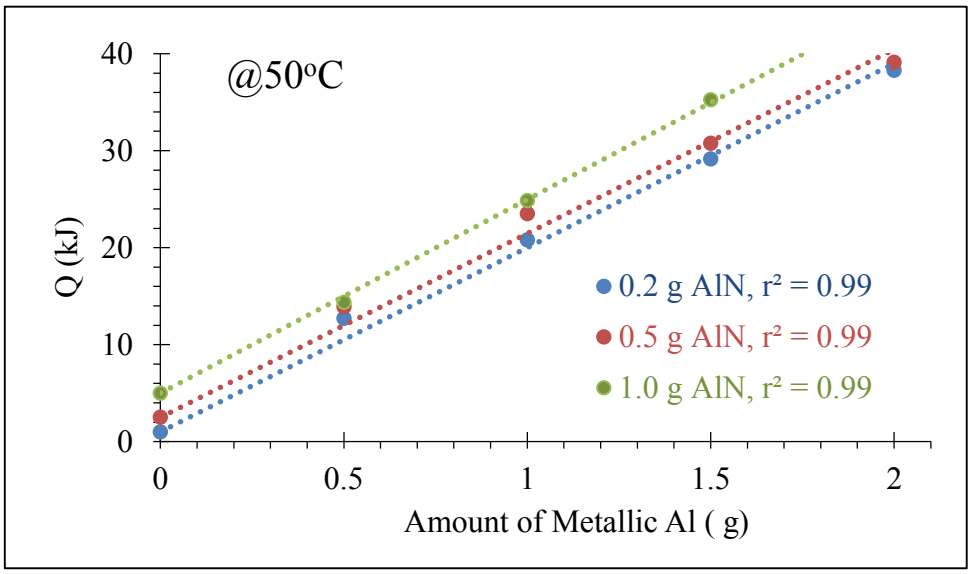



**Figure 4-13: The role of AlN in SC simulators on heat generation potential at 50 °C**

The synergetic effects of Al species in SC samples are rather complicated. For example, the exclusion of $Al_2O_3$ from the mixture containing 10% AlN and 20% metallic Al resulted in an increase in $t_{max\text{-}T}$ by ~ 12 h as presented in Figure 4-12. It is noted that $Al_2O_3$ is a relatively non-reactive Al species. Similar to temperature change, Al species had significant impacts on the heat generation potential from SC. The data presented in Table 4-11 indicate that mixture of Al/AlN or Al/$Al_4C_3$ generated significantly higher heat as compared to that of individual Al species which is indicative of "synergetic effects" as previously concluded with the temperature increase data. Additionally, at the same AlN content, the heat generation potential increased with the increase in Al content following a linear relationship (Figure 4-13). When the AlN content was increased from 0.2 to 1.0 g, similar relationship was observed with minimal impact on the value of heat generated (Figure 4-13). In other words, at the same Al content, the difference in heat generation potential was minimal when the AlN varied between 0.2 and 1.0 g (Table 4-11). This indicates that the presence of AlN in SC is necessary for the reaction to generate measurable heat; however, the magnitude of heat generation is not linearly correlated to the amount of AlN.

## 4.2 Reactivity Indices

### 4.2.1 Maximum Temperature Change ($\Delta T_{max}$)

SC samples were reacted as outlined in section 2.3.3 and the reaction temperature was monitored for 72 h. A temperature increase above background was observed in all 39 SC samples analyzed (at 37 and 50°C environmental temperature conditions). On average, $\Delta T_{max}$ at 37°C and 50°C were 22 and 29°C, and ranged from 3.4 to 64 and 4.4 to 56°C, respectively, as presented in Table 4-12. The details of the temperature profile investigation results are provided in Appendix D.

Based on the ANOVA test with the facility and environmental temperature, the $\Delta T_{max}$ at the 50°C is significantly higher than that at 37°C ($p < 0.01$). This supports the earlier conclusion that environmental temperatures can impact the reaction rate of SC samples. For instance, at an environmental temperature of 37°C, approximately 80% of the samples exhibited a $\Delta T_{max} < 25$°C; while at an environmental temperature of 50°C, approximately 50% of the samples exhibited a $\Delta T_{max} < 25$°C. These results suggest that environmental temperature has significant implications for the disposal of SC in MSW landfills. Depending on their design and capacity, MSW landfills tend to be well insulated and operate at elevated temperatures (compared to ambient conditions) and as a result, the reaction of SC with liquids

tends to become more problematic. The elevated temperatures combined with the leachate in MSW landfills may initiate the SC reaction with liquids and the insulating properties of the MSW can further intensify the reaction.

**Table 4-12: $\Delta T_{max}$ (°C) for salt cake reaction by facility**

| Facility | Sample number | 37°C | | | 50°C | | |
|---|---|---|---|---|---|---|---|
| | | Mean | $UCL_{95}$ | Range | Mean | $UCL_{95}$ | Range |
| B | 4 | 11±2.7 | 16 | 8.5-15 | 18±6.9 | 29 | 13-28 |
| C | 2 | 6.7±4.7 | 49 | 3.4-10 | 5.7±1.8 | 22 | 4.4-6.9 |
| D | 4 | 12±1.7 | 15 | 10-14 | 18±1.5 | 21 | 17-20 |
| E | 2 | 16±0.1 | 17 | 16-16 | 23±0.4 | 27 | 23-23 |
| F | 5 | 50±6.4 | 57 | 39-56 | 48±2.7 | 52 | 45-52 |
| H | 5 | 18±3.8 | 23 | 14-24 | 40±9.0 | 51 | 34-56 |
| J | 4 | 8.0±1.1 | 9.7 | 6.6-9.2 | 15±5.8 | 24 | 9.4-23 |
| L | 5 | 29±11 | 43 | 15-44 | 37±9.8 | 49 | 28-51 |
| M | 4 | 44±18 | 72 | 20-64 | 42±15 | 66 | 22-56 |
| N | 4 | 8.3±2.7 | 13 | 6.7-12 | 14±5.1 | 22 | 7.0-18 |
| All Facilities | 39 | 22±17 | 28 | 3.4-64 | 29±16 | 34 | 4.4-56 |

### 4.2.2 Time to Reach the Maximum Temperature ($t_{max-T}$)

While evaluation of $\Delta T_{max}$ can provide information with regards to the reactivity of the SC samples, the time it takes a sample to reach the maximum temperature ($t_{max-T}$) can provide some insight into the intensity of the reaction. To that end, $t_{max-T}$ for SC samples was evaluated at the two environmental temperatures examined. The average $t_{max-T}$ was observed to be 1.6 and 0.9 h, with a range of 0.5 to 4.1 and 0.3 to 2.1 h, at 37°C and 50°C, respectively, as presented in Table 4-13.

**Table 4-13: $t_{max-T}$ (h) for salt cake reaction by facility**

| Facility | Sample number | 37 °C | | | 50 °C | | |
|---|---|---|---|---|---|---|---|
| | | Mean | $UCL_{95}$ | Range | Mean | $UCL_{95}$ | Range |
| B | 4 | 2.2±0.46 | 2.9 | 1.5-2.6 | 1.0±0.29 | 1.5 | 0.7-1.3 |
| C | 2 | 2.5±2.3 | 23 | 0.9-4.1 | 2.1±0.07 | 2.7 | 2-2.1 |
| D | 4 | 1.4±0.42 | 2.1 | 0.8-1.7 | 0.52±0.17 | 0.79 | 0.3-0.7 |
| E | 2 | 1.1±0.14 | 2.4 | 1-1.2 | 0.80±0.14 | 2.1 | 0.7-0.9 |
| F | 5 | 0.94±0.21 | 1.2 | 0.7-1.2 | 0.48±0.08 | 0.58 | 0.4-0.6 |
| H | 5 | 2.4±0.49 | 3.0 | 1.7-2.9 | 1.0±0.16 | 1.2 | 0.8-1.2 |
| J | 4 | 1.2±0.25 | 1.6 | 0.9-1.5 | 0.85±0.21 | 1.2 | 0.6-1.1 |
| L | 5 | 1.5±0.43 | 2.0 | 1-1.9 | 0.68±0.18 | 0.90 | 0.4-0.9 |
| M | 4 | 1.1±0.49 | 1.8 | 0.5-1.7 | 0.50±0.28 | 0.95 | 0.3-0.9 |
| N | 4 | 2.3±0.79 | 3.5 | 1.3-2.9 | 1.30±0.50 | 2.1 | 0.7-1.9 |
| All Facilities | 39 | 1.6±0.79 | 1.9 | 0.5-4.1 | 0.85±0.44 | 0.99 | 0.3-2.1 |

As expected, the time to reach the highest temperature at the 50°C environmental condition was significantly less than that at the 37°C ($p < 0.01$, ANOVA test). There was also an inverse correlation between the $\Delta T_{max}$ and $t\text{-}_{maxT}$. The Pearson product moment and Spearman rank order correlation coefficients at 37°C were -0.552 and -0.483 ($p < 0.01$), respectively. The corresponding coefficients at 50°C were -0.576 and -0.586 ($p < 0.01$), respectively. While the heat generation potential per gram of SC sample is a sample specific parameter, the results here suggests that higher environmental temperatures tend to intensify the SC reaction. This means that with increasing temperature the rate of SC reaction will also increase. As stated earlier, this is of particular significance for the disposal of SC. Depending on their design and capacity, MSW landfills tend to be well insulated and operate at elevated temperatures. When these conditions exist, the reaction of SC with liquids tends to become more problematic since the elevated temperatures in

MSW landfills may initiate the SC reaction and the insulating properties of the MSW may further intensify the reaction. Furthermore, the self-propagation effect that was stated earlier can also magnify the impact of the reaction.

### 4.2.3 Heat Generation Potential

The heat generation potential of SC was evaluated as described in section 2.3.3.2. Based on the results of factors impacting SC reactivity, heat generation was completed only at 37°C environmental temperature. Earlier results (section 4.1.1) demonstrated that heat generation potential, in general, is minimally impacted by environmental temperature. Results are outlined in Table 4-14 and the heat ranged from 0.2-3.6 kJ $g^{-1}$ with an average of 1.0 kJ $g^{-1}$. Samples from Facilities B and N had the lowest heat generation potential while sample from Facility M had the highest. Heat generation showed a correlation with metallic aluminum abundance of the samples but since the XRD data are semi-quantitative in nature, an accurate relationship between Al content and the heat generation of SC cannot be drawn. Given the wide range of variables that impact SC reactivity, it may be very simplistic to conclude that heat generation and reactivity are correlated to a single parameter. Furthermore, there was a positive correlation of all samples between the $\Delta T_{max}$ and heat as presented in Figure 4-14 ($r^2 = 0.62$, $p < 0.01$, and $p = 0.95$ bivariate normal ellipse is the green region in the plot), though the responses were different among facilities ($p < 0.01$, ANOVA test).

**Table 4-14: Heat (kJ $g^{-1}$) for salt cake reaction by facility at 37 °C**

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| B | 4 | 0.43±0.20 | 0.76 | 0.20-0.70 |
| C | 2 | 0.65±0.11 | 1.7 | 0.57-0.73 |
| D | 4 | 0.52±0.21 | 0.86 | 0.4-0.84 |
| E | 2 | 1.0±0.27 | 3.4 | 0.81-1.2 |
| F | 5 | 1.3±0.42 | 1.9 | 1.0-2.1 |
| H | 5 | 1.0±0.20 | 1.3 | 0.74-1.2 |
| J | 4 | 0.93±0.50 | 1.7 | 0.41-1.5 |
| L | 5 | 0.82±0.19 | 1.1 | 0.64-1.2 |
| M | 4 | 2.8±1.1 | 4.5 | 1.3-3.6 |
| N | 4 | 0.43±0.27 | 0.85 | 0.28-0.83 |
| All Facilities | 39 | 1.0±0.77 | 1.3 | 0.20-3.6 |

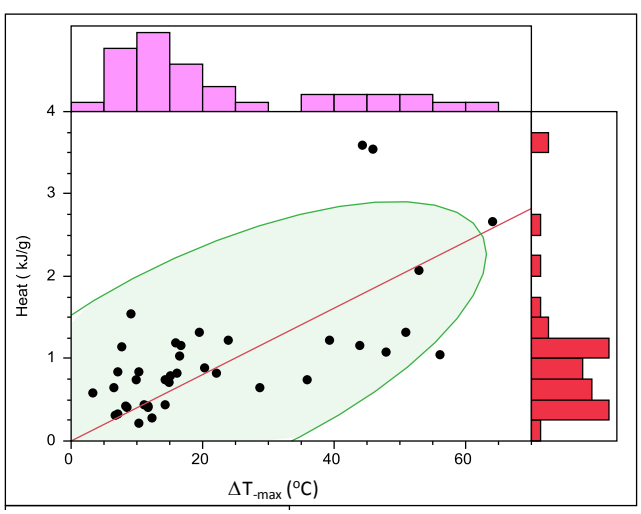



**Figure 4-14: Correlation between $\Delta T_{max}$ and heat generation**

### 4.2.4 Gas Generation Potential

The volume of gas generated as a result of SC contact with DIW at 50°C environmental temperature with a L/S ratio of 20:1 during 5 d reaction was measured for all SC samples collected. As presented in Table 4-15, the net gas volume generated ranged from 1.5 to 210 ml $g^{-1}$ of SC with an average of 35 ml $g^{-1}$ (35 $m^3$ $ton^{-1}$). Sample 2609 from Facility M generated the largest volume of gas (210 ml $g^{-1}$ of SC) while Sample 2536 from Facility D generated the lowest (1.5 ml $g^{-1}$). Half of the samples evaluated generated less than 22 ml $g^{-1}$ of SC. The detailed gas volumes generated from SC reaction with DIW by facility and the gas composition are presented in Appendix E (Table E-1). It is noted that while this test was carried out over a period of 5 d, most likely the gas generation occurred over a much shorter period, perhaps within 24 h. As expected, there was a positive correlation between the gas volume generated and the heat generation potential (Figure 4-15, $r^2 = 0.597$, $p < 0.01$). The ANOVA for Regression tests further demonstrated that the facility is not related to the correlation directly ($p = 0.34$).

**Table 4-15: Total generated gas volume (ml $g^{-1}$) from salt cake reactivity**

| Facility | Sample number | Mean | $UCL_{95}$ | Range |
|---|---|---|---|---|
| B | 4 | 17±9.9 | 33 | 7.5-30 |
| C | 2 | 28±4.0 | 64 | 25-31 |
| D | 4 | 18±11 | 35 | 1.5-25 |
| E | 2 | 36±11 | 130 | 28-43 |
| F | 5 | 48±12 | 62 | 31-60 |
| H | 5 | 15±5.1 | 22 | 9.1-22 |
| J | 4 | 26±7.1 | 37 | 19-35 |
| L | 5 | 20±16 | 39 | 1.5-40 |
| M | 4 | 130±75 | 250 | 47-210 |
| N | 4 | 18±3.9 | 24 | 12-221 |
| All Facilities | 39 | 35±40 | 48 | 1.5-210 |

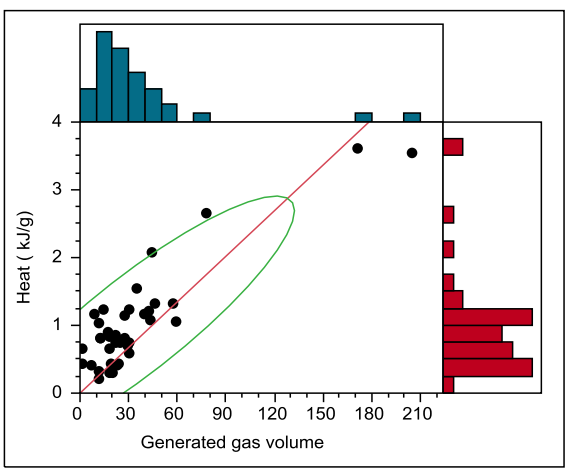



**Figure 4-15: Correlation between gas volume generated and heat generation**

To determine the composition of the generated gas, $H_2$, $CH_4$, Ammonia, $N_2O$ and $H_2S$ concentration were measured. However, $H_2S$ concentration was non-measurable because the adopted technique needed relatively large gas volumes that were not generated by the SC reaction.

#### 4.2.4.1 Hydrogen ($H_2$)

Hydrogen was the dominant gas generated (median 84% as presented in Figure 4-16) by the interaction of SC with water and was detected in all but one sample. As presented in Table 4-16, the overall concentration of $H_2$ in the collected gas samples ranged from below the MRL (< 0.14%) to 95%, with an average of 79%. It should be noted that the percentages presented here are based on the evolved gas generated from the reaction of the SC samples. The average productivity of $H_2$ from SC was 1.2 mmol $g^{-1}$, or 27 $m^3$ $tonne^{-1}$ (Table 4-16). Most SC samples had a productivity of less than 2 mmol $g^{-1}$ or 50 $m^3$ $tonne^{-1}$, but some samples released a considerable amount of $H_2$ (e.g., one tonne sample of 2609-M could produce as high as 180 $m^3$ $H_2$ ) in the current laboratory setting. Based on the samples evaluated, a conservative estimate of the mean ($UCL_{95}$ on the mean) for the productivity of $H_2$ from SC is approximately 39 $m^3$ $tonne^{-1}$ of SC.

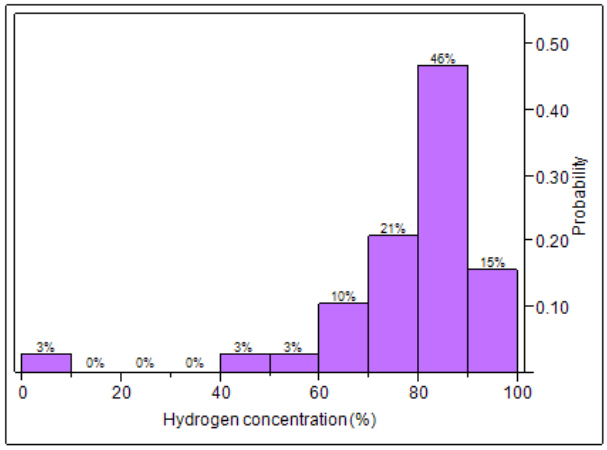



**Figure 4-16: Distribution of hydrogen concentration from gas generated by salt cake reaction**

**Table 4-16: Hydrogen generation from salt cake reaction**

| Facility | Sample number | Productivity (mmol $g^{-1}$) | | Concentration (%) | |
|---|---|---|---|---|---|
| | | Mean | $UCL_{95}$ | Mean | $UCL_{95}$ |
| B | 4 | 0.51±0.33 | 1.0 | 70±7 | 72 |
| C | 2 | 0.96±0.16 | 2.4 | 85±10 | 85 |
| D | 4 | 0.56±0.38 | 1.2 | 59±40 | 72 |
| E | 2 | 1.2±0.39 | 4.8 | 85±15 | 85 |
| F | 5 | 1.7±0.39 | 2.1 | 86±3 | 87 |
| H | 5 | 0.50±0.13 | 0.63 | 76±5 | 78 |
| J | 4 | 0.90±0.30 | 1.4 | 85±5 | 87 |
| L | 5 | 0.60±0.49 | 1.2 | 70±15 | 75 |
| M | 4 | 5.0±3.1 | 9.8 | 95±3 | 96 |
| N | 4 | 0.60±0.12 | 0.78 | 84±4 | 85 |
| All Facilities | 39 | 1.2±1.6 | 1.8 | 79±16 | 84 |

A positive correlation was observed between the $\Delta T_{max}$ and the $H_2$ productivity (Figure 4-17a) as well as heat and the $H_2$ productivity (Figure 4-17b, $r^2 = 0.720$, $p < 0.01$). The ANOVA for Regression tests indicated that the contribution of facility is not significant ($p = 0.26$). For example, the Pearson Product Moment and Spearman Rank Order correlation coefficients for $\Delta T_{max}$ and productivity of $H_2$ were 0.544 ($p < 0.01$) and 0.445 ($p < 0.01$) at 37°C and the corresponding coefficients were 0.438 ($p < 0.01$) and 0.430 ($p < 0.01$) at 50°C. The presence of $H_2$ gas at such high levels poses a particular concern for the end-of-life management of SC. $H_2$ is explosive in a range (in air) of 4 to 79%,

and as a result, can potentially cause fires. $H_2$ is of even greater concern for the landfill gas export if generated in MSW landfills. Apart from being a fire hazard when mixed with $CH_4$, landfill gas collection, transportation and treatment systems are not designed to handle $H_2$ since MSW landfill gas generally consists of $CH_4$ and $CO_2$ at ~ 50/50 ratio, since the full combustion requirement of oxygen for $CH_4$ and mixed gas are different from that of $H_2$ [102]. This combination can make more trouble for the landfill gas transport and other gas applications [103].

According to the stoichiometry of Al reactions with water, one mole metallic aluminum generates 1.5 mole $H_2$ or 36.7 L $H_2$ at room temperature. Compared to the results of $H_2$ productivity in the current laboratory setting, it was found that not all metallic aluminum in SC was reacted; on average, about one of third of metallic aluminum in SC remains unreacted based on the semi-quantitative measurement of metallic aluminum by XRD (Figure 4-18).

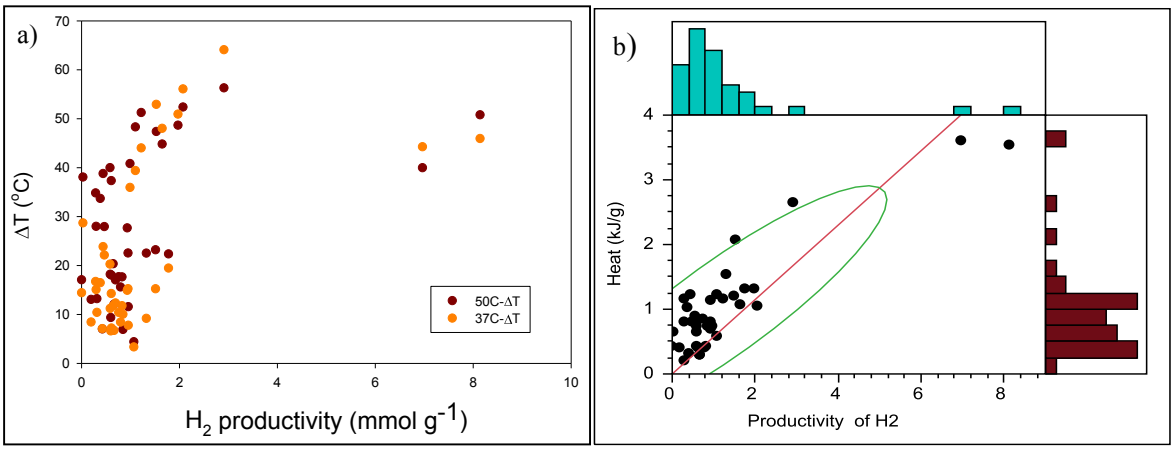



**Figure 4-17: Relationship between (a) $H_2$ productivity and $\Delta T_{max}$, and (b) $H_2$ productivity and heat generation**

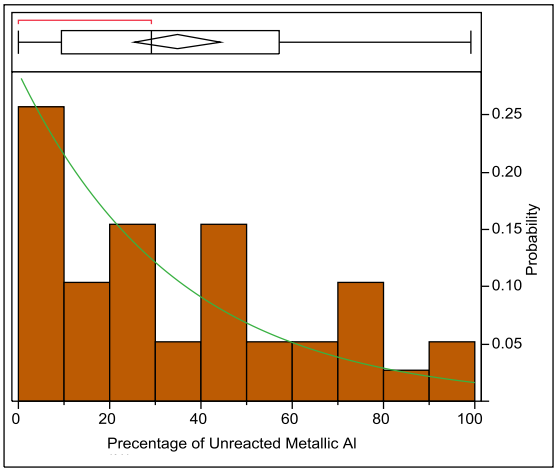



**Figure 4-18: Variability of percentage of unreacted metallic aluminum in SC after gas experiments**

#### 4.2.4.2 Methane ($CH_4$)

Methane was the second most abundant gas detected from SC reaction. $CH_4$ was measured in 99% of the samples evaluated (Table 4-17), but typically at concentrations less than those measured for $H_2$. Approximately half of the samples generated $CH_4$ gas at less than 16% by volume (Figure 4-19). The average productivity of $CH_4$ was ~ 0.2 mmol $g^{-1}$ (4.5 $m^3$ $tonne^{-1}$ SC) as presented in Table 4-17.

Assuming that the $CH_4$ generation resulted from the reaction of aluminum carbide with liquids, the average aluminum

carbide content of SC would be 0.6% with a 0.8% $UCL_{95}$ on the mean. These concentrations are well below the XRD's detection limit supporting the crystalline phase analysis presented earlier. Compared to $H_2$, the average productivity of $CH_4$ was about 83% lower. Only two samples of all 39 studied SC samples had a higher productivity of $CH_4$ than $H_2$. Like $H_2$, $CH_4$ is an explosive gas and care must be taken if SC reacted with liquids. But unlike $H_2$, in an MSW landfill, the gas collection system is designed to handle $CH_4$ which is a greenhouse gas generated during the anaerobic decomposition of MSW.

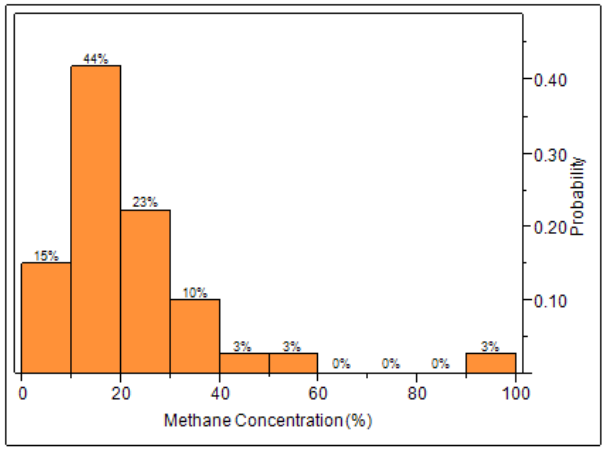



**Figure 4-19: Distribution of methane concentration from gas generated by salt cake reaction**

**Table 4-17: Methane generation from salt cake reaction**

| Facility | Sample number | Productivity (mmol $g^{-1}$) | | Concentration (%) | |
|---|---|---|---|---|---|
| | | Mean | $UCL_{95}$ | Mean | $UCL_{95}$ |
| B | 4 | 0.19±0.07 | 0.31 | 30±7 | 37 |
| C | 2 | 0.17±0.01 | 0.3 | 15±1 | 16 |
| D | 4 | 0.17±0.1 | 0.34 | 41±40 | 80 |
| E | 2 | 0.22±0.05 | 0.66 | 15±2 | 17 |
| F | 5 | 0.27±0.1 | 0.41 | 14±3 | 17 |
| H | 5 | 0.15±0.08 | 0.25 | 24±5 | 29 |
| J | 4 | 0.15±0.03 | 0.2 | 15±5 | 19 |
| L | 5 | 0.20±0.15 | 0.39 | 30±16 | 44 |
| M | 4 | 0.25±0.08 | 0.38 | 5±4 | 8 |
| N | 4 | 0.12±0.05 | 0.19 | 16±4 | 19 |
| All Facilities | 39 | 0.19±0.09 | 0.22 | 21±17 | 26 |

#### 4.2.4.3 Ammonia

Ammonia, both gaseous and aqueous, is also a byproduct of SC reaction with liquids. As presented in Table 4-18, the average concentration of $NH_{3\,(gas)}$ was 1.1% and ranged from 0.001 to 10% (Figure 4-20). The average productivity of gaseous $NH_3$ was 6.4 µmol $g^{-1}$. $NH_3$ gas has a distinctive odor and humans are able to detect it down to a level as low as 5 parts per million (ppmv). From a human health perspective, the concentration of gaseous $NH_3$ resulting from the SC reaction was relatively high and can potentially be of concern.

The concentration of aqueous ammonia ($NH_4^+$) ranged from 30 to 1,480 mg $L^{-1}$ with an average of 570 mg $L^{-1}$ (Table 4-18). As presented in Figure (4-20) ~ 70% of the SC samples generated < 1% ammonia gas (Figure 4-20a) while ~ 20 % of the aqueous concentrations were > 1,000 mg $L^{-1}$ (Figure 4-20b). The productivity in the gaseous $NH_3$ was significantly lower than $NH_4^+$ in the liquid despite the alkaline pH of the liquid phase. On average, the productivity of

gaseous $NH_3$ was only about 2% of the total ammonia productivity. This may be attributed to the fact that the experiment was conducted in a closed system where the relatively low Henry's constant for ammonia gas (0.75atm, [104]) resulted in the majority of the ammonia gas fraction being in the liquid phase. To put this in context, $H_2$ and $CH_4$ gases have Henry's constants of 68,300 atm and 37,600 atm, respectively [104].

The total ammonia productivity ranged from 0.32 to 1.2 mmol $g^{-1}$, with an average of 0.68 mmol $g^{-1}$ (Table 4-18). Around 75% of the samples had a total ammonia productivity less than 1.0 mmol $g^{-1}$ (Figure 4-21a). It is noted that MSW landfills are open systems and thus, gaseous $NH_3$ may be significantly higher than the levels detected in this experiment and thus odor problems in landfills as a result of SC reactivity can still be of concern. On the other hand, the level of $NH_4^+$ resulting from the SC reaction is significantly lower compared to the levels found in landfills. Therefore, the ammonia generated from SC might not have a detrimental impact on the performance of MSW landfills.

**Table 4-18: Ammonia generation from salt cake reaction**

| Facility | Sample number | $NH_{3\,(gas)}$ (%) | | $NH_4^+$ (mg $L^{-1}$) | | Total ammonia ( mmol $g^{-1}$) | |
|---|---|---|---|---|---|---|---|
| | | Mean | $UCL_{95}$ | Mean | $UCL_{95}$ | Mean | $UCL_{95}$ |
| B | 4 | 1.2±0.68 | 0.21 | 1,000±200 | 1360 | 1.2±0.2 | 1.6 |
| C | 2 | 0.93±0.89 | 1.22 | 490±350 | 3610 | 0.59±0.4 | 4.2 |
| D | 4 | 3.2±4.6 | 0.37 | 420±380 | 1020 | 0.50±0.4 | 1.2 |
| E | 2 | 0.55±0.18 | 1.21 | 550±470 | 4750 | 0.65±0.5 | 5.6 |
| F | 5 | 0.27±0.24 | 0.17 | 650±440 | 1200 | 0.78±0.5 | 1.4 |
| H | 5 | 2.0±0.44 | 0.28 | 680±450 | 1240 | 0.81±0.5 | 1.5 |
| J | 4 | 0.41±0.16 | 0.11 | 270±210 | 607 | 0.32±0.2 | 0.72 |
| L | 5 | 1.3±1.5 | 0.19 | 720±700 | 1580 | 0.85±0.8 | 1.9 |
| M | 4 | 0.13±0.11 | 0.15 | 480±150 | 870 | 0.57±0.2 | 1.0 |
| N | 4 | 0.65±0.79 | 0.23 | 290±160 | 537 | 0.34±0.2 | 0.63 |
| All Facilities | 39 | 1.1±1.7 | 0.15 | 570±420 | 710 | 0.68±0.5 | 0.84 |

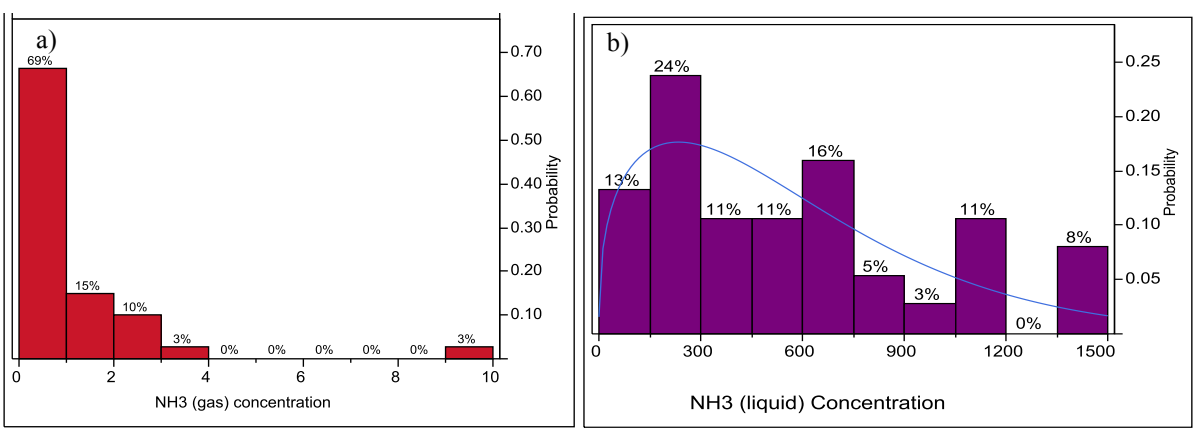



**Figure 4-20: Distribution of ammonia after salt cake reaction (a) $NH_{3(gas)}$, and (b) $NH_4^+$**

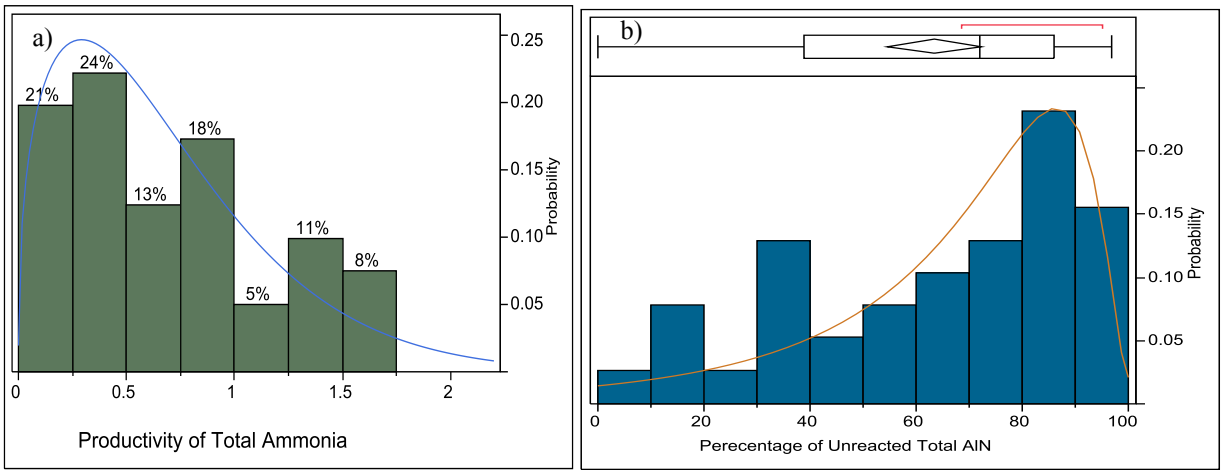



**Figure 4-21: Distribution of the total ammonia productivity and unreacted AlN in SC after gas experiments**

Based on the stoichiometry of AlN, one mole AlN or AlN oxide with water will generate one mole total $NH_3$. Compared to the total productivity of $NH_3$ of SC in the current laboratory setting, it was found that only about 35% of the AlN in SC had reacted with water (Figure 4-21b). XRD further confirmed that metallic aluminum and AlN can be detected in the residue of SC, even after five month long reactions (37°C /50°C, data not shown).

#### 4.2.4.4 Nitrous Oxide

Salt cake reaction with liquids can also generate nitrous oxide ($N_2O$) gas which is a greenhouse gas that contributes approximately 6% to the total radiative forcing of the earth's atmosphere. $N_2O$ gas was detected with an average concentration of 1,200 ppbv from an extremely large range of 20 to 18,000 ppbv (Table 4-19). The productivity of $N_2O$ ranged from 0.08 to 2.0 nmol $g^{-1}$ with an average of 0.57 nmol $g^{-1}$ (Table 4-19), and about half of the studied SC samples were lower than 0.25 nmol $g^{-1}$ (Figure 4-22). Research suggests that $N_2O$ gas can be generated from $NH_3$ gas directly [105, 106]. No correlation between $N_2O$ and the $NH_3$ gas was observed, but a significant correlation between the productivity of $N_2O$ and the total $NH_3$ was confirmed (Figure 4-21). The value of productivity of $N_2O$ in this study was only 0.01% of the productivity of $NH_3$ gas. The correlation coefficients of Pearson Product Moment and Spearman Rank Order were 0.522 ($p < 0.001$) and 0.604 ($p < 0.001$), respectively.

**Table 4-19: Nitrous oxide generation from salt cake reaction**

| Facility | Sample number | Productivity (nmol $g^{-1}$) | | Concentration (ppmv) | | |
|---|---|---|---|---|---|---|
| | | Mean | $UCL_{95}$ | Mean | $UCL_{95}$ | Range |
| B | 4 | 1.2±0.6 | 2.1 | 2.5±2.1 | 5.9 | 0.4-4.9 |
| C | 2 | 0.21±0.1 | 1.1 | 0.18±0.06 | 0.72 | 0.14-0.23 |
| D | 4 | 0.82±0.7 | 1.9 | 5.2±8.8 | 1.9 | 0.88-18 |
| E | 2 | 0.09±0.02 | 0.09 | 0.06±0.02 | 0.24 | 0.05-0.08 |
| F | 5 | 0.38±0.5 | 1.0 | 0.18±0.2 | 0.42 | 0.05-0.52 |
| H | 5 | 0.37±0.4 | 0.87 | 0.7±1.2 | 2.2 | 0.15-2.8 |
| J | 4 | 0.59±0.9 | 2.1 | 0.75±1.3 | 2.8 | 0.07-2.7 |
| L | 5 | 0.62±0.7 | 1.4 | 0.96±0.79 | 1.9 | 0.08-1.8 |
| M | 4 | 0.81±0.7 | 1.9 | 0.2±0.23 | 0.57 | 0.02-0.51 |
| N | 4 | 0.28±0.2 | 0.61 | 0.24±0.12 | 0.43 | 0.16-0.42 |
| All Facilities | 39 | 0.57±0.59 | 0.76 | 1.2±3.0 | 2.1 | 0.02-18 |

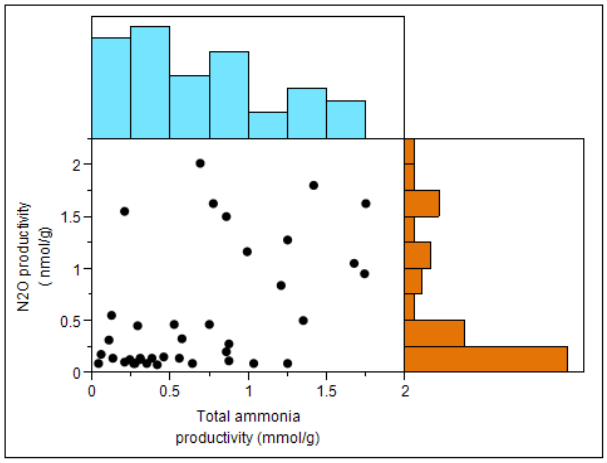



**Figure 4-22: Relationship between the productivity of total $NH_3$ and $N_2O$ from salt cake reaction**

# 5. CONCLUSION

The findings presented in this report provide valuable information that may be used by managers of SAP facilities and solid waste disposal facilities and regulators to determine the best management practices for SC (a major type of SAP waste). A total of 39 SC samples collected from 10 SAP facilities across the U.S. were characterized and tested for reactivity with water under controlled laboratory conditions. The major findings of this research are highlighted as follows:

## 5.1 Salt Cake Characteristics

The 39 SC samples were evaluated for moisture content, water holding capacity, pH, electric conductivity, total metals, leachable metals, and crystalline phases. Overall, SC samples had a large degree of inter- and intra-sample variability which could be attributed to variability of the Al feed stock, conditions at the processing facilities, and sample processing and analysis.

The moisture content of the SC samples investigated had an average value of 0.80% and ranged from 0.01 to 2.0% (w/w). The average water holding capacity of SC was 25% and ranged from 1.4 to 70%. SC samples were highly alkaline. When reacted with DIW, pH 6.2, S/L ratio of 1:20, 50°C, 5d test, the pH of SC ranged from 9.6 to 11 with an average of 10.5. The electrical conductivity (EC) of SC was high and exhibited high variability among samples. The average EC was 30 mS $cm^{-1}$ and ranged from 18 to 47 mS $cm^{-1}$.

The average total Al content in the SC samples was 14%, and ranged from 6.7 to 42%. Approximately 70% of the SC samples contained total Al at concentrations between 8 and 16%, and only 6% of the SC samples contained Al at concentrations greater than 24%. Other metals were also detected in SC samples and the general order of the elemental content was: Al > Na > K > Mg > Ca > Fe > Mn > Cu > Zn > S > Cr >> Pb > As > Se >> Cd. There was a direct correlation between most of the trace metals concentrations and the Al content of the samples which may be attributed to the presence of some of these metals in various Al alloys.

Al was detected in the leachate from all SC samples. The average concentration of Al was 41 mg $L^{-1}$ with a range of 2.2 to150 mg $L^{-1}$. The concentrations of all heavy metals in the leachate were relatively low, at the μg $L^{-1}$ level, quantified in the following order: Cu > Zn > Se ≈ Mn >> Pb, As, and Cr >> Cd. The leachability of an element was defined as the percentage of the mass leached divided by the mass determined using total metal analysis. The average leachability Al from SC was 0.6%. Approximately 80% of the SC samples leached less than 1% of the total Al content and the rest of the samples leached up to 2% of total Al content. The other metals in SC can be divided into three groups based on their leachability:

- Group I which leached 60 to 100% of the corresponding metal content in SC. This group contained potassium, sodium, and sulfur and leached in the order: K > S > Na.
- Group II (up to 20%) contained selenium, arsenic, copper, lead and zinc.
- Group III (< 1%) contained manganese, magnesium, iron and chromium.
- The leachability of calcium was highly dependent on the source of SC samples.

None of the SC samples were observed to be a characteristic hazardous waste based on TCLP. While, the SC samples contained elevated total metal content, none of the SC samples had TCLP concentrations exceeding the toxicity limit for seven of the eight RCRA metals (Hg was not analyzed).

The dominant crystalline Al minerals in the SC samples were metallic aluminum, aluminum nitride (AlN) and its oxides, spinel (magnesium aluminum oxide, $Al_2MgO_4$), elpasolite ($K_2NaAlF_6$) and different forms of aluminum oxide (e.g., $Al_2O_3$, $Al_{2.67}O_4$). Sylvite (KCl), halite (NaCl), and fluorite ($CaF_2$) were also identified by XRD analysis. Metallic aluminum abundance in SC was found to have a high variability, from 1 to 18%, with an average of 3.2% of the mass; whereas the total AlN abundance ranged from 3.8 to 13% with an average of 8%. Both aluminum carbide ($Al_4C_3$) and aluminum sulfide ($Al_2S_3$) were not detected by XRD analysis.

## 5.2 Salt Cake Reactivity

The temperature profile of the SC reaction with water (the maximum temperature change, $\Delta T_{max}$ (°C), the time to reach the highest post-reaction temperature, $t_{max}$ (h)), the heat generation potential, and the gas generation potential were the key indices employed to evaluate the SC reactivity with water. The impact of various factors on SC reactivity was also investigated. The findings of these evaluations are highlighted below:

The environmental temperature, particle size, liquid composition, L/S ratio, and Al speciation in SC were found to significantly influence SC reactivity.

The increase in environmental temperature resulted in increasing $\Delta T_{max}$, decreasing the $t_{max}$ (h) and increasing $H_2$ productivity. The differences between the behaviors of the SC samples in response to a change in environmental temperature could be attributed to variation in the speciation of Al present in the SC samples. Environmental temperature, on the other hand, had minimal impact on the heat generation potential.

The decrease in particle size resulted in an increase in $\Delta T_{max}$ and a decrease in the time needed to achieve maximum temperature ($t_{max\text{-}T}$) because the surface area to volume ratio increases as the particle size is reduced. While it was difficult to quantify the sample heterogeneity and its impact on heat and gas generation, there was a clear trend across the samples that SC with smaller particle size generated more heat and more $H_2$ gas.

The chemical composition of the liquid reacting with SC impacted the temperature change and gas generation potential. The maximum temperature increase, faster reactions, and the higher gas productivity occurred when SC was reacted with alkaline solution (0.1M NaOH) or DIW. Unlike temperature change and gas productivity, the heat generation potential was minimally impacted by changes in composition of liquid.

Various aluminum species tend to react with liquids at different rates. The data presented in this report demonstrated that the individual aluminum species are unable to produce elevated temperatures at concentrations many times higher than those observed in actual SC samples. Thus, the results are unable to explain some of the field data observed at SC disposal sites. The reaction temperatures achieved independently by each aluminum species are not additive which further suggests a synergetic effect between aluminum species. Similar to temperature change, aluminum species had significant impacts on the heat generation potential from SC. The results also showed that the presence of AlN in SC is necessary for the reaction to generate measurable heat; however, the magnitude of heat generation is not linearly correlated to the concentration of AlN.

The metallic aluminum in SC is the key component responsible for SC reactivity. There is a trigger effect of AlN and $Al_4C_3$ involved in the reaction of Al with water. The trigger effect of AlN on the reaction of metallic aluminum with DIW was demonstrated.

Based on the 39 SC samples investigated, on average, the $\Delta T_{max}$ for reaction temperatures of 37°C and 50°C was 22 and 29°C, and ranged from 3.4 to 64 and 4.4 to 56°C, respectively. The average time to reach the peak temperature ($t_{maxT}$) for reaction temperatures of 37°C and 50°C was 1.6 and 0.9 h, and ranged from 0.5 to 4.1 and 0.3 to 2.1 h, respectively.

The heat generation potential of the 39 SC samples ranged from 0.2 to 3.6 kJ $g^{-1}$ with an average of 1.0 kJ $g^{-1}$.

Heat generation showed a correlation with elemental Al content of the samples, but since the XRD data are semi-quantitative in nature, an accurate relationship between Al content and the heat generation of SC cannot be drawn. Given the wide range of variables that impact SC reactivity, it may be very simplistic to conclude that heat generation and reactivity are correlated to a single parameter. Furthermore, there was a positive correlation between the $\Delta T_{max}$ and heat.

The generated gas volume of the 39 SC samples ranged from 1.5 to 210 ml $g^{-1}$ of SC with an average of 35 ml $g^{-1}$ (35 $m^3$ $tonne^{-1}$) with half of the samples generating < 22 ml $g^{-1}$ of SC.

Hydrogen ($H_2$) was the dominant gas generated from SC. The overall concentration of $H_2$ in the collected gas samples ranged from below the MRL (< 0.14%) to 95%, with an average of 79%. The average productivity of $H_2$ from SC was

1.2 mmol $g^{-1}$, or 27 $m^3$ $tonne^{-1}$. A positive correlation was observed between the $\Delta T_{max}$ and the $H_2$ productivity as well as heat.

Methane ($CH_4$) was the second most abundant gas detected from the SC reaction. $CH_4$ was measured in all samples evaluated, but typically at concentrations less than those measured for $H_2$. Approximately half of the samples generated $CH_4$ gas at less than 16% by volume. The average productivity of $CH_4$ was ~ 0.2 mmol $g^{-1}$ (4.5 $m^3$ $tonne^{-1}$ SC).

The average concentration of ammonia in gas was 1.1% and ranged from 0.001 to 10%. The average productivity of gaseous $NH_3$ was 6.4 μmol $g^{-1}$. The concentration of aqueous ammonia ($NH_4^+$) ranged from 30 to 1,480 mg $L^{-1}$ with an average of 570 mg $L^{-1}$. Approximately, 70% of the SC samples generated < 1% ammonia gas while ~ 20% of the aqueous ammonia concentrations were > 1,000 mg $L^{-1}$. The total ammonia productivity ranged from 0.32 to 1.2 mmol $g^{-1}$, with an average of 0.68 mmol $g^{-1}$. Approximately 75% of the samples had a total ammonia productivity less than 1.0 mmol $g^{-1}$.

Based on the gas analysis results and the stoichiometry of aluminum reactions, not all aluminum species in the SC reacted with water under the investigated conditions. On average, only 65% of the metallic aluminum and 35% of the AlN reacted with water to generate $H_2$ or total ammonia.

## 5.3 Implications

While the percent metal leached from SC was relatively low, the leachable metal content may still pose a contamination concern and potential human and ecological exposure if uncontrollably released to the environment. As a result, SC should always be managed at facilities that utilize synthetic liner systems with leachate collection (the salt content of the leachate will increase the hydraulic conductivity of clay liners within a few years of installation). The mineral phase analysis showed that various species of aluminum are present in the SC samples with a large degree of variability. The relative abundance of various aluminum species was evaluated but it is noted that the method used is a semi-quantitative method and as a result, there is a limitation for the data use. The analysis only showed a few aluminum species present in SC which does not exclude the presence of other crystalline species especially in light of the variability observed in the samples.

Results presented here are of particular importance when trying to understand concerns associated with the disposal of SC in MSW landfills. From an end-of-life management perspective, data presented here suggest that SC should not be size reduced before disposal. Also, care should be taken not to size reduce it after disposal by compacting it with heavy bulldozers. The MSW decomposition process is an exothermic, and as a result MSW landfill temperatures are typically greater than 37°C and may reach 50°C or greater. The elevated temperatures present in most MSW landfills can be conducive and could initiate SC reactions that otherwise may not take place. Furthermore, elevated temperatures can increase the rate of the SC reaction which further propagates the SC reaction. Even outside of MSW landfills, once a SC reaction begins, it may self-propagate and increase the rate at which the reaction occurs. While the heat generation potential per gram of SC sample is a sample specific parameter, the results presented herein suggest that higher environmental temperatures tend to intensify the SC reaction. This means that with increasing temperature, the rate of SC reaction will also increase. As stated earlier, this is of particular significance for the disposal of SC. Depending on their design and capacity, MSW landfills tend to be well insulated and operate at elevated temperatures. When these conditions exist, the reaction of SC with liquids tends to become more problematic since the elevated temperatures in MSW landfills may initiate the SC reaction, and the insulating properties of the MSW may further intensify the reaction. Furthermore, the propagation effect that has been stated earlier can also magnify the impact of the reaction.

At elevated temperatures encountered in MSW landfills, elevated levels of $H_2$ gas may be generated as a result of SC reactivity and will be of concern. $H_2$ is an explosive gas, can potentially cause fires and $H_2$ is of even greater concern if generated in MSW landfills. Apart from being a fire hazard when mixed with $CH_4$, landfill gas collection systems are not designed to handle $H_2$ since MSW landfill gas generally consists of $CH_4$ and $CO_2$ at ~ 50/50 ratio. Like $H_2$, $CH_4$ is an explosive gas and care must be taken if SC reacts with liquids. But unlike $H_2$, in an MSW landfill, the gas collection system is designed to handle $CH_4$ gas which is a greenhouse gas generated during the anaerobic decomposition of MSW. Since the full combustion of $CH_4$ requires more oxygen than that of $H_2$, an unstable

composition of landfill gas can cause trouble for its management and applications. From a human health perspective, the concentration of ammonia gas resulting from the SC reaction was relatively high and can potentially be of concern. Due to the fact of the level of $NH_4^+$ resulted from the SC reaction is significantly lower compared to the levels found in MSW landfills, the ammonia generated from SC might not have a detrimental impact on the performance of landfills.

It is important to note that all of the data presented in this report were generated under controlled conditions in a laboratory setting with some degree of uncertainties as a result of the analytical techniques and the experimental setup and thus, this should be taken into consideration if this data are to be used for design or modeling purposes. Also, additional work is necessary to provide a full understanding of the mechanisms of SC reactivity when exposed to liquids. Many questions concerning the management of SC disposal are still unanswered and more research is necessary for determining best management practices for SC disposal.

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*Secondary Aluminum Processing Waste: Salt Cake Characterization and Reactivity*

# 7. APPENDIX A: MDL and MRL for Metal and Gas Analysis

**Table A-1: Method detection limit and method reporting limit for metal analysis using ICP**

| Element | MDL | MRL -leachate | MRL-solid |
|---|---|---|---|
| **Al** | **33** | **95** | **47** |
| **Ca** | **86** | **257** | **129** |
| **Cu** | **2.0** | **7** | **3.5** |
| **Fe** | **35** | **105** | **53** |
| **K** | **284** | **850** | **426** |
| **Mg** | **3.0** | **10** | **4.9** |
| **Mn** | **5.0** | **14** | **7.2** |
| **Na** | **457** | **1400** | **686** |
| **S** | **109** | **330** | **163** |
| **Zn** | **2.3** | **5.2** | **3.5** |

**Table A-2: Method detection limit and method reporting limit for metal analysis using GFAA**

| Element | GFAA* | | Standard Addition Method** | |
|---|---|---|---|---|
| | MDL ($\mu g\ L^{-1}$) | MRL-solid ($mg\ kg^{-1}$) | MDL ($\mu g\ L^{-1}$) | MRL-leachate ($\mu g\ L^{-1}$) |
| As | 0.67 | 1.0 | 0.5 | 1.8 |
| Cd | 0.21 | 0.3 | NA | NA |
| Cr | 0.97 | 1.5 | 0.23 | 0.75 |
| Pb | 4.4 | 6.6 | 0.75 | 2.5 |
| Se | 2.2 | 3.3 | 0.6 | 10 |

*GFAA: Graphite furnace AA, utilized for measuring trace metals;
** Standard Addition Methods: utilized for analyzing the leachable trace metals; NA: Not analyzed

**Table A-3: Method reporting limit for gas analysis using GC**

| Gas | MRL |
|---|---|
| $H_2$ | 0.14% |
| $CH_4$ | 0.2% |
| $N_2O$ | 20 ppbv |

*Secondary Aluminum Processing Waste: Salt Cake Characterization and Reactivity*

# 8. APPENDIX B: Physical and Chemical Properties of Salt Cake

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**Table B-1: Physical properties of salt cake**

| Sample ID | WHC % | MC % | pH | EC mS cm$^{-1}$ | Sample ID | WHC % | MC % | pH | EC mS cm$^{-1}$ |
|---|---|---|---|---|---|---|---|---|---|
| 2452-B-SC | 2.43 | 0.51 | 10.72 | 25.16 | 2559-H-SC | 15.7 | 1.12 | 10.57 | 33.00 |
| 2454-B-SC | 2.23 | 1.26 | 10.72 | 23.46 | 2568-H-SC | 43.58 | 0.79 | 10.52 | 23.63 |
| 2468-B-SC | 2.96 | 0.43 | 11.27 | 25.42 | 2512-J-SC | 30.19 | 0.27 | 11.02 | 32.70 |
| 2478-B-SC | 1.69 | 0.66 | 10.49 | 32.90 | 2515-J-SC | 21.21 | 0.35 | 9.60 | 29.48 |
| 2433-C-SC | 9.93 | 0.52 | 10.00 | 24.72 | 2517-J-SC | 24.91 | 1.09 | 9.93 | 30.90 |
| 2435-C-SC | 3.02 | 1.31 | 10.01 | 32.20 | 2519-J-SC | 19.81 | 0.7 | 10.04 | 26.13 |
| 2532-D-SC | 10.96 | 2.22 | 10.74 | 46.60 | 2482-L-SC | 40.11 | 0.53 | 10.96 | 33.20 |
| 2536-D-SC | 11.36 | 1.3 | 10.85 | 28.05 | 2484-L-SC | 43.85 | 1.02 | 10.82 | 20.97 |
| 2538-D-SC | 1.97 | 0.15 | 10.31 | 35.40 | 2486-L-SC | 41.94 | 0.8 | 10.73 | 22.30 |
| 2540-D-SC | 1.38 | 0.51 | 10.27 | 41.30 | 2488-L-SC | 37.91 | 2.06 | 10.66 | 33.20 |
| 2437-E-SC | 24.38 | 0.74 | 10.41 | 32.60 | 2490-L-SC | 69.24 | 0.29 | 10.86 | 29.41 |
| 2439-E-SC | 12.78 | 0.77 | 10.63 | 28.75 | 2601-M-SC | 43.84 | 1.48 | 10.47 | 29.03 |
| 2502-F-SC | 3.07 | 1.02 | 10.53 | 31.40 | 2605-M-SC | 59.22 | 0.16 | 10.06 | 18.40 |
| 2504-F-SC | 14.86 | 1.08 | 10.85 | 25.93 | 2609-M-SC | 69.48 | 1.88 | 10.35 | 22.00 |
| 2506-F-SC | 4.95 | 0.99 | 11.15 | 47.40 | 2613-M-SC | 47.86 | 0.01 | 10.05 | 31.30 |
| 2508-F-SC | 17.8 | 0.67 | 10.59 | 28.76 | 2492-N-SC | 23.8 | 0.47 | 10.27 | 27.07 |
| 2510-F-SC | 52.86 | 0.04 | 11.07 | 37.00 | 2494-N-SC | 29.4 | 0.78 | 10.45 | 27.27 |
| 2046-H-SC | 10.97 | 1.15 | 10.91 | 26.49 | 2496-N-SC | 2.9 | 0.13 | 10.20 | 23.26 |
| 2544-H-SC | 33.87 | 1.51 | 10.66 | 30.60 | 2498-N-SC | 63.06 | 0.28 | 10.69 | 30.20 |
| 2555-H-SC | 29.43 | 1.12 | 10.59 | 27.37 | | | | | |

**Table B-2: Major elemental composition of salt cake (EPA Method 6010C)**

| Sample ID | Al | Fe | Ca | K | Mg | Na | S | Zn | Cu | Mn |
|---|---|---|---|---|---|---|---|---|---|---|
| | % | | | | | | mg kg$^{-1}$ | | | |
| 2452-B-SC | 8.46 | 0.34 | 1.15 | 5.77 | 1.67 | 10.02 | <163 | 349 | 217 | 2766 |
| 2468-B-SC | 10.62 | 0.36 | 1.72 | 4.65 | 1.69 | 10.53 | <163 | 376 | 126 | 2857 |
| 2478-B-SC | 13.82 | 0.29 | 1.04 | 4.43 | 1.18 | 9.36 | <163 | 230 | 88 | 2075 |
| 2454-B-SC | 11.00 | 0.45 | 1.16 | 7.56 | 1.58 | 7.28 | 915 | 367 | 174 | 1955 |
| 2433-C-SC | 11.68 | 0.37 | 2.07 | 4.30 | 2.34 | 10.73 | 163 | 902 | 1719 | 1034 |
| 2435-C-SC | 12.99 | 0.40 | 1.67 | 3.77 | 2.50 | 11.45 | 465 | 1445 | 2028 | 1046 |
| 2532--D-SC | 9.46 | 0.54 | 0.56 | 12.51 | 3.72 | 11.94 | 300 | 650 | 532 | 766 |
| 2536-D-SC | 11.57 | 0.53 | 0.47 | 10.27 | 1.70 | 9.20 | 210 | 493 | 417 | 1136 |
| 2538-D-SC | 9.19 | 0.37 | 0.52 | 12.79 | 2.07 | 11.73 | 377 | 618 | 374 | 589 |
| 2540-D-SC | 10.56 | 0.46 | 0.64 | 8.51 | 1.84 | 12.07 | 458 | 536 | 511 | 1191 |
| 2437-E-SC | 12.09 | 0.36 | 1.98 | 5.39 | 3.54 | 10.52 | 366 | 709 | 554 | 1322 |
| 2439-E-SC | 11.79 | 0.32 | 2.15 | 6.17 | 2.97 | 10.88 | 411 | 547 | 377 | 1763 |
| 2502-F-SC | 16.04 | 1.37 | 0.56 | 3.00 | 1.08 | 11.30 | <163 | 618 | 251 | 2994 |
| 2504-F-SC | 12.94 | 0.42 | 0.79 | 4.06 | 2.66 | 11.49 | <163 | 499 | 153 | 2067 |
| 2506-F-SC | 9.24 | 0.15 | 0.37 | 7.36 | 2.80 | 16.54 | <163 | 458 | 397 | 160 |
| 2508-F-SC | 12.74 | 0.21 | 0.72 | 4.72 | 1.31 | 12.29 | <163 | 556 | 183 | 1653 |
| 2510-F-SC | 14.25 | 0.32 | 1.41 | 5.12 | 3.01 | 12.34 | <163 | 380 | 263 | 2033 |
| 2046-H-SC | 21.22 | 0.37 | 1.08 | 5.41 | 0.70 | 7.90 | 566 | 834 | 557 | 1427 |
| 2544-H-SC | 17.45 | 0.32 | 1.13 | 6.90 | 0.86 | 9.68 | 163 | 772 | 415 | 1052 |
| 2555-H-SC | 16.00 | 0.29 | 1.17 | 5.75 | 0.56 | 8.70 | <163 | 648 | 381 | 632 |
| 2559-H-SC | 15.72 | 0.49 | 1.29 | 6.26 | 0.71 | 8.46 | <163 | 582 | 433 | 970 |
| 2568-H-SC | 12.71 | 0.37 | 1.00 | 6.51 | 0.48 | 7.53 | <163 | 700 | 370 | 564 |
| 2512-J-SC | 17.35 | 2.40 | 0.91 | 7.00 | 2.04 | 9.56 | <163 | 2451 | 4925 | 1400 |
| 2515-J-SC | 15.08 | 1.04 | 1.15 | 6.14 | 2.78 | 9.36 | <163 | 2655 | 6214 | 1294 |
| 2517-J-SC | 10.20 | 1.10 | 1.00 | 5.70 | 1.66 | 8.62 | <163 | 2215 | 3871 | 726 |
| 2519-J-SC | 13.56 | 0.98 | 1.26 | 6.91 | 2.11 | 9.00 | 541 | 2434 | 3859 | 1012 |
| 2482-L-SC | 11.56 | 0.21 | 0.32 | 3.84 | 4.03 | 12.60 | <163 | 196 | 85 | 529 |
| 2484-L-SC | 11.83 | 0.26 | 0.50 | 2.90 | 4.10 | 10.15 | <163 | 114 | 94 | 1137 |
| 2486-L-SC | 16.22 | 0.70 | 1.13 | 4.81 | 1.71 | 9.20 | 908 | 534 | 415 | 2263 |
| 2488-L-SC | 13.05 | 0.45 | 0.69 | 4.99 | 2.45 | 12.32 | 475 | 364 | 273 | 906 |
| 2490-L-SC | 13.80 | 0.70 | 2.40 | 3.97 | 2.23 | 11.61 | 1415 | 1054 | 1334 | 1225 |
| 2601-M-SC | 21.81 | 0.44 | 1.01 | 7.73 | 3.98 | 11.70 | 808 | 561 | 557 | 2497 |
| 2605-M-SC | 21.37 | 0.20 | 0.40 | 2.74 | 0.83 | 5.58 | 222 | 509 | 224 | 1428 |
| 2609-M-SC | 41.77 | 0.47 | 0.84 | 7.19 | 4.03 | 6.20 | 549 | 457 | 841 | 5344 |
| 2613-M-SC | 31.77 | 0.68 | 1.18 | 9.02 | 3.40 | 7.31 | 547 | 645 | 717 | 2656 |
| 2492-N-SC | 6.70 | 1.15 | 0.51 | 2.66 | 0.11 | 10.85 | <163 | 473 | 58 | 135 |
| 2494-N-SC | 6.85 | 1.24 | 0.51 | 4.04 | 0.05 | 10.40 | <163 | 478 | 35 | 126 |
| 2496-N-SC | 8.57 | 0.99 | 0.43 | 3.14 | 0.27 | 11.64 | <163 | 462 | 59 | 174 |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 2498-N-SC | 9.25 | 0.47 | 0.51 | 3.18 | 0.84 | 12.05 | <163 | 479 | 249 | 342 |

*The MRL of total Sulfur by ICP is 163 mg $Kg^{-1}$.

**Table B-3: Trace metal content of salt cake (EPA Method 6010C)**

| Sample ID | As mg kg$^{-1}$ | Cd µg kg$^{-1}$ | Cr mg kg$^{-1}$ | Pb mg kg$^{-1}$ | Se mg kg$^{-1}$ |
|---|---|---|---|---|---|
| 2452-B-SC | 7.0 | 52.9 | 139.4 | 12.8 | 18.4 |
| 2468-B-SC | 16.6 | 70.9 | 120.0 | 11.0 | 4.5 |
| 2478-B-SC | 12.8 | 95.1 | 128.4 | 6.9 | 30.2 |
| 2454-B-SC | 29.9 | 69.1 | 110.6 | 10.0 | 14.4 |
| 2433-C-SC | 20.9 | 303.9 | 215.9 | 36.4 | 2.6 |
| 2435-C-SC | 23.6 | 388.9 | 264.1 | 42.1 | 4.0 |
| 2532--D-SC | 7.4 | 86.2 | 298.6 | 98.8 | 3.2 |
| 2536-D-SC | 12.9 | 111.7 | 273.2 | 30.0 | 13.1 |
| 2538-D-SC | 5.7 | 249.9 | 355.2 | 16.1 | 4.5 |
| 2540-D-SC | 10.1 | 463.9 | 285.2 | 17.8 | 4.4 |
| 2437-E-SC | 8.6 | 343.5 | 167.8 | 27.1 | 5.4 |
| 2439-E-SC | 8.6 | 195.4 | 179.7 | 25.5 | 5.5 |
| 2502-F-SC | 17.7 | 83.4 | 838.6 | 6.9 | 20.0 |
| 2504-F-SC | 7.9 | 95.3 | 285.4 | 6.8 | 9.7 |
| 2506-F-SC | 2.8 | 37.8 | 263.3 | 4.9 | 3.3 |
| 2508-F-SC | 7.0 | 59.9 | 135.3 | 7.4 | 7.4 |
| 2510-F-SC | 8.4 | 82.4 | 180.8 | 8.0 | 22.1 |
| 2046-H-SC | 18.5 | 524.6 | 783.0 | 90.6 | 7.5 |
| 2544-H-SC | 6.9 | 890.8 | 584.2 | 103.7 | 8.5 |
| 2555-H-SC | 7.6 | 696.7 | 489.7 | 42.6 | 8.3 |
| 2559-H-SC | 10.6 | 363.0 | 600.9 | 51.9 | 8.9 |
| 2568-H-SC | 26.8 | 1051.5 | 438.2 | 50.9 | 6.1 |
| 2512-J-SC | 15.2 | 581.7 | 461.1 | 101.4 | 10.3 |
| 2515-J-SC | 11.7 | 1048.2 | 478.5 | 314.1 | 8.6 |
| 2517-J-SC | 13.0 | 588.7 | 356.6 | 48.0 | 4.3 |
| 2519-J-SC | 13.1 | 502.1 | 439.0 | 172.7 | 9.2 |
| 2482-L-SC | 16.7 | 92.1 | 128.7 | 5.5 | 7.8 |
| 2484-L-SC | 7.5 | 74.0 | 206.4 | 4.7 | 11.2 |
| 2486-L-SC | 14.8 | 533.6 | 402.3 | 21.2 | 16.1 |
| 2488-L-SC | 9.6 | 79.7 | 320.1 | 19.2 | 10.2 |
| 2490-L-SC | 10.8 | 526.9 | 424.9 | 78.4 | 9.9 |
| 2601-M-SC | 16.3 | 342.6 | 253.9 | 46.4 | 14.4 |
| 2605-M-SC | 22.6 | 318.4 | 76.9 | 19.2 | 12.3 |
| 2609-M-SC | 38.3 | 849.9 | 304.1 | 48.4 | 25.6 |
| 2613-M-SC | 40.8 | 817.8 | 658.7 | 78.1 | 24.6 |
| 2492-N-SC | 3.0 | 64.4 | 225.6 | 6.0 | 2.4 |
| 2494-N-SC | 4.5 | 26.6 | 437.7 | 4.6 | 1.3 |
| 2496-N-SC | 6.8 | 19.7 | 139.2 | 6.0 | 2.4 |
| 2498-N-SC | 10.4 | 217.7 | 401.3 | 11.3 | 3.2 |

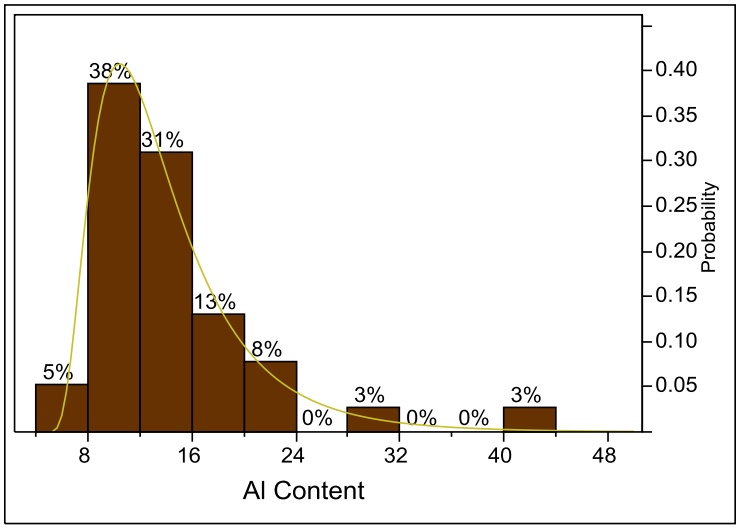


**Figure B-1: Total Al distribution in salt cake samples**

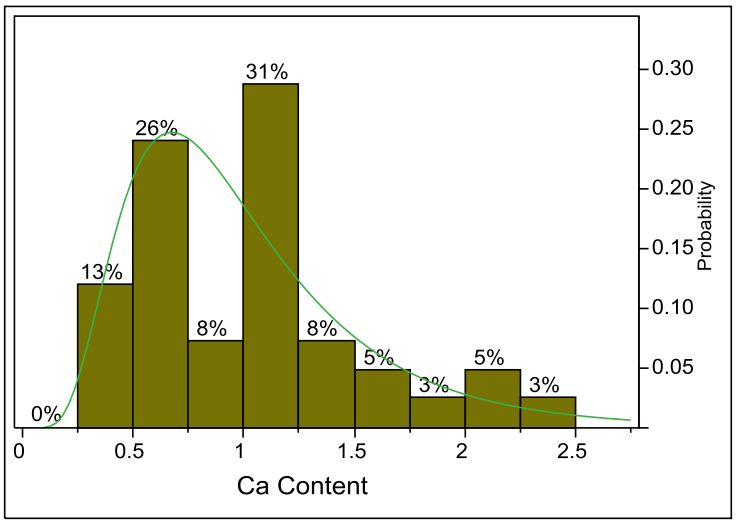


**Figure B-2: Total Ca distribution in salt cake samples**

**Table B-4: Total calcium content (%) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 1.3±0.3 | 1.8 | 1.0±1.7 |
| C | 1.9±0.28 | 4.4 | 1.7±2.8 |
| D | 0.55±0.07 | 0.66 | 0.47±0.64 |
| E | 2.1±0.12 | 3.1 | 1.9±2.1 |
| F | 0.77±0.39 | 1.3 | 0.37±1.4 |
| H | 1.1±0.11 | 1.3 | 1.0±1.3 |
| J | 1.1±0.16 | 1.3 | 0.91±1.3 |
| L | 1.0±0.83 | 2.1 | 0.32±2.4 |
| M | 0.86±0.34 | 1.4 | 0.40±1.2 |
| N | 0.49±0.04 | 0.55 | 0.43±0.51 |

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| All Facilities | 1.0±0.53 | 1.2 | 0.32±2.4 |

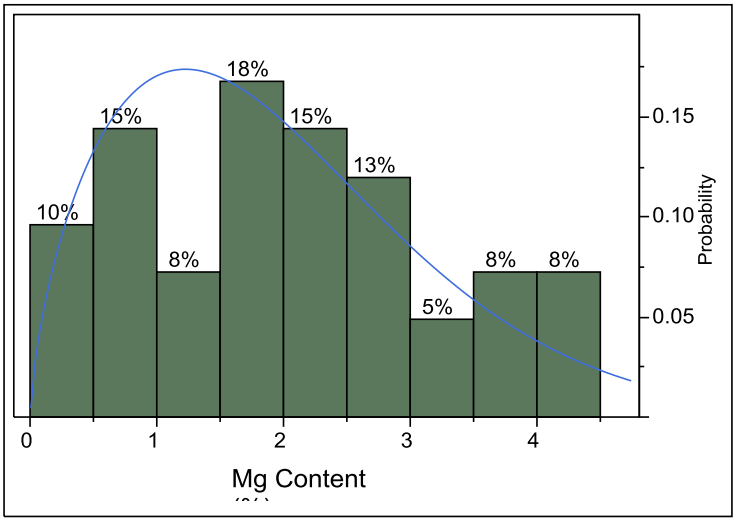


**Figure B-3: Total Mg content distribution in salt cake samples**

**Table B-5: Total magnesium content (%) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 1.5±0.24 | 1.9 | 1.2-1.7 |
| C | 2.4±0.11 | 3.4 | 2.3-2.5 |
| D | 2.3±0.94 | 3.8 | 1.7-3.7 |
| E | 3.3±0.4 | 6.9 | 3.0-3.5 |
| F | 2.2±0.9 | 3.3 | 1.1-3.0 |
| H | 0.66±0.15 | 0.85 | 0.48-0.86 |
| J | 2.2±0.46 | 2.9 | 1.7-2.8 |
| L | 2.9±1.1 | 4.3 | 1.7-4.1 |
| M | 3.1±1.5 | 5.5 | 0.83-4.0 |
| N | 0.32±0.36 | 0.89 | 0.05-0.84 |
| All Facilities | 2.0±1.2 | 2.4 | 0.05-4.1 |

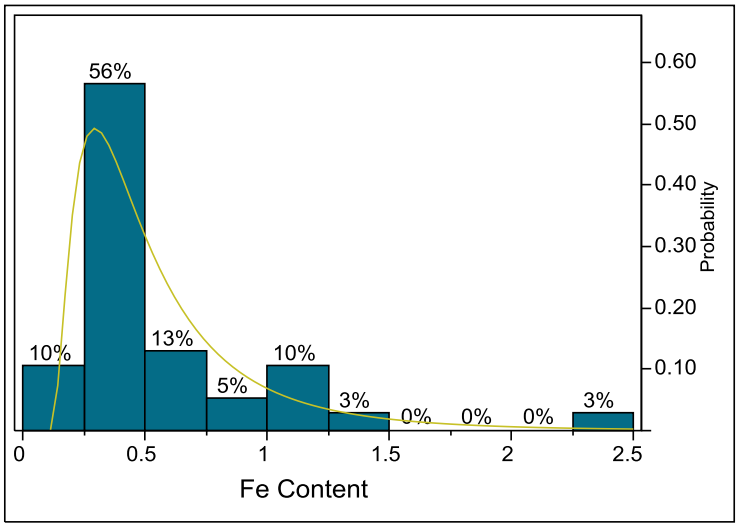


**Figure B-4: Total Fe distribution in salt cake samples**

**Table B-6: Total iron content (%) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 0.36±0.07 | 0.47 | 0.29-0.45 |
| C | 0.39±0.02 | 0.58 | 0.37-0.4 |
| D | 0.48±0.08 | 0.60 | 0.37-0.54 |
| E | 0.34±0.03 | 0.59 | 0.32-0.36 |
| F | 0.49±0.5 | 1.1 | 0.15-1.4 |
| H | 0.37±0.08 | 0.46 | 0.29-0.49 |
| J | 1.9±0.7 | 2.5 | 0.98-2.4 |
| L | 0.46±0.2 | 0.75 | 0.21-0.7 |
| M | 0.45±0.2 | 0.76 | 0.20-0.68 |
| N | 0.96±0.3 | 1.5 | 0.47-1.2 |
| All Facilities | 0.58±0.4 | 0.72 | 0.15-2.4 |

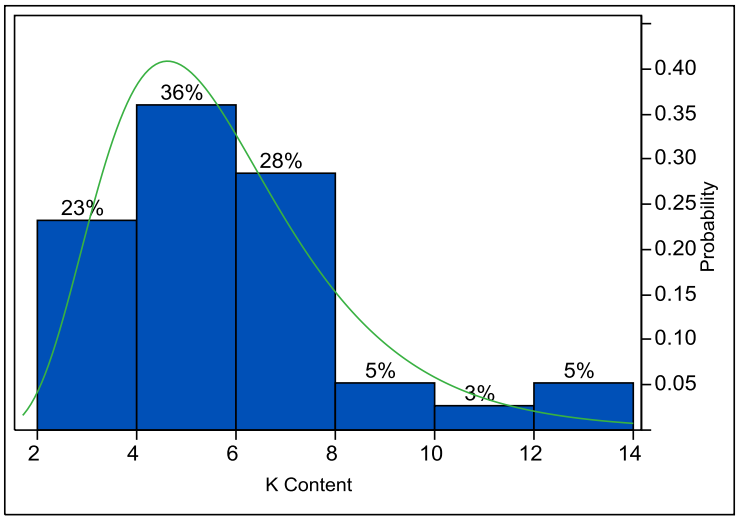


**Figure B-5: Total potassium content overall distribution**

**Table B-7: Total K content (%) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 5.6±1.4 | 7.9 | 4.4-7.6 |
| C | 4.0±0.37 | 7.4 | 3.8-4.3 |
| D | 11±2.0 | 14 | 8.5-13 |
| E | 5.8±0.55 | 11 | 5.4-6.2 |
| F | 4.9±1.6 | 6.9 | 3.0-7.4 |
| H | 6.1±0.59 | 6.9 | 5.4-6.9 |
| J | 6.4±0.63 | 7.4 | 5.7-7.0 |
| L | 4.1±0.84 | 5.1 | 2.9-5.0 |
| M | 6.7±2.7 | 11 | 2.7-9.0 |
| N | 3.3±0.57 | 4.2 | 2.7-4.0 |
| All Facilities | 5.9±2.4 | 6.6 | 2.7-13 |

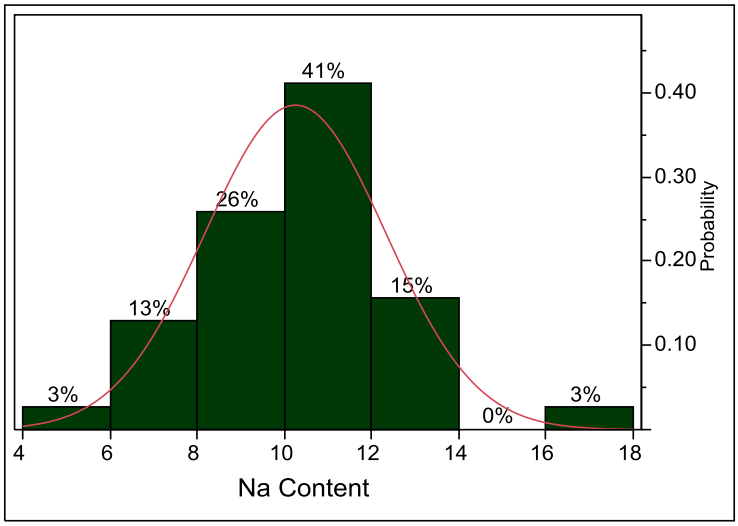


**Figure B-6: Total sodium distribution in salt cake samples**

**Table B-8: Total Na content (%) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 9.3±1.4 | 11 | 7.3-11 |
| C | 11±0.51 | 16 | 11-12 |
| D | 11±1.4 | 13 | 9.2-12 |
| E | 11±0.25 | 13 | 10-11 |
| F | 13±2.2 | 15 | 11-17 |
| H | 8.5±0.83 | 9.5 | 7.5-9.7 |
| J | 9.1±0.41 | 9.8 | 8.6-9.6 |
| L | 11±1.5 | 13 | 9.2-13 |
| M | 7.7±2.8 | 12 | 5.6-12 |
| N | 11±0.75 | 12 | 10-12 |
| All Facilities | 10±2.1 | 11 | 5.6-17 |

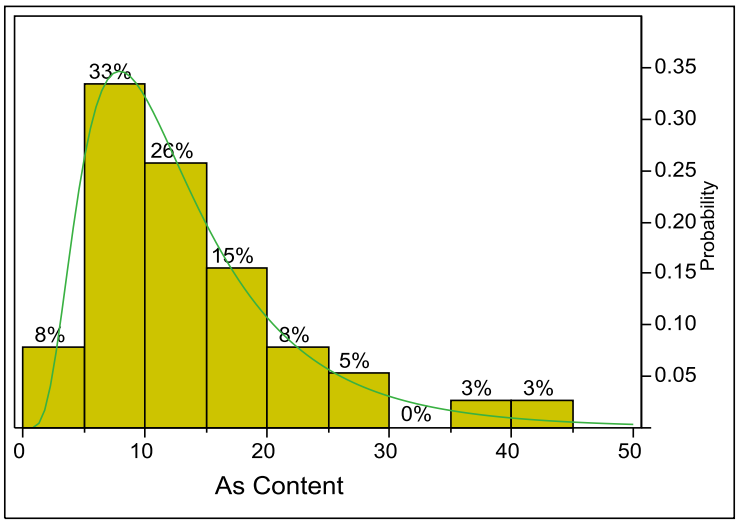


**Figure B-7: Total arsenic distribution in salt cake samples**

**Table B-9: Total As content (mg kg$^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 17±9.7 | 32 | 7-30 |
| C | 22±1.9 | 39 | 21-24 |
| D | 9.0±3.2 | 14 | 5.7-13 |
| E | 8.6±0 | 8.6 | 8.0-9.0 |
| F | 8.8±5.5 | 16 | 2.8-18 |
| H | 14±8.5 | 25 | 6.9-27 |
| J | 13±1.4 | 16 | 12-15 |
| L | 12±3.8 | 17 | 7.5-17 |
| M | 30±12 | 48 | 16-41 |
| N | 6.2±3.2 | 11 | 3.0-10 |
| All Facilities | 14±8.8 | 17 | 2.8-41 |

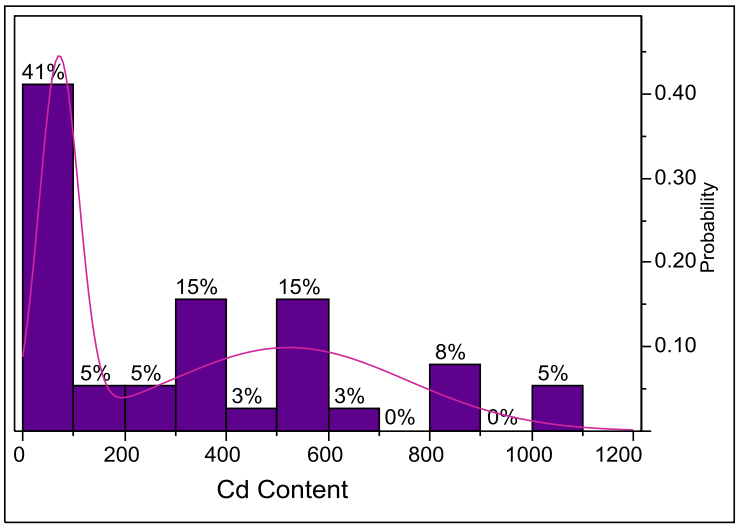

**Figure B-8: Total cadmium distribution in salt cake samples**

**Table B-10: Total Cd content (µg kg$^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 72±17 | 100 | 53±95 |
| C | 350±60 | 890 | 300±390 |
| D | 230±170 | 500 | 86±460 |
| E | 270±100 | 1,200 | 190±340 |
| F | 72±23 | 100 | 38±95 |
| H | 700±280 | 1,100 | 360±1,000 |
| J | 680±250 | 1,000 | 500±1,000 |
| L | 260±250 | 570 | 74±530 |
| M | 580±290 | 1,000 | 320±850 |
| N | 82±93 | 230 | 20±230 |
| All Facilities | 330±300 | 430 | 20±1,000 |

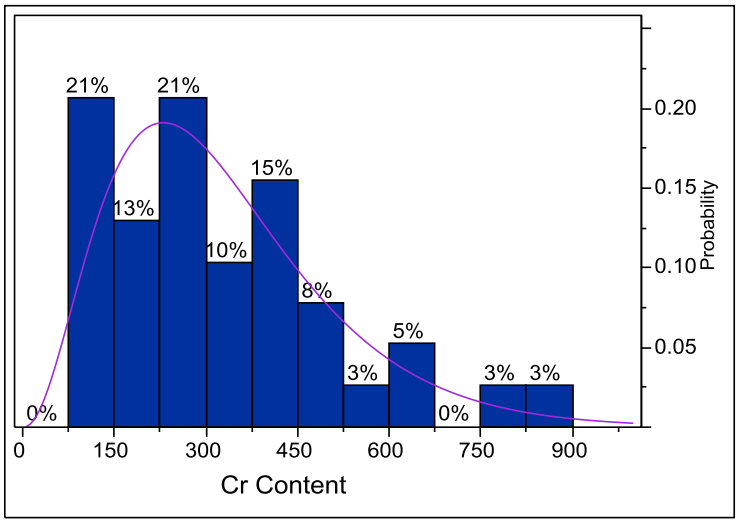


**Figure B-9: Total chromium distribution in salt cake samples**

**Table B-11: Total Cr content (mg kg$^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 130±12 | 140 | 110-140 |
| C | 240±34 | 550 | 220-260 |
| D | 300±36 | 360 | 270-360 |
| E | 170±8.4 | 250 | 170-180 |
| F | 340±280 | 690 | 140-840 |
| H | 580±130 | 740 | 440-780 |
| J | 430±54 | 520 | 360-480 |
| L | 300±130 | 450 | 130-430 |
| M | 320±240 | 710 | 76.9-660 |
| N | 300±140 | 530 | 140-440 |
| All Facilities | 330±190 | 390 | 77-840 |

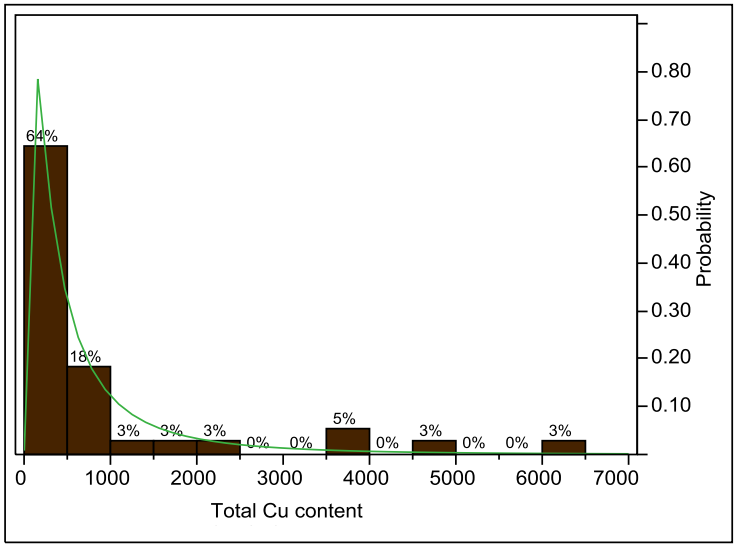


**Figure B-10: Total copper distribution in salt cake samples**

**Table B-12: Total Cu content (mg kg$^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 150±60 | 240 | 88-220 |
| C | 1,900±230 | 3800 | 1700-2,000 |
| D | 460±75 | 580 | 370-530 |
| E | 470±120 | 1600 | 380-550 |
| F | 250±94 | 370 | 150-400 |
| H | 430±75 | 520 | 370-560 |
| J | 4,700±1,100 | 6500 | 3,900-6,200 |
| L | 440±520 | 1100 | 86-1,300 |
| M | 590±270 | 1000 | 220-840 |
| N | 100±100 | 260 | 35-250 |
| All facilities | 880 | 1300 | 35-6,200 |

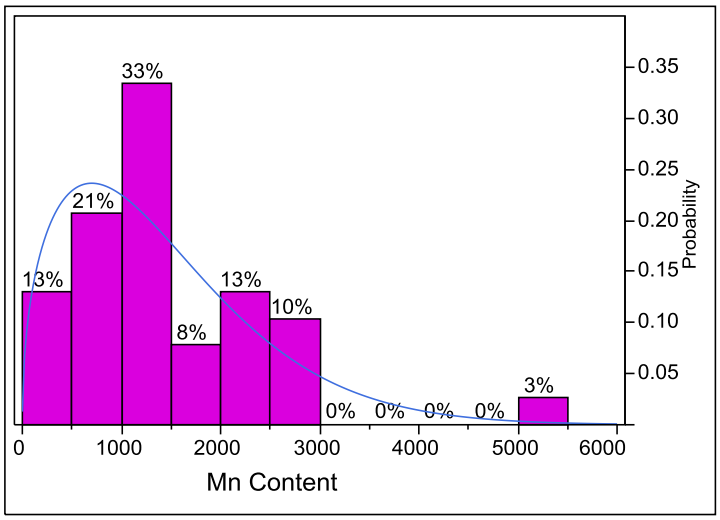


**Figure B-11: Total manganese distribution in salt cake samples**

**Table B-13: Total Mn content (mg kg$^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 2,400±460 | 3,600 | 1,900-2,900 |
| C | 1,000±8.3 | 1,100 | 1,000-1,100 |
| D | 920±290 | 1,400 | 590-1,200 |
| E | 1,500±310 | 4,300 | 1,300-1,800 |
| F | 1,800±1,000 | 3,100 | 160-3,000 |
| H | 930±350 | 1,400 | 560-1,400 |
| J | 1,100±300 | 1,600 | 720-1,400 |
| L | 1,200±650 | 2,000 | 530-2,300 |
| M | 3,000±1,700 | 5,600 | 1,400-5,300 |
| N | 190±100 | 350 | 130-340 |
| All Facilities | 1,400±1,000 | 1,700 | 130-5,300 |

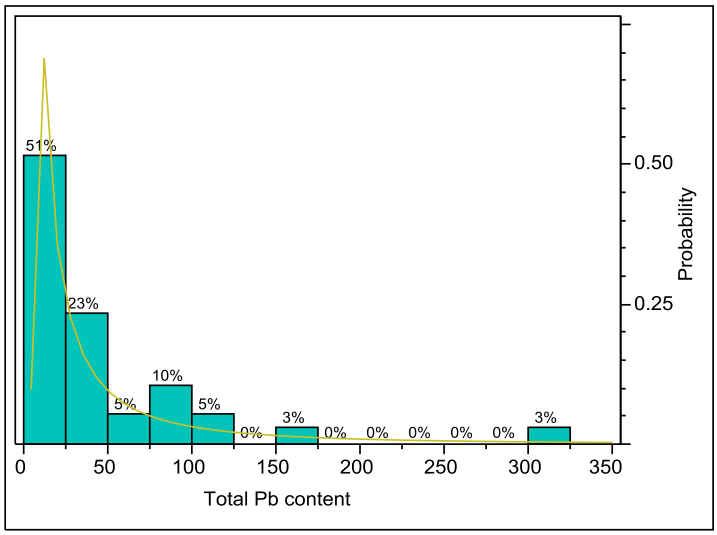


**Figure B-12: Total lead distribution in salt cake samples**

**Table B-14: Total Pb content (mg kg$^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 10±2.5 | 14 | 6.9-13 |
| C | 39±4.0 | 76 | 36-42 |
| D | 41±39 | 100 | 16-99 |
| E | 26±1.1 | 36 | 25-27 |
| F | 6.8±1.2 | 8.2 | 4.9-8.0 |
| H | 68±27 | 100 | 42-100 |
| J | 160±120 | 340 | 48-310 |
| L | 26±30 | 63 | 4.7-78 |
| M | 48±24 | 86 | 19-78 |
| N | 7.0±3.0 | 11 | 4.6-11 |
| All Facilities | 43±58 | 62 | 4.6-310 |

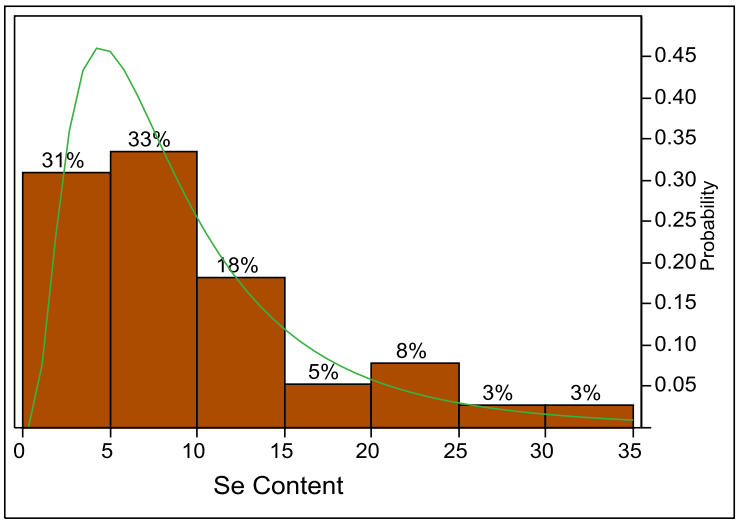


**Figure B-13: Total selenium content distribution in salt cake samples**

**Table B-15: Total Se content (mg kg$^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 17±11 | 33 | 4.5-30 |
| C | 3.3±0.99 | 12 | 2.6-4.0 |
| D | 6.3±4.6 | 14 | 3.2-13 |
| E | 5.5±0.1 | 6.1 | 5.4-5.5 |
| F | 13±8.2 | 23 | 3.3-22 |
| H | 7.9±1.1 | 9.2 | 6.1-8.9 |
| J | 8.1±2.6 | 12 | 4.3-10 |
| L | 11±3.1 | 15 | 7.8-16 |
| M | 19±6.9 | 30 | 12-25 |
| N | 2.3±0.8 | 3.6 | 1.3-3.2 |
| All Facilities | 9.9±7.1 | 12 | 1.3-30 |

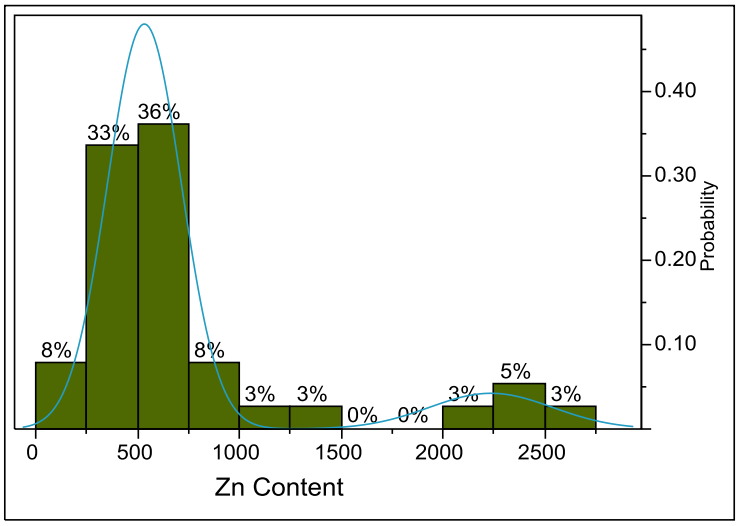


**Figure B-14: Total zinc content distribution in salt cake samples**

**Table B-16: Total Zn content (mg kg$^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 330±68 | 440 | 230-370 |
| C | 1,200±380 | 4,600 | 900-1,400 |
| D | 570±72 | 690 | 490-650 |
| E | 630±120 | 1,700 | 550-710 |
| F | 500±91 | 610 | 380-620 |
| H | 710±99 | 830 | 580-830 |
| J | 2,400±180 | 2,700 | 2,200-2,700 |
| L | 450±370 | 910 | 110-1100 |
| M | 540±80 | 670 | 460-640 |
| N | 470±7.9 | 480 | 460-480 |
| All Facilities | 750±620 | 950 | 110-2,600 |

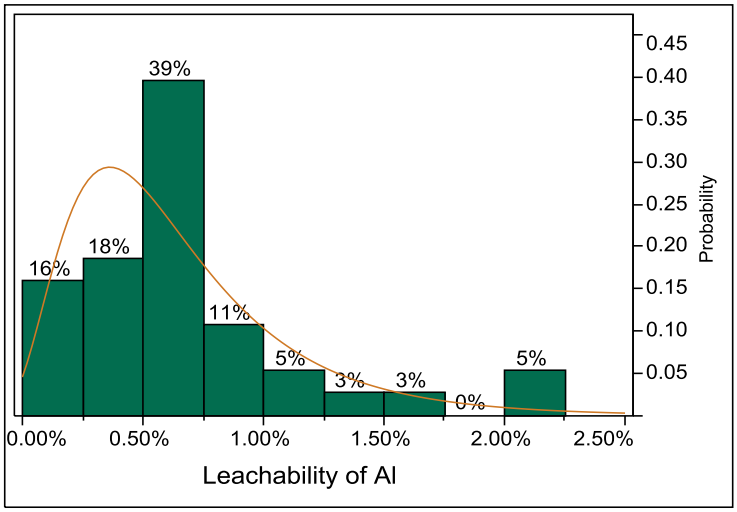


**Figure B-15: Distribution of leachability of aluminum in salt cake samples**

**Table B-17: Correlation Coefficients between Metals in Salt Cakes**

| Metal | | Pearson product moment | | Spearman rank order | |
|---|---|---|---|---|---|
| | | Correlation coefficient | *P* value | Correlation coefficient | *P* value |
| Al | As | 0.72 | 0.000001 | 0.53 | 0.001 |
| | Cd | 0.55 | 0.001 | 0.63 | 0.0001 |
| | Cr | 0.32 | 0.05 | 0.40 | 0.01 |
| | Cu | 0.082 | 0.6 | 0.44 | 0.005 |
| | Mn | 0.70 | 0.000001 | 0.55 | 0.0003 |
| | Pb | 0.23 | 0.2 | 0.53 | 0.0005 |
| | Se | 0.61 | 0.0001 | 0.67 | 0.00001 |
| | Zn | 0.039 | 0.8 | 0.32 | 0.05 |
| As | Cd | 0.45 | 0.004 | 0.48 | 0.002 |
| | Mn | 0.56 | 0.0002 | 0.44 | 0.005 |
| | Pb | 0.10 | 0.5 | 0.39 | 0.02 |
| | Se | 0.46 | 0.003 | 0.40 | 0.01 |
| Cd | Cr | 0.51 | 0.001 | 0.60 | 0.00005 |
| | Cu | 0.51 | 0.0009 | 0.74 | 0.000001 |
| | Pb | 0.65 | 0.000001 | 0.82 | 0.000001 |
| | Zn | 0.52 | 0.000001 | 0.69 | 0.000001 |
| Cr | Pb | 0.41 | 0.009 | 0.57 | 0.0002 |
| | Zn | 0.32 | 0.05 | 0.60 | 0.000001 |
| Cu | Pb | 0.80 | 0.000001 | 0.85 | 0.000001 |
| | Zn | 0.97 | 0.000001 | 0.78 | 0.000001 |
| Mn | Se | 0.74 | 0.000001 | 0.74 | 0.000001 |

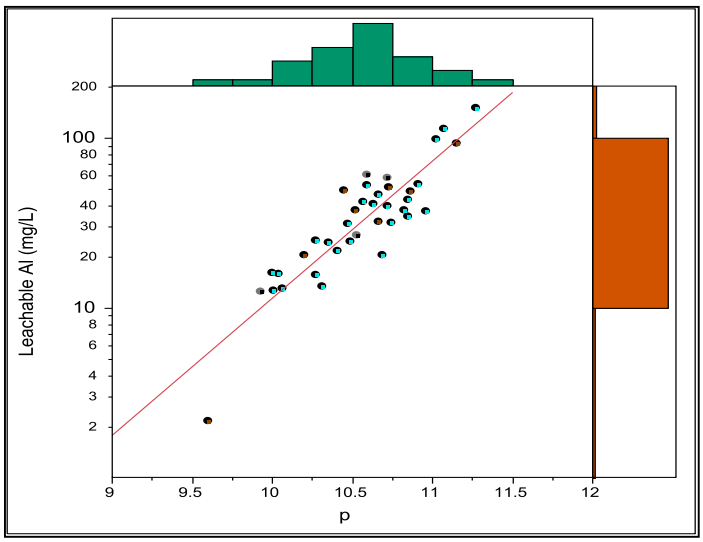

**Figure B-16: Correlation between leachable Al and pH in leachate of salt cake**

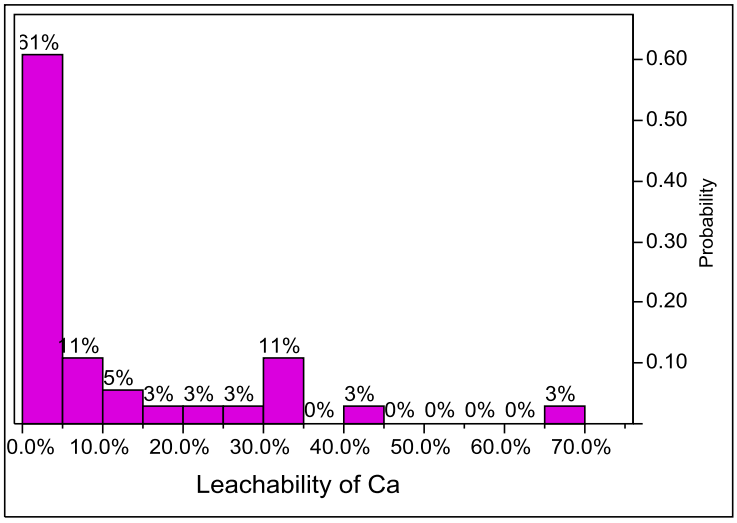


**Figure B-17: Distribution of leachability of calcium in salt cake samples**

**Table B-18: Leachable Ca (mg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 55±77 | 180 | 0.89-170 |
| C | 44±37 | 380 | 17-70 |
| D | 3.5±1.3 | 5.5 | 1.8-5.0 |
| E | 300±66 | 900 | 260-350 |
| F | 71±95 | 190 | 12-230 |
| H | 12±4.4 | 17 | 7.3-19 |
| J | 220±130 | 430 | 66-390 |
| L | 17±22 | 45 | 1.1-55 |
| M | 54±53 | 180 | 3.9-110 |
| N | 0.82±0.36 | 1.4 | 0.33-1.2 |
| All Facilities | 65-100 | 100 | 0.33-390 |

**Table B-19: Leachable Mg (μg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | <10 | 10 | <10 |
| C | 210±130 | 1,400 | 110-300 |
| D | 70±80 | 190 | 10-160 |
| E | <10 | 10 | <10 |
| F | 10±10 | 20 | 10-30 |
| H | <10 | 10 | <10 |
| J | 660±880 | 2,100 | 10-2,000 |
| L | 20±10 | 30 | 10-40 |
| M | 20±10 | 50 | 10-30 |
| N | 70±120 | 260 | 10-250 |
| All Facilities | 110±320 | 210 | 10-2,000 |

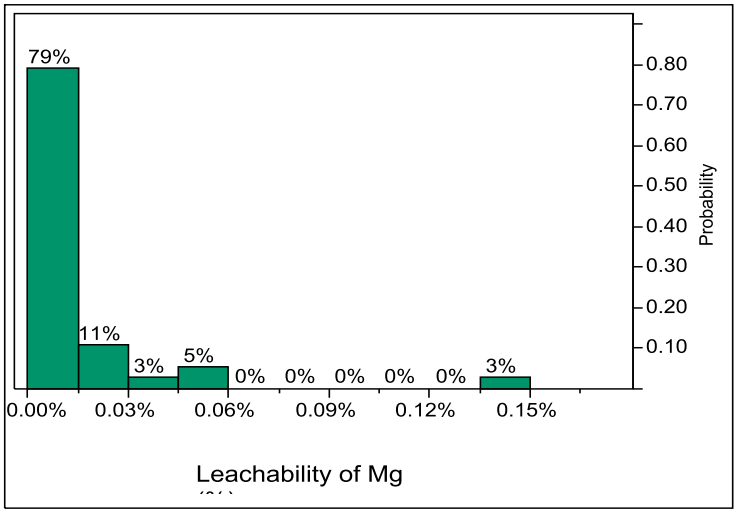


**Figure B-18: Distribution of leachability of Mg in salt cake samples**

**Table B-20: Leachable K (mg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 2,700±500 | 3,600 | 2,300-3,500 |
| C | 1,900±100 | 2,900 | 1,900-2,000 |
| D | 4,800±900 | 6,200 | 3,600-5,600 |
| E | 2,600±300 | 5,700 | 2,400-2,800 |
| F | 2,100±700 | 3,000 | 1,300-3,200 |
| H | 2,500±200 | 2,800 | 2,300-2,800 |
| J | 2,600±400 | 3,200 | 2,200-3,000 |
| L | 1,700±300 | 2,100 | 1,300-2,100 |
| M | 2,500±1,000 | 5,200 | 1,300-3,200 |
| N | 1,400±200 | 1,700 | 1,100-1,700 |
| All Facilities | 2,500±1,000 | 2,800 | 1,100-5,600 |

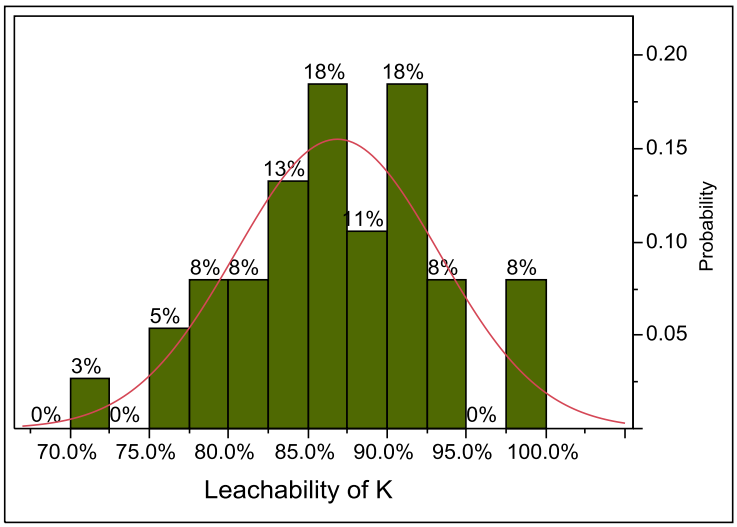


**Figure B-19: Distribution of leachability of K in salt cake samples**

**Table B-21: Leachable Na (mg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 3,000±300 | 3,500 | 2,500-3,200 |
| C | 3,300±100 | 4,200 | 3,200-3,300 |
| D | 2,800±200 | 3,100 | 2,600-3,000 |
| E | 2,800±30 | 3,000 | 2,700-2,800 |
| F | 3,200±200 | 3,400 | 3,100-3,500 |
| H | 2,400±100 | 2,500 | 2,300-2,500 |
| J | 2,500±70 | 2,600 | 2,400-2,600 |
| L | 2,700±200 | 2,900 | 2,400-2,900 |
| M | 2,000±500 | 3,200 | 1,700-2,600 |
| N | 2,800±300 | 3,300 | 2,500-3,100 |
| All Facilities | 2,700±400 | 2,900 | 1,700-3,500 |

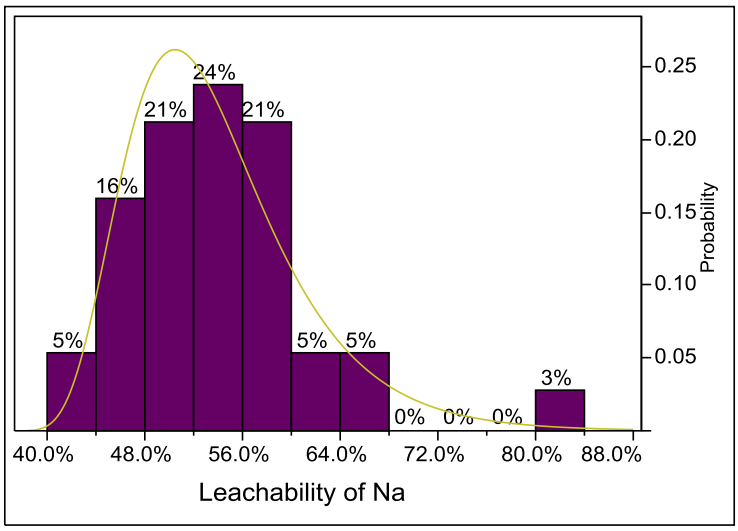


**Figure B-20: Distribution of leachability of Na in salt cake samples**

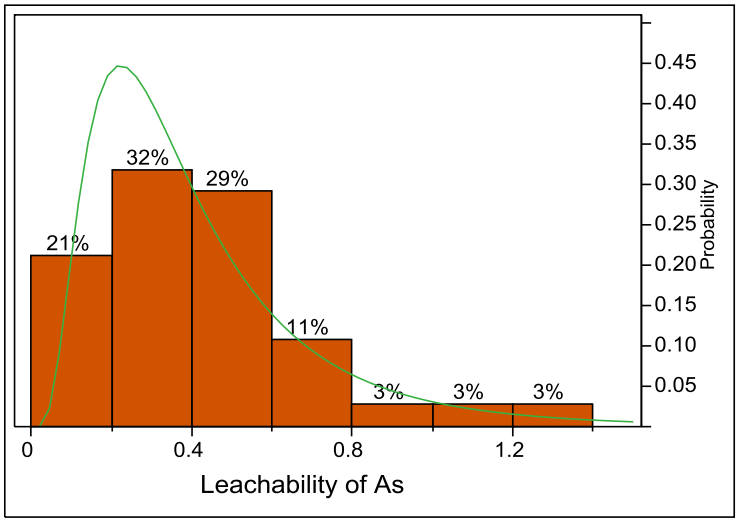


**Figure B-21: Distribution of leachability of As in salt cake samples**

**Table B-22: Leachable arsenic (µg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 2.3±0.9 | 3.7 | <1.8-3.6 |
| C | <1.8 | 1.8 | <1.8 |
| D | <1.8 | 1.8 | <1.8 |
| E | 2.7±0.15 | 4.1 | 2.6-2.8 |
| F | <1.8 | 1.8 | <1.8 |
| H | <1.8 | 1.9 | <1.8-1.9 |
| J | 2.7±1.0 | 4.3 | <1.8-3.6 |
| L | <1.8 | 1.8 | <1.8 |
| M | <1.8 | 1.8 | <1.8 |
| N | <1.8 | 1.8 | <1.8 |
| All Facilities | 2.0± | 2.2 | <1.8-3.6 |

**Table B-23: Leachable Cr (µg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 1.3±1.0 | 3.0 | 0.75-2.6 |
| C | < 0.75 | 0.75 | < 0.75 |
| D | 3.9±6.6 | 14 | 0.75-14 |
| E | <0.75 | 0.75 | <0.75 |
| F | 0.89±0.31 | 1.3 | 0.75-1.4 |
| H | 1.8±1.7 | 3.9 | 0.75-4.6 |
| J | 1.0±0.54 | 1.9 | 0.75-1.8 |
| L | 1.2±0.76 | 2.1 | 0.75-2.5 |
| M | <0.75 | 0.75 | <0.75 |
| N | 1.6±1.7 | 4.4 | 0.75-4.2 |
| All Facilities | 1.5±2.3 | 2.2 | 0.75-4.6 |

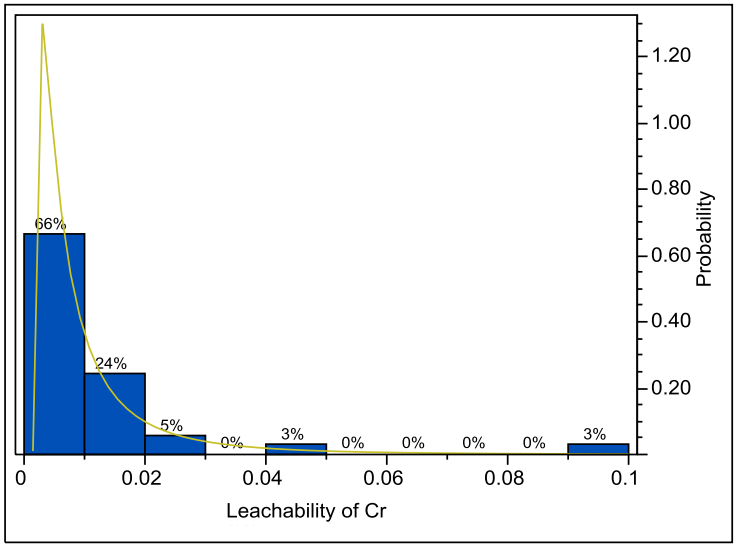


**Figure B-22: Distribution of leachability of Cr in salt cake samples**

**Table B-24: Leachable Cu (μg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 10±6.2 | 20 | 7.1-19 |
| C | 460±640 | 6,200 | 7.1-91 |
| D | 140±13 | 34 | 7.1-34 |
| E | 150±10 | 11 | 7.1-22 |
| F | 200±24 | 50 | 20-61 |
| H | 8.9±3.9 | 14 | 7.1-16 |
| J | 290±490 | 1,000 | 16-1,000 |
| L | 79±110 | 220 | 7.1-270 |
| M | 4.2±3.1 | 12 | <7.1 |
| N | 8.7±3.8 | 15 | 7.1-14 |
| All Facilities | 73±220 | 150 | 7.1-1,000 |

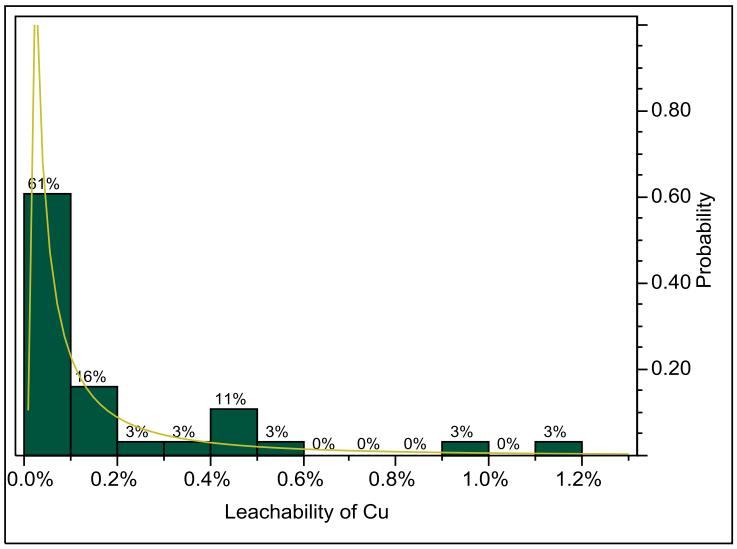

**Figure B-23: Distribution of leachability of Cu in salt cake samples**

**Table B-25: Leachable Mn (µg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 20±0.97 | 22 | 20-22 |
| C | 20±0.42 | 24 | 20-21 |
| D | 21±3.0 | 26 | 18-25 |
| E | 18±0.14 | 19 | 18-18 |
| F | 19±1.5 | 21 | 17-21 |
| H | 19±1.4 | 21 | 18-21 |
| J | 18±1.0 | 19 | 16-19 |
| L | 19±0.88 | 20 | 18-20 |
| M | 19±1.2 | 23 | 18-21 |
| N | 19±1.2 | 21 | 18-20 |
| All Facilities | 19±1.6 | 20 | 16-25 |

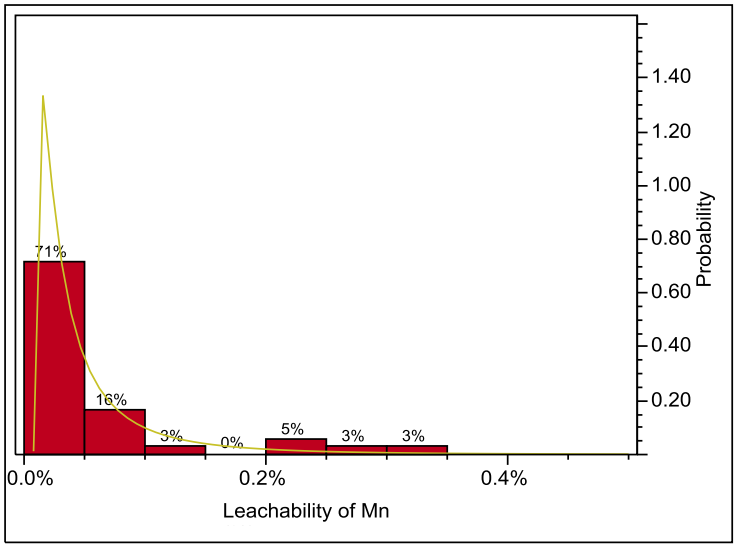


**Figure B-24: Distribution of leachability of Mn in salt cake samples**

**Table B-26: Leachable Pb (µg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 4.0±1.8 | 6.8 | 2.5-5.6 |
| C | <2.5 | 2.5 | <2.5 |
| D | 6.6±4.0 | 13 | 2.5-10 |
| E | <2.5 | 2.5 | <2.5 |
| F | <2.5 | 2.5 | <2.5 |
| H | <2.5 | 2.5 | <2.5 |
| J | 2.6±0.1 | 2.7 | 2.5-2.7 |
| L | 2.6±0.3 | 3.0 | 2.5-3.2 |
| M | 4.4±3.3 | 13 | 2.5-8.3 |
| N | <2.5 | 2.5 | <2.5 |
| All Facilities | 3.3±2.0 | 3.9 | 2.5-10 |

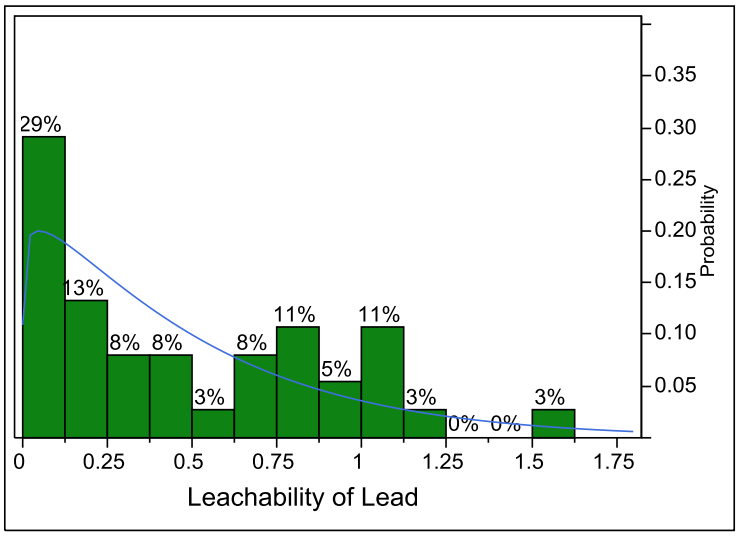


**Figure B-25: Distribution of leachability of Pb in salt cake samples**

**Table B-27: Leachable Se (µg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 120±64 | 223 | 37-190 |
| C | <10 | 10 | <10 |
| D | <10 | 10 | <10 |
| E | <10 | 10 | <10 |
| F | 27±28 | 61 | 10-75 |
| H | 10±1.0 | 12 | 10-12 |
| J | 15±8.9 | 29 | 10-29 |
| L | 10±0.36 | 11 | 10-11 |
| M | <10 | 10 | <10 |
| N | <10 | 10 | <10 |
| All Facilities | 24±40 | 38 | 10-190 |

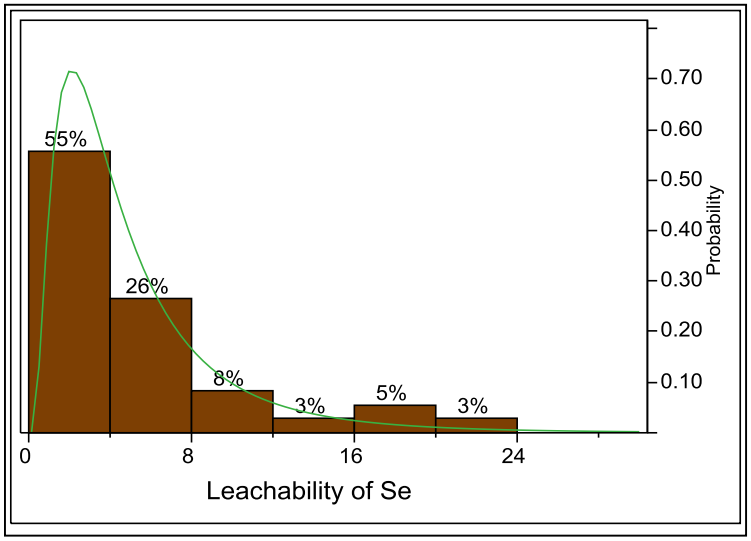


**Figure B-26: Distribution of leachability of Se in salt cake samples**

**Table B-28: Leachable Zinc (μg $L^{-1}$) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | <5.2 | 5.2 | <5.2 |
| C | 57±74 | 72 | 5.2-110 |
| D | <5.2 | 5.2 | <5.2 |
| E | <5.2 | 5.2 | <5.2 |
| F | 37±63 | 110 | 5.2-150 |
| H | 47±27 | 81 | 11-68 |
| J | 61±9.4 | 210 | 5.2-200 |
| L | 26±18 | 48 | 5.2-47 |
| M | 5.9±1.1 | 8.9 | 5.2-7.3 |
| N | 5.9±1.0 | 7.2 | 5.2-7.1 |
| All Facilities | 26±43 | 40 | 5.2-200 |

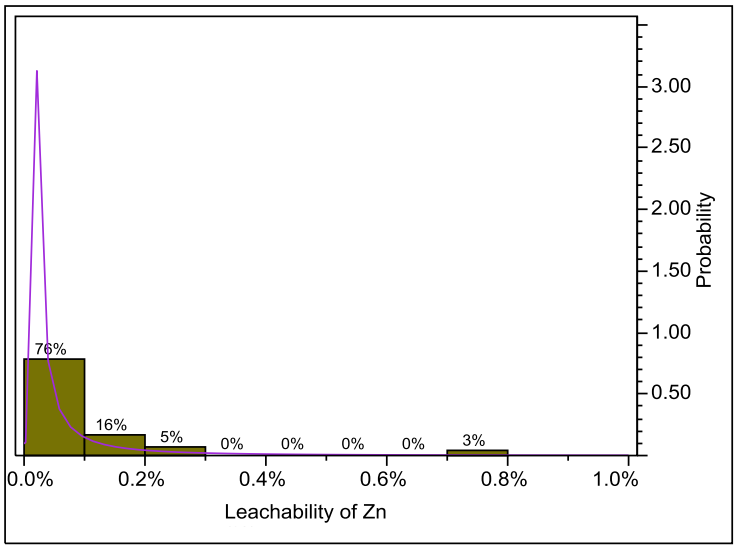


**Figure B-27: Distribution of leachability of Zn in salt cake samples**

**Table B-29: TCLP results for Al (mg $L^{-1}$) by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 150±280 | 590 | 0.95-570 |
| C | 1.1±0.21 | 3.0 | 0.95-1.2 |
| D | 5.7±3.7 | 12 | 2.5-11 |
| E | 4.5±1.9 | 22 | 3.1-5.9 |
| F | 120±230 | 400 | 4.0-520 |
| H | 180±30 | 220 | 150-240 |
| J | 180±140 | 410 | 1.6-330 |
| L | 18±20 | 42 | 2.1-51 |
| M | 1.5±0.72 | 3.3 | 0.95-2.3 |
| N | 110±19 | 140 | 99-140 |
| All Facilities | 90±140 | 140 | 0.95-570 |

**Table B-30: TCLP results for Ag (mg $L^{-1}$) by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | <MRL | <MRL | <MRL |
| C | <MRL | <MRL | <MRL |
| D | <MRL | <MRL | <MRL |
| E | <MRL | <MRL | <MRL |
| F | <MRL | <MRL | <MRL |
| H | <MRL | <MRL | <MRL |
| J | <MRL | <MRL | <MRL |
| L | <MRL | <MRL | <MRL |
| M | <MRL | <MRL | <MRL |
| N | <MRL | <MRL | <MRL |
| All Facilities | <MRL | <MRL | <MRL |

*All of samples had Ag concentration below the MRL (<0.008 mg $L^{-1}$)

**Table B-31: TCLP results for As (mg $L^{-1}$) by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 0.03±0.004 | 0.03 | 0.02-0.03 |
| C | 0.03±0.01 | 0.11 | 0.02-0.04 |
| D | 0.03±0.02 | 0.06 | 0.02-0.06 |
| E | 0.02 | 0.02 | 0.02 |
| F | 0.03±0.02 | 0.05 | 0.02-0.06 |
| H | 0.02±0.002 | 0.03 | 0.02-0.03 |
| J | 0.04±0.03 | 0.09 | 0.02-0.08 |
| L | 0.02±0 | 0.02 | 0.02-0.02 |
| M | 0.03±0.01 | 0.05 | 0.02-0.04 |
| N | 0.03±0.01 | 0.05 | 0.02-0.05 |
| All Facilities | 0.03±0.01 | 0.03 | 0.02-0.08 |

**Table B-32: TCLP results for Ba (mg $L^{-1}$) by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 7.2±4.0 | 14 | 2.0-11 |
| C | 5.5±0.6 | 11 | 5.1-5.9 |
| D | 2.8±1.2 | 4.7 | 1.3-3.9 |
| E | 2.9±1.6 | 18 | 1.7-4.1 |
| F | 1.7±1.3 | 3.3 | 0.6-3.7 |
| H | 16±6.2 | 23 | 8.9-25 |
| J | 9.5±3.8 | 16 | 6.1-15 |
| L | 3.6±2.2 | 6.2 | 0.6-5.8 |
| M | 0.7±0.15 | 1.1 | 0.6-0.86 |
| N | 14±21 | 48 | 1.2-46 |
| All Facilities | 6.8±8.5 | 9.6 | 0.6-46 |

**Table B-33: TCLP results for Cd (mg $L^{-1}$) by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 0.07±0.004 | 0.01 | 0.004-0.01 |
| C | 0.01±0.01 | 0.06 | 0.004-0.01 |
| D | 0.01±0.01 | 0.02 | 0.004-0.02 |
| E | 0.004±0 | 0.004 | 0.004 |
| F | 0.01±0.01 | 0.02 | 0.004-0.02 |
| H | 0.01±0.01 | 0.02 | 0.004-0.02 |
| J | 0.02±0.02 | 0.04 | 0.004-0.04 |
| L | 0.004±0.001 | 0.005 | 0.004-0.01 |
| M | 0.07±0.01 | 0.02 | 0.004-0.01 |

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| N | 0.01±0.01 | 0.02 | 0.004-0.02 |
| All Facilities | 0.01±0.01 | 0.01 | 0.004-0.04 |

**Table B-34: TCLP results for Cr (mg $L^{-1}$) by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 0.06±0.07 | 0.17 | 0.02-0.2 |
| C | 0.02±0.002 | 0.04 | 0.02-0.02 |
| D | 0.03±0.01 | 0.04 | 0.02-0.04 |
| E | 0.02 | 0.02 | 0.02-0.02 |
| F | 0.07±0.1 | 0.19 | 0.02-0.24 |
| H | 0.1±0.03 | 0.13 | 0.07-0.13 |
| J | 0.19±0.19 | 0.49 | 0.02-0.46 |
| L | 0.02±0.002 | 0.02 | 0.02-0.03 |
| M | 0.03±0.01 | 0.04 | 0.02-0.03 |
| N | 0.06±0.02 | 0.09 | 0.04-0.08 |
| All Facilities | 0.06±0.08 | 0.09 | 0.02-0.46 |

**Table B-35: TCLP results for Pb (mg $L^{-1}$) by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 0.06±0.09 | 0.20 | 0.01-0.19 |
| C | 0.02±0.004 | 0.06 | 0.01-0.02 |
| D | 0.02±0.02 | 0.05 | 0.01-0.05 |
| E | 0.014±0.0001 | 0.02 | 0.01-0.01 |
| F | 0.04±0.05 | 0.09 | 0.01-0.12 |
| H | 0.63±0.40 | 1.1 | 0.17-1.2 |
| J | 2.0±1.5 | 4.4 | 0.01-3.3 |
| L | 0.03±0.01 | 0.04 | 0.01-0.04 |
| M | 0.02±0.01 | 0.04 | 0.01-0.03 |
| N | 0.02±0.02 | 0.05 | 0.01-0.05 |
| All Facilities | 0.32±0.77 | 0.57 | 0.01-3.3 |

**Table B-36: TCLP results for Se (mg $L^{-1}$) by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 0.06±0.09 | 0.76 | 0.02-0.72 |
| C | 0.02±0.004 | 0.10 | 0.04-0.05 |
| D | 0.02±0.02 | 0.08 | 0.03-0.08 |
| E | 0.01±0.0001 | 0.18 | 0.02-0.04 |
| F | 0.04±0.05 | 0.18 | 0.02-0.18 |
| H | 0.63±0.40 | 0.03 | 0.02-0.03 |
| J | 2.0±1.5 | 0.06 | 0.02-0.06 |
| L | 0.03±0.01 | 0.06 | 0.03-0.07 |
| M | 0.02±0.01 | 0.07 | 0.02-0.05 |
| N | 0.02±0.02 | 0.06 | 0.02-0.06 |
| All Facilities | 0.32±0.77 | 0.10 | 0.02-0.72 |

**Table B-37: Aluminum mineral phase (%) in salt cake**

| sample ID | Al | Al N | $Al_{2.85}O_{3.45}N_{0.55}$ | $Al_{2.81}O_{3.56}N_{0.44}$ | $Al_{2.78}O_{3.65}N_{0.35}$ | total AlN | $Al_2O_3$ | $Al_{2.667}O_4$ | total $Al_2O_3$ | $K_2NaAlF_6$ | $MgAl_2O_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 2452-B | 2 | 4 | 10 | 6 | 5 | 6.9 | 5 | 10 | 36.4 | 3 | 13 |
| 2454-B | 2 | 6 | 14 | 10 | 8 | 10.4 | 3 | 14 | 49.2 | 3 | 7 |
| 2468-B | 2 | 4 | 10 | 6 | 5 | 6.9 | 7 | 8 | 35.7 | 3 | 11 |
| 2478-B | 2 | 4 | 14 | 10 | 7 | 8.3 | 6 | 12 | 48.6 | 2 | 10 |
| 2433-C | 2 | 5 | 11 | 6 | 5 | 8.1 | 9 | 9 | 39.9 | 2 | 5 |
| 2435-C | 2 | 6 | 10 | 6 | 5 | 8.9 | 11 | 9 | 41.0 | 2 | 7 |
| 2532-D | 2 | 0 | 12 | 7 | 6 | 3.5 | 3 | 10 | 37.8 | 2 | 5 |
| 2536-D | 2 | 6 | 8 | 5 | 4 | 8.4 | 4 | 7 | 27.9 | 3 | 10 |
| 2538-D | 2 | 2 | 10 | 7 | 5 | 5.1 | 2 | 10 | 34.2 | 3 | 8 |
| 2540-D | 2 | 2 | 12 | 8 | 7 | 5.7 | 3 | 11 | 40.9 | 4 | 10 |
| 2437-E | 3 | 5 | 9 | 6 | 5 | 7.8 | 9 | 7 | 35.5 | 3 | 9 |
| 2439-E | 3 | 4 | 10 | 6 | 5 | 6.9 | 5 | 8 | 33.7 | 3 | 10 |
| 2502-F | 5 | 5 | 9 | 6 | 5 | 7.8 | 10 | 8 | 37.9 | 5 | 9 |
| 2504-F | 4 | 4 | 13 | 8 | 6 | 7.8 | 6 | 11 | 43.8 | 4 | 10 |
| 2506-F | 4 | 1 | 15 | 8 | 6 | 5.1 | 2 | 13 | 44.2 | 4 | 6 |
| 2508-F | 2 | 3 | 17 | 11 | 8 | 8.0 | 4 | 15 | 54.9 | 2 | 7 |
| 2510-F | 3 | 5 | 11 | 7 | 6 | 8.3 | 5 | 9 | 37.6 | 3 | 12 |
| 2046-H | 2 | 10 | 10 | 6 | 5 | 12.9 | 10 | 9 | 40.0 | 3 | 9 |
| 2544-H | 1 | 7 | 11 | 7 | 5 | 10.2 | 8 | 9 | 39.7 | 2 | 7 |
| 2555-H | 2 | 10 | 6 | 5 | 5 | 12.2 | 14 | 6 | 35.8 | 3 | 9 |
| 2559-H | 2 | 9 | 9 | 6 | 5 | 11.8 | 9 | 7 | 35.5 | 3 | 10 |
| 2568-H | 2 | 6 | 9 | 6 | 5 | 8.8 | 12 | 8 | 39.9 | 3 | 8 |
| 2512-J | 4 | 3 | 12 | 9 | 7 | 6.9 | 7 | 12 | 47.1 | 3 | 6 |
| 2515-J | 4 | 4 | 10 | 6 | 5 | 6.9 | 9 | 9 | 39.0 | 4 | 8 |
| 2517-J | 1 | 4 | 16 | 12 | 9 | 9.1 | 5 | 15 | 56.8 | 2 | 5 |
| 2519-J | 1 | 4 | 12 | 8 | 6 | 7.6 | 6 | 11 | 43.0 | 2 | 5 |
| 2482-L | 3 | 5 | 13 | 8 | 6 | 8.8 | 3 | 11 | 40.8 | 4 | 18 |
| 2484-L | 4 | 6 | 7 | 4 | 3 | 8.0 | 6 | 5 | 24.7 | 6 | 23 |
| 2486-L | 2 | 9 | 7 | 5 | 4 | 11.2 | 8 | 7 | 31.1 | 3 | 10 |
| 2488-L | 1 | 6 | 14 | 10 | 7 | 10.3 | 3 | 13 | 47.0 | 2 | 9 |
| 2490-L | 3 | 5 | 9 | 6 | 5 | 7.8 | 8 | 8 | 35.9 | 4 | 8 |
| 2601-M | 6 | 2 | 9 | 6 | 5 | 4.8 | 1 | 10 | 31.5 | 4 | 5 |
| 2505-M | 3 | 4 | 14 | 10 | 9 | 8.5 | 6 | 14 | 53.1 | 3 | 5 |
| 2609-M | 18 | 2 | 11 | 8 | 6 | 5.5 | ND | 10 | 34.8 | 6 | 13 |
| 2613-M | 14 | 3 | 8 | 5 | 4 | 5.4 | 2 | 7 | 25.9 | 6 | 8 |
| 2492-N | 1 | 3 | 13 | 9 | 7 | 7.0 | 23 | 12 | 63.9 | 1 | 2 |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 2494-N | 1 | 3 | 9 | 7 | 6 | 6.0 | 33 | 9 | 64.0 | 1 | 3 |
| 2496-N | 1 | 3 | 16 | 13 | 10 | 8.4 | 12 | 17 | 68.2 | 1 | 3 |
| 2498-N | 2 | 6 | 9 | 6 | 4 | 8.7 | 12 | 8 | 39.0 | 3 | 12 |

**Table B-38: Non-Aluminum mineral phase (%) in salt cake**

| Sample ID | KCl | NaCl | $CaF_2$ | Total salt | MgO | $CaSO_4$ | $CaCO_3$ | $SiO_2$ |
|---|---|---|---|---|---|---|---|---|
| 2452-B | 9 | 21 | 6 | 36 | 2 | 6 | ND | 1 |
| 2454-B | 8 | 12 | 7 | 27 | 2 | ND | 1 | 3 |
| 2468-B | 8 | 20 | 5 | 33 | 2 | 6 | ND | 4 |
| 2478-B | 6 | 17 | 3 | 26 | 1 | 5 | ND | ND |
| 2433-C | 8 | 23 | 7 | 38 | 1 | 5 | 1 | 2 |
| 2435-C | 7 | 21 | 5 | 33 | 1 | 5 | ND | 1 |
| 2532-D | 18 | 18 | 11 | 47 | 1 | 5 | ND | ND |
| 2536-D | 17 | 15 | 11 | 43 | 1 | 5 | 1 | ND |
| 2538-D | 19 | 19 | 12 | 50 | 1 | ND | ND | 1 |
| 2540-D | 11 | 16 | 8 | 35 | 1 | 5 | ND | 1 |
| 2437-E | 10 | 18 | 8 | 36 | 2 | 6 | 1 | 1 |
| 2439-E | 11 | 20 | 7 | 38 | 1 | 5 | 1 | 1 |
| 2502-F | 5 | 20 | 6 | 31 | 1 | 6 | ND | 1 |
| 2504-F | 5 | 20 | ND | 25 | 3 | 5 | 1 | ND |
| 2506-F | 9 | 30 | ND | 39 | 2 | ND | ND | ND |
| 2508-F | 5 | 19 | 2 | 26 | 1 | 4 | ND | ND |
| 2510-F | 8 | 22 | ND | 30 | 4 | 6 | ND | ND |
| 2046-H | 9 | 16 | 5 | 30 | ND | 5 | ND | 1 |
| 2544-H | 10 | 18 | 6 | 34 | ND | 5 | 1 | 1 |
| 2555-H | 10 | 12 | 9 | 31 | ND | 8 | 2 | ND |
| 2559-H | 11 | 16 | 8 | 35 | ND | 6 | ND | ND |
| 2568-H | 10 | 15 | 6 | 31 | 1 | 5 | ND | 4 |
| 2512-J | 10 | 15 | 5 | 30 | 1 | 5 | ND | 3 |
| 2515-J | 12 | 21 | ND | 33 | 1 | 5 | 1 | 2 |
| 2517-J | 7 | 13 | 3 | 23 | 1 | 4 | ND | 2 |
| 2519-J | 11 | 15 | 12 | 38 | 1 | 4 | 1 | 2 |
| 2482-L | 4 | 20 | 2 | 26 | 4 | ND | ND | ND |
| 2484-L | 5 | 16 | 4 | 25 | 4 | 6 | ND | ND |
| 2486-L | 10 | 19 | 7 | 36 | 1 | 6 | 1 | ND |
| 2488-L | 5 | 19 | 3 | 27 | 2 | 5 | ND | ND |
| 2490-L | 7 | 18 | 5 | 30 | 2 | 7 | 1 | 5 |
| 2601-M | 14 | 20 | ND | 34 | 2 | ND | ND | 16 |
| 2505-M | 5 | 17 | 3 | 25 | 1 | 5 | ND | 1 |
| 2609-M | 11 | 11 | ND | 22 | 2 | ND | ND | 1 |
| 2613-M | 13 | 11 | 8 | 32 | 2 | 6 | ND | 1 |
| 2492-N | 4 | 17 | 2 | 23 | ND | 6 | ND | 1 |
| 2494-N | 5 | 12 | 3 | 20 | ND | 8 | ND | 1 |
| 2496-N | 3 | 14 | 1 | 18 | 1 | 5 | ND | 1 |
| 2498-N | 5 | 20 | 4 | 29 | 1 | 7 | ND | ND |

ND: Not detected

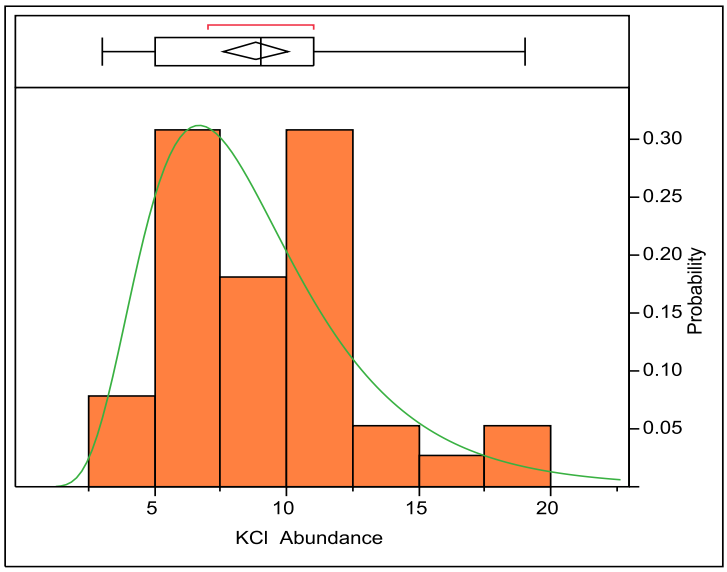


**Figure B-28: Distribution of the KCl abundance in salt cake samples**

**Table B-39: KCl abundance of salt cake by facility (%)**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 7.8±1.3 | 9.8 | 6-9 |
| C | 7.5±0.7 | 14 | 7-8 |
| D | 16±3.6 | 22 | 11-19 |
| E | 11±0.8 | 16.9 | 10-11 |
| F | 6.4±1.9 | 8.8 | 5-9 |
| H | 10±0.7 | 11 | 9-11 |
| J | 10±2.2 | 13 | 7-12 |
| L | 6.2±2.4 | 9.2 | 4-10 |
| M | 11±4.0 | 17 | 5-14 |
| N | 4.3±1.0 | 5.8 | 3-5 |
| All Facilities | 8.8±3.8 | 10 | 3-19 |

**Table B-40: NaCl abundance of salt cake by facility (%)**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 18±4.0 | 24 | 12-21 |
| C | 22±1.4 | 35 | 21-23 |
| D | 17±1.8 | 20 | 15-19 |
| E | 19±1.4 | 32 | 18-20 |
| F | 22±4.5 | 28 | 19-30 |
| H | 15±2.2 | 18 | 12-18 |
| J | 16±3.5 | 22 | 13-21 |
| L | 18±1.5 | 20 | 16-20 |
| M | 15±4.5 | 22 | 11-20 |
| N | 16±3.5 | 21 | 12-20 |
| All Facilities | 18±3.8 | 19 | 11-30 |

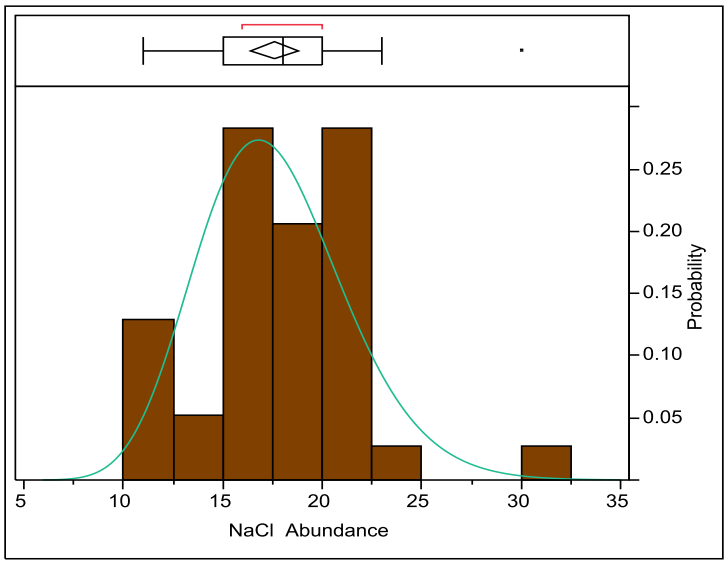


**Figure B-29: Distribution of the NaCl abundance in salt cake samples**

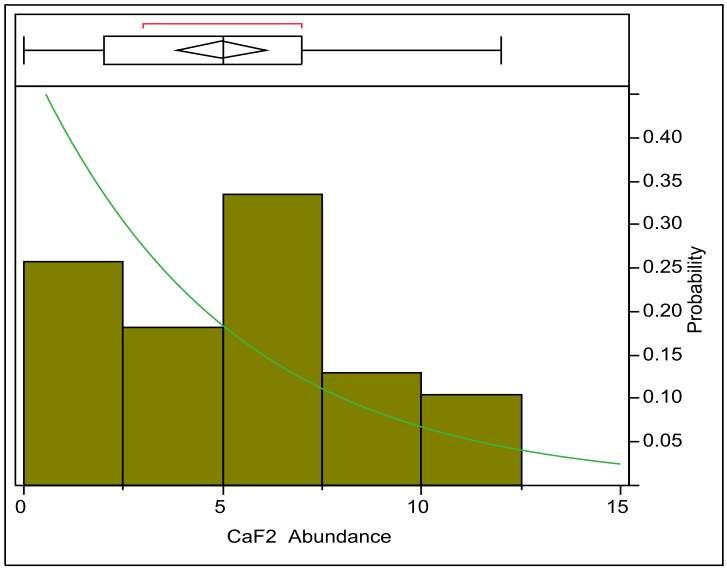


**Figure B-30: Distribution of the fluorite abundance in salt cake samples**

**Table B-41: Fluorite abundance of salt cake by facility (%)**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 5.3±1.7 | 8.0 | 3-7 |
| C | 6±1.4 | 19 | 5-7 |
| D | 11±1.7 | 13 | 8-12 |
| E | 7.5±0.7 | 14 | 7-8 |
| F | 1.6±2.6 | 4.8 | ND-6 |
| H | 6.8±1.6 | 8.8 | 5-9 |
| J | 5±5.1 | 13 | ND-12 |
| L | 4.2±1.9 | 6.6 | 2-7 |
| M | 2.8±3.8 | 8.8 | ND-8 |
| N | 2.5±1.3 | 4.6 | 1-4 |

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| All Facilities | 5.0±3.5 | 6.1 | ND-12 |

* ND: Undetectable

**Table B-42: Total salt content (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 31±4.8 | 38 | 26-36 |
| C | 31±3.5 | 67 | 33-38 |
| D | 44±6.5 | 54 | 35-50 |
| E | 37±1.4 | 50 | 36-38 |
| F | 30±5.5 | 37 | 25-39 |
| H | 32±2.2 | 35 | 30-35 |
| J | 31±6.3 | 41 | 23-38 |
| L | 29±4.4 | 34 | 25-36 |
| M | 28±5.7 | 37 | 22-34 |
| N | 23±4.8 | 30 | 18-29 |
| All Facilities | 31±6.9 | 34 | 18-50 |

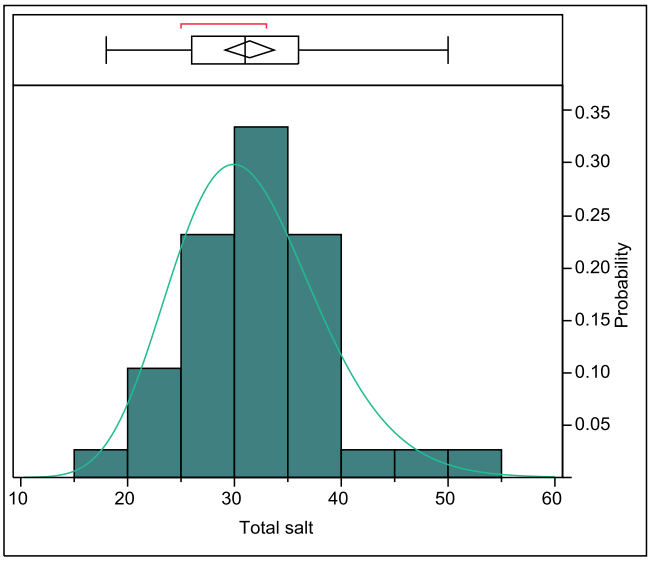


**Figure B-31: Distribution of the total salt flux in salt cake samples**

**Table B-43: Metallic Al abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 2±0 | 2 | 2-2 |
| C | 2±0 | 2 | 2-2 |
| D | 2±0 | 2 | 2-2 |
| E | 3±0 | 3 | 3-3 |
| F | 3.6±1.1 | 5.0 | 2-5 |
| H | 1.8±0.4 | 2.4 | 1-2 |
| J | 2.5±1.7 | 5.3 | 1-4 |
| L | 2.6±1.1 | 4.0 | 1-4 |
| M | 10±6.9 | 21 | 3-18 |
| N | 1.3±0.5 | 2.0 | 1-2 |

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| All Facilities | 3.1±3.3 | 4.2 | 1-18 |

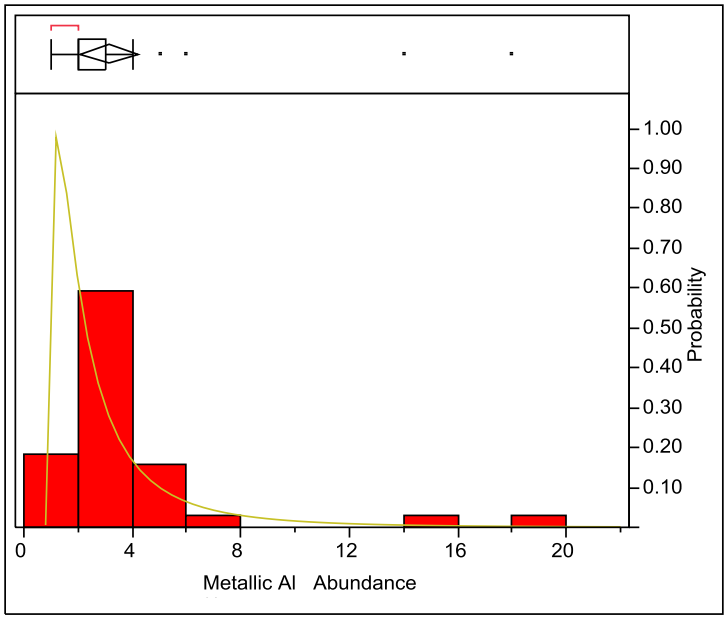


**Figure B-32: Distribution of the metallic aluminum in salt cake samples**

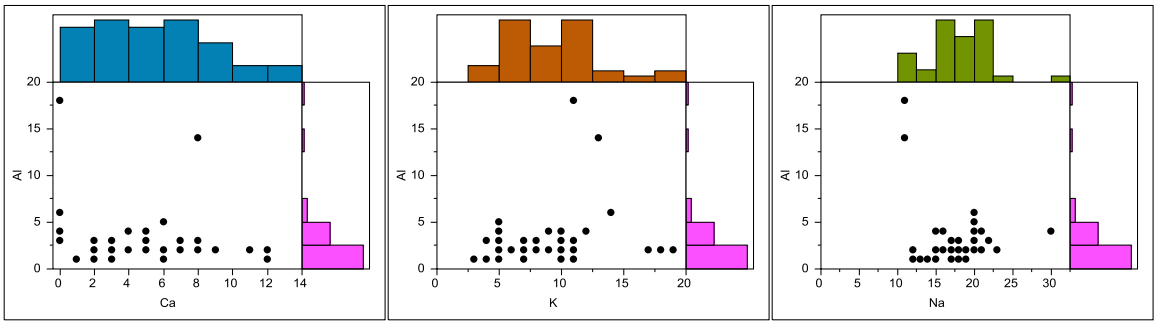


**Figure B-33: Relations between the abundance of metallic Al and salt in salt cake**

**Table B-44: Mineral AlN abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 4.5±1 | 6.1 | 4-6 |
| C | 5.5±0.7 | 12 | 5-6 |
| D | 2.5±2.5 | 6.5 | ND-6 |
| E | 4.5±0.7 | 11 | 4-5 |
| F | 3.6±1.7 | 5.7 | 1-5 |
| H | 8.4±1.8 | 11 | 6-10 |
| J | 3.8±0.5 | 4.5 | 3-4 |
| L | 6.2±1.6 | 8.2 | 5-9 |
| M | 2.8±1.0 | 4.3 | 2-4 |
| N | 3.8±1.5 | 6.1 | 3-6 |
| All Facilities | 4.6±2.3 | 5.4 | ND-10 |

* ND: Undetectable

**Table B-45: $Al_{2.85}O_{3.45}N_{0.55}$ abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 12±2.3 | 16 | 10-14 |
| C | 11±0.7 | 17 | 10-11 |
| D | 11±1.9 | 14 | 8-12 |
| E | 9.5±0.7 | 16 | 9-10 |
| F | 13±3.2 | 17 | 9-17 |
| H | 9±1.9 | 11 | 6-11 |
| J | 13±2.5 | 17 | 10-16 |
| L | 10±3.3 | 14 | 7-17 |
| M | 11±2.6 | 15 | 8-14 |
| N | 12±3.4 | 17 | 9-16 |
| All Facilities | 11±2.7 | 12 | 6-17 |

**Table B-46: $Al_{2.78}O_{3.65}N_{0.35}$ abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 6.3±1.5 | 8.6 | 5-8 |
| C | 5±0 | 5 | 5-5 |
| D | 5.5±1.3 | 7.6 | 4-7 |
| E | 5±0 | 5 | 5-5 |
| F | 6.2±1.1 | 7.6 | 5-8 |
| H | 5±0 | 5 | 5-5 |
| J | 6.8±1.7 | 9.5 | 5-9 |
| L | 5±1.6 | 7.0 | 3-7 |
| M | 6±2.2 | 9.4 | 4-9 |
| N | 6.8±2.5 | 10.7 | 4-10 |
| All Facilities | 5.8±1.5 | 6.3 | 3-10 |

**Table B-47: $Al_{2.81}O_{3.56}N_{0.44}$ abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 8±2.3 | 12 | 6-10 |
| C | 6±0 | 6 | 6-6 |
| D | 6.8±1.3 | 8.8 | 5-8 |
| E | 6±0 | 6 | 6-6 |
| F | 8±1.9 | 10 | 6-11 |
| H | 6±0.7 | 6.9 | 5-7 |
| J | 8.8±2.5 | 13 | 6-12 |
| L | 6.6±2.4 | 9.6 | 4-10 |
| M | 7.3±2.2 | 11 | 5-10 |
| N | 8.8±3.1 | 14 | 6-13 |
| All Facilities | 7.3±2.1 | 8.0 | 4-13 |

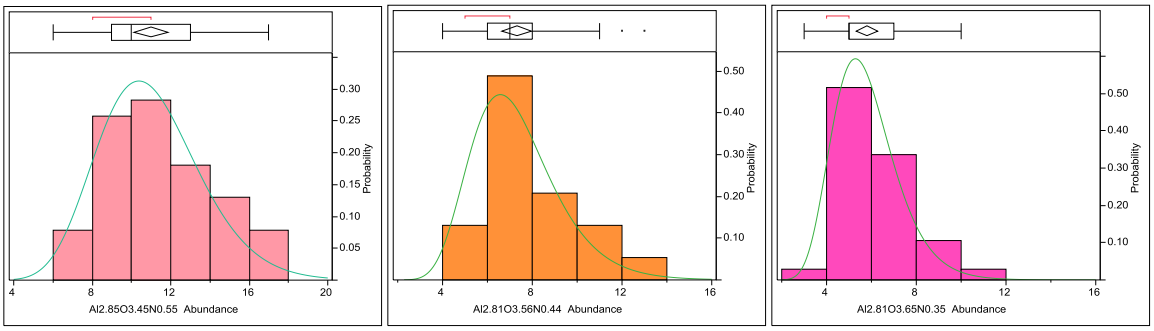


**Figure B-34: Overall distribution of the three aluminum nitride oxides in salt cake**

**Table B-48: Total AlN abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 8.1±1.7 | 10.8 | 6.9-10 |
| C | 8.5±0.6 | 13.6 | 8.1-8.9 |
| D | 5.7±2.0 | 8.9 | 3.5-8.4 |
| E | 7.4±0.6 | 13 | 6.9-7.8 |
| F | 7.4±1.3 | 9.0 | 5.1-8.3 |
| H | 11±1.7 | 13 | 8.8-13 |
| J | 7.6±1.0 | 9.3 | 6.9-9.1 |
| L | 9.2±1.5 | 11 | 7.8-11 |
| M | 6.1±1.7 | 8.7 | 4.8-8.5 |
| N | 7.5±1.3 | 9.5 | 6-8.7 |
| All Facilities | 8.0±2.1 | 8.6 | 3.5-13 |

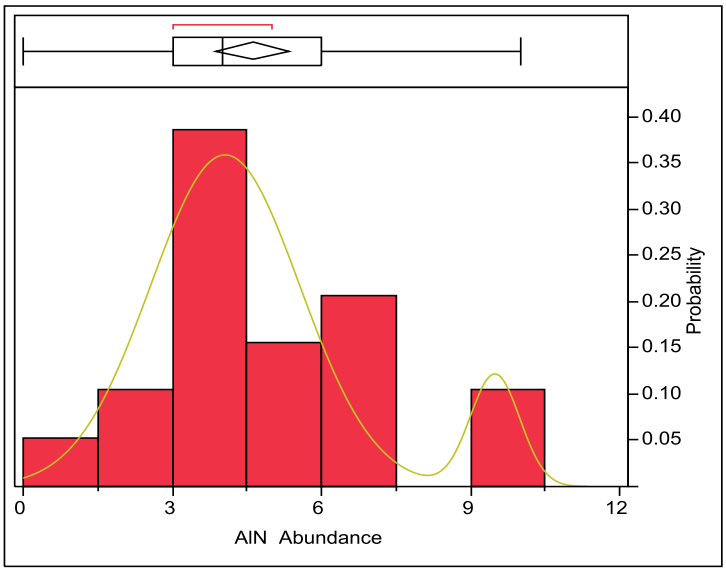


**Figure B-35: Distribution of the aluminum nitride in salt cake samples**

**Table B-49: Alumina abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 5.3-1.7 | 8.0 | 3-7 |
| C | 10-1.4 | 23 | 9-11 |
| D | 3-0.8 | 4.3 | 2-4 |
| E | 7-2.8 | 32 | 5-9 |
| F | 5.4-3.0 | 9.1 | 2-10 |
| H | 11-2.4 | 14 | 8-14 |
| J | 6.8-1.7 | 9.5 | 5-9 |
| L | 5.6-2.5 | 8.7 | 3-8 |
| M | 2.3-2.6 | 6.4 | ND-6 |
| N | 20-10 | 36 | 12-33 |
| All Facilities | 7.5-6.0 | 9.4 | ND-33 |

* ND: Undetectable

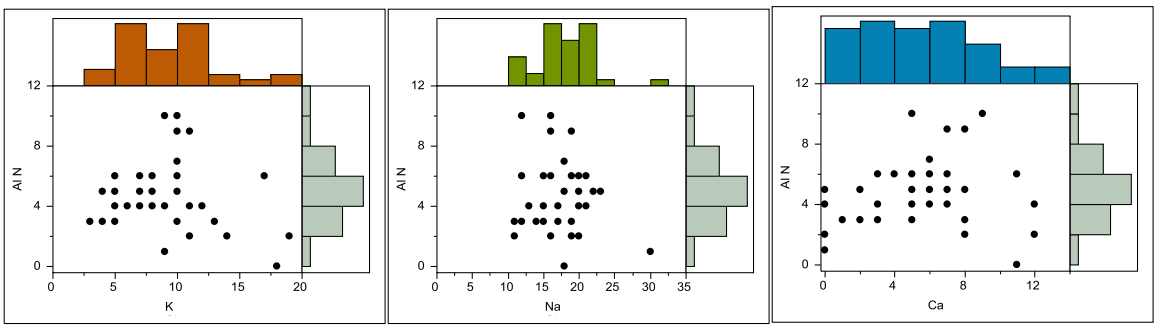


**Figure B-36: Relations between the abundance of AlN and salt in salt cake**

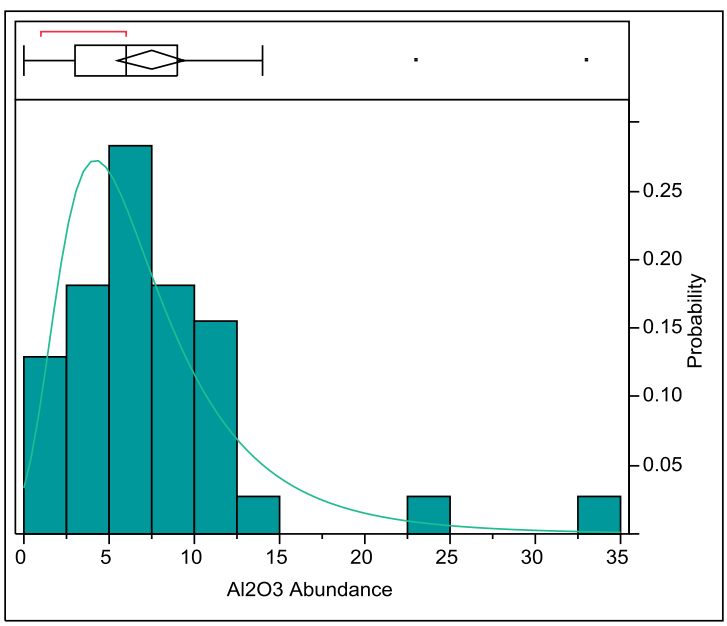


**Figure B-37: Distribution of the alumina in salt cake samples**

**Table B-50: Alumina abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 5.3-1.7 | 8.0 | 3-7 |
| C | 10-1.4 | 23 | 9-11 |
| D | 3-0.8 | 4.3 | 2-4 |
| E | 7-2.8 | 32 | 5-9 |
| F | 5.4-3.0 | 9.1 | 2-10 |
| H | 11-2.4 | 14 | 8-14 |
| J | 6.8-1.7 | 9.5 | 5-9 |
| L | 5.6-2.5 | 8.7 | 3-8 |
| M | 2.3-2.6 | 6.4 | ND-6 |
| N | 20-10 | 36 | 12-33 |
| All Facilities | 7.5-6.0 | 9.4 | ND-33 |

* ND: Undetectable

**Table B-51: $Al_{2.667}O_4$ abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 11±15 | 15 | 8-14 |
| C | 9±9 | 9 | 9-9 |
| D | 9.5±12 | 12 | 7-11 |
| E | 7.5±14 | 14 | 7-8 |
| F | 11±15 | 15 | 8-15 |
| H | 7.8±9.4 | 9.4 | 6-9 |
| J | 12±16 | 16 | 9-15 |
| L | 8.8±13 | 13 | 5-13 |
| M | 10±15 | 15 | 7-14 |
| N | 11±18 | 18 | 8-17 |
| All Facilities | 9.9±11 | 11 | 5-17 |

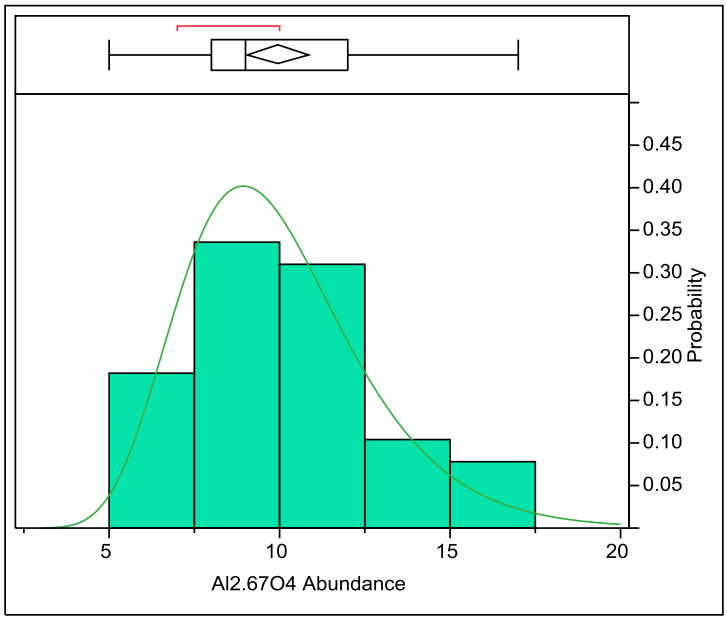


**Figure B-38: Distribution of $Al_{2.67}O_4$ in salt cake samples**

**Table B-52: Total abundance of Aluminum oxides (%) in salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 43±7.4 | 54 | 36-49 |
| C | 41±0.8 | 47 | 40-41 |
| D | 35±5.6 | 44 | 28-41 |
| E | 35±1.3 | 46 | 34-36 |
| F | 44±7.0 | 52 | 38-55 |
| H | 38±2.3 | 41 | 36-40 |
| J | 47±7.6 | 59 | 39-57 |
| L | 36±8.6 | 47 | 25-47 |
| M | 36±12 | 55 | 26-53 |
| N | 59±13 | 80 | 39-68 |
| All Facilities | 41±10 | 45 | 25-68 |

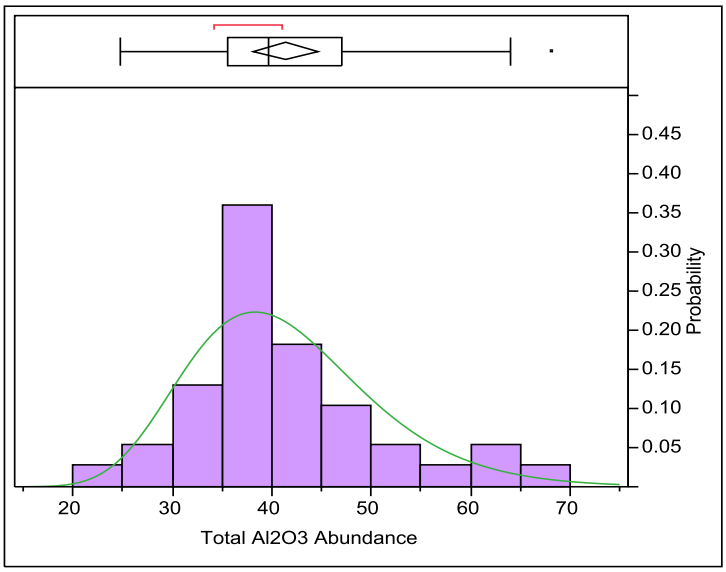


**Figure B-39: Distribution of total aluminum oxides in salt cake samples**

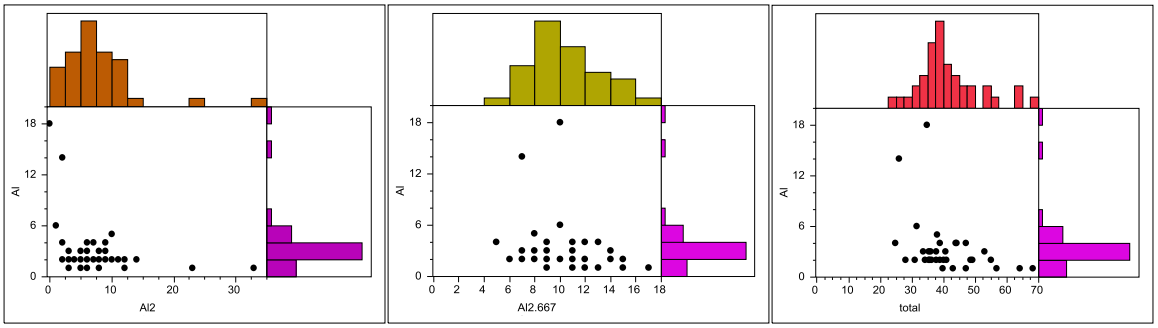


**Figure B-40: Relations between alumina oxides and metallic Al in salt cake**

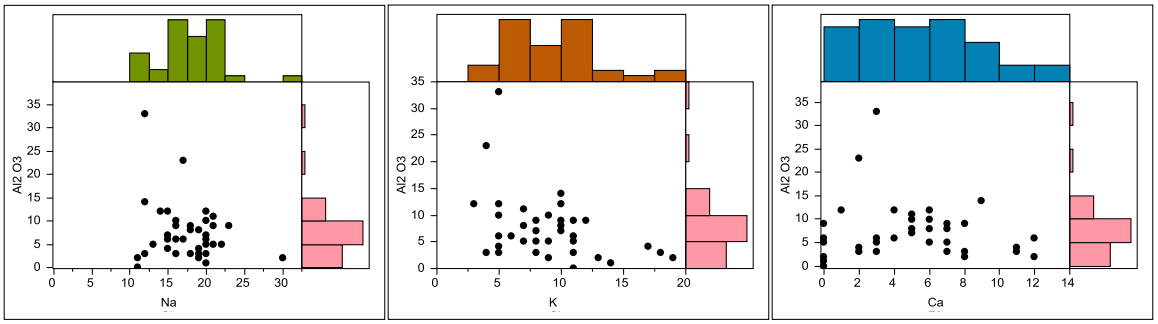


**Figure B-41: Relations between salt and alumina in salt cake**

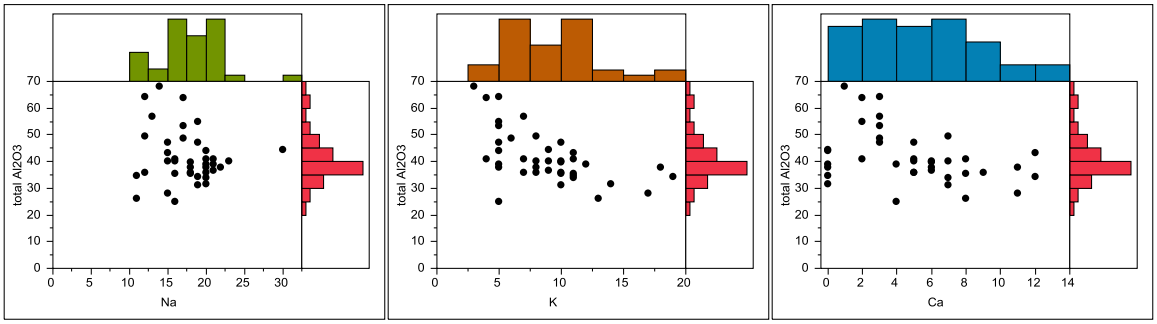


**Figure B-42: Relations between salt and total aluminum oxides in salt cake**

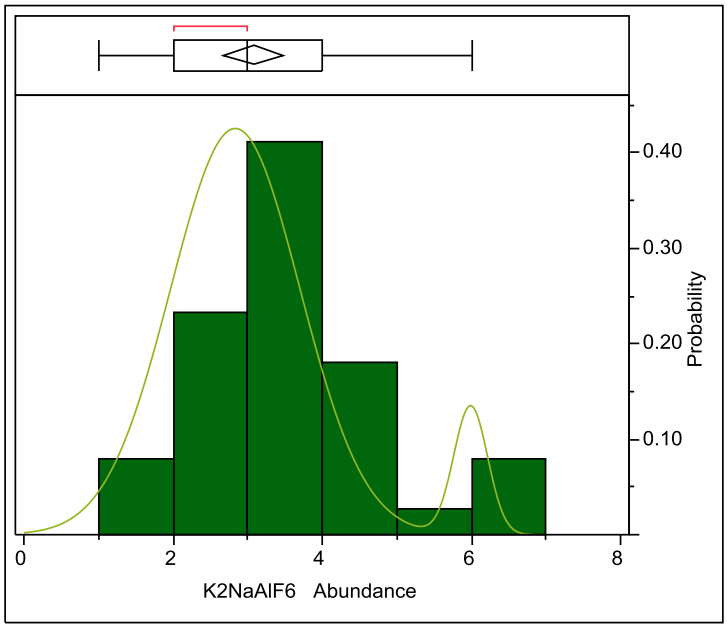


**Figure B-43: Distribution of the elpasolite abundance in salt cake samples**

**Table B-53: Elpasolite abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 2.8±0.5 | 3.5 | 2-3 |
| C | 2±0 | 2 | 2-2 |
| D | 3±0.8 | 4.3 | 2-4 |
| E | 3±0 | 3 | 3-3 |
| F | 3.6±1.1 | 5.0 | 2-5 |
| H | 2.8±0.4 | 3.4 | 2-3 |
| J | 2.8±1.0 | 4.3 | 2-4 |
| L | 3.8±1.5 | 5.6 | 2-6 |

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| M | 4.8±1.5 | 7.1 | 3-6 |
| N | 1.5±1 | 3.1 | 1-3 |
| All Facilities | 3.1±1.2 | 3.5 | 1-6 |

**Table B-54: Spinel abundance (%) of salt cake by facility**

| Facility | Mean | $UCL_{95}$ | Range |
|---|---|---|---|
| B | 10±2.5 | 14.2 | 7-13 |
| C | 6±1.4 | 18.7 | 5-7 |
| D | 8.3±2.4 | 12.0 | 5-10 |
| E | 9.5±0.7 | 15.9 | 9-10 |
| F | 8.8±2.4 | 11.8 | 6-12 |
| H | 8.6±1.1 | 10.0 | 7-10 |
| J | 6±1.4 | 8.3 | 5-8 |
| L | 14±6.6 | 21.8 | 8-23 |
| M | 7.8±3.8 | 13.8 | 5-13 |
| N | 5±4.7 | 12.4 | 2-12 |
| All Facilities | 8.6±3.9 | 9.9 | 2-23 |

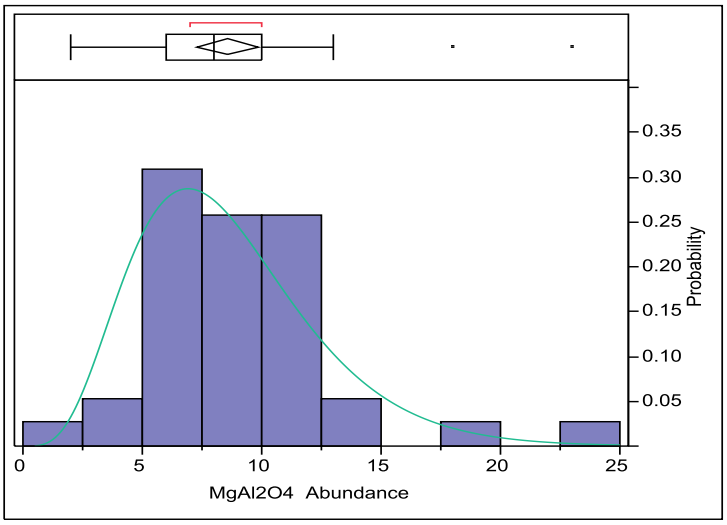


**Figure B-44: Distribution of the spinel abundance in salt cake samples**

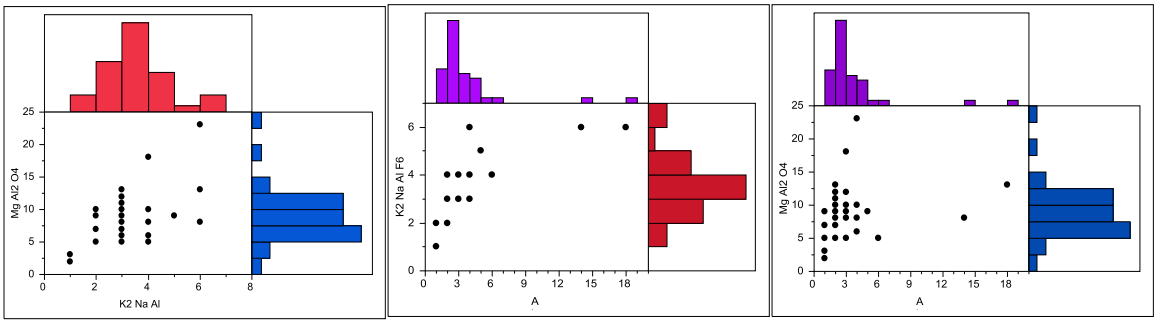


**Figure B-45: Relations between metallic Al, elpasolite and spinel in salt cake**

*Secondary Aluminum Processing Waste: Salt Cake Characterization and Reactivity*

# 9. APPENDIX C: XRD Data

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9.4: 2613-M-SC....336
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10.3: 2496-N-SC.....356
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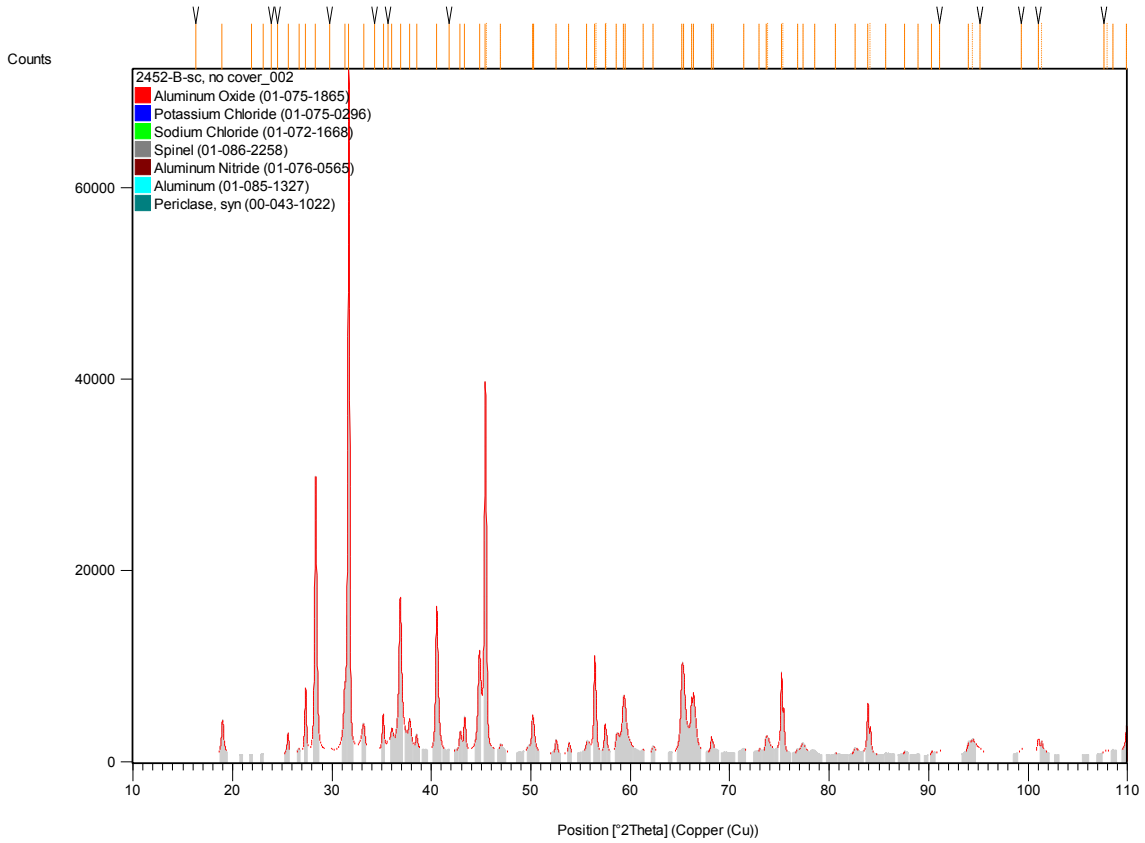
## FACILITY B

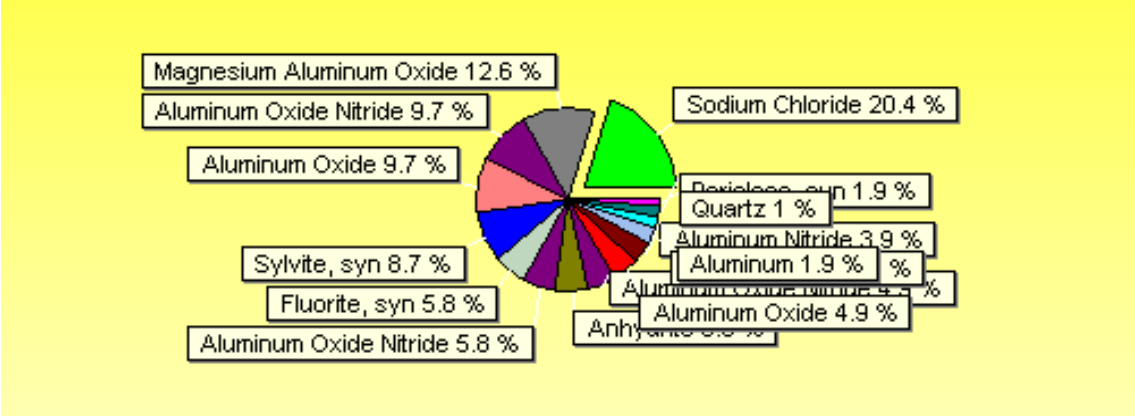
### 1.1: 2452-B-SC

#### 1.1.1 Measurement Conditions

| | |
|---|---|
| Dataset Name | 2452-B-SC, no cover_002 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2452-B-sc, no cover_002.sd |
| Sample Identification | 2452-B-SC, no cover |
| Comment | Exported by X'Pert SW Generated by todd in project Ft. Devens. |
| Measurement Date / Time | 9/13/2010 9:26:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

#### 1.1.2: Main Graphics, Analyze View of 2452-B




**1.1.3: Pattern List of 2452-B**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 54 | Aluminum Oxide | Al2 O3 | 5 | 20 | 0 |
| 00-004-0587 | 49 | Sylvite, syn | K Cl | 9 | 10 | 0 |
| 01-077-2064 | 51 | Sodium Chloride | Na Cl | 21 | 9 | 0 |
| 01-074-1132 | 55 | Magnesium Aluminum Oxide | Mg Al2 O4 | 13 | 14 | 0 |
| 01-075-1620 | 32 | Aluminum Nitride | Al N | 4 | 8 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 10 | 12 | 0 |
| 01-080-2172 | 19 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 19 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 12 | 0 |
| 01-085-1327 | 22 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 34 | Periclase, syn | Mg O | 2 | 7 | 0 |
| 00-004-0864 | 17 | Fluorite, syn | Ca F2 | 6 | 6 | 0 |
| 00-022-1235 | 25 | Elpasolite, syn | K2 Na Al F6 | 3 | 16 | 0 |
| 01-086-2270 | 21 | Anhydrite | Ca ( S O4 ) | 6 | 39 | 0 |
| 01-080-1385 | 19 | Aluminum Oxide | Al2.667 O4 | 10 | 12 | 0 |
| 01-085-0930 | 14 | Quartz | Si O2 | 1 | 18 | 0 |

**1.1.4: Peak List of 2452-B**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 16.3120 | 97.75 | 0.2362 | 5.43416 | 0.14 | |
| 18.9666 | 3551.60 | 0.0492 | 4.67914 | 4.98 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 21.9210 | 135.51 | 0.2362 | 4.05473 | 0.19 | 00-022-1235 |
| 23.0550 | 182.34 | 0.3149 | 3.85781 | 0.26 | 01-086-2270 |
| 23.8802 | 163.97 | 0.2362 | 3.72633 | 0.23 | |
| 24.4954 | 315.41 | 0.1574 | 3.63412 | 0.44 | |
| 25.5660 | 2302.18 | 0.1181 | 3.48431 | 3.23 | 01-075-1862; 01-086-2270 |
| 26.6574 | 727.14 | 0.1378 | 3.34409 | 1.02 | 01-085-0930 |
| 27.3460 | 6982.14 | 0.0984 | 3.26143 | 9.80 | 01-077-2064 |
| 28.3548 | 28990.78 | 0.1673 | 3.14765 | 40.69 | 00-004-0587; 00-004-0864 |
| 29.7838 | 676.66 | 0.2362 | 2.99980 | 0.95 | |
| 31.2722 | 7215.31 | 0.1181 | 2.86034 | 10.13 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 31.6806 | 71247.75 | 0.1673 | 2.82439 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.2189 | 3129.19 | 0.2362 | 2.69703 | 4.39 | 01-075-1620 |
| 34.3249 | 661.14 | 0.3149 | 2.61262 | 0.93 | |
| 35.1383 | 4146.22 | 0.0984 | 2.55398 | 5.82 | 01-075-1862 |
| 35.6056 | 1514.56 | 0.1181 | 2.52153 | 2.13 | |
| 36.0140 | 2690.27 | 0.1771 | 2.49387 | 3.78 | 01-075-1620 |
| 36.8589 | 16383.13 | 0.1574 | 2.43862 | 22.99 | 01-074-1132; 00-043-1022 |
| 37.7908 | 3649.17 | 0.1968 | 2.38060 | 5.12 | 01-075-1862; 01-075-1620 |
| 38.4973 | 1993.74 | 0.1771 | 2.33852 | 2.80 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.4885 | 14140.14 | 0.1279 | 2.22799 | 19.85 | 00-004-0587 |
| 41.8142 | 420.56 | 0.2362 | 2.16037 | 0.59 | |
| 42.9022 | 2295.15 | 0.0689 | 2.10807 | 3.22 | 00-043-1022 |
| 43.3116 | 3822.06 | 0.0492 | 2.08909 | 5.36 | 01-075-1862; 01-086-2270 |
| 44.8249 | 10652.60 | 0.1574 | 2.02202 | 14.95 | 01-074-1132; 01-085-1327 |
| 45.4042 | 38085.40 | 0.1680 | 1.99590 | 53.45 | 01-077-2064; 01-086-2270 |
| 45.5424 | 21621.73 | 0.0960 | 1.99511 | 30.35 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 46.9563 | 919.84 | 0.1440 | 1.93348 | 1.29 | 00-004-0864; 01-086-2270 |
| 50.1396 | 3520.41 | 0.0720 | 1.81794 | 4.94 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 50.2852 | 3415.84 | 0.0960 | 1.81301 | 4.79 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 52.5329 | 1476.12 | 0.1200 | 1.74061 | 2.07 | 01-075-1862 |
| 53.8203 | 1211.78 | 0.1440 | 1.70197 | 1.70 | 01-077-2064 |
| 55.6047 | 1188.15 | 0.2400 | 1.65150 | 1.67 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4188 | 10160.12 | 0.1440 | 1.62959 | 14.26 | 01-077-2064 |
| 56.6081 | 4642.62 | 0.0960 | 1.62863 | 6.52 | |
| 57.4488 | 3054.41 | 0.1080 | 1.60280 | 4.29 | 01-075-1862 |
| 57.6144 | 1930.18 | 0.1200 | 1.60255 | 2.71 | |
| 58.6033 | 1917.57 | 0.1920 | 1.57394 | 2.69 | 00-004-0587 |
| 59.3284 | 6090.31 | 0.0840 | 1.55642 | 8.55 | 01-074-1132; 01-075-1620 |
| 59.5020 | 4972.67 | 0.0960 | 1.55230 | 6.98 | 01-075-1862; 01-074-1132 |
| 61.2636 | 392.20 | 0.1920 | 1.51182 | 0.55 | 01-075-1862 |
| 62.2711 | 785.63 | 0.1440 | 1.48975 | 1.10 | 00-043-1022; 01-086-2270 |
| 65.2078 | 9262.63 | 0.1440 | 1.42958 | 13.00 | 01-074-1132; 01-085-1327 |
| 65.3831 | 8345.74 | 0.1440 | 1.42617 | 11.71 | 01-074-1132; 01-086-2270 |
| 66.1455 | 5669.36 | 0.1200 | 1.41157 | 7.96 | 01-077-2064; 01-075-1620 |
| 66.3426 | 6344.42 | 0.1080 | 1.40785 | 8.90 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-1385 |
| 68.1612 | 1637.29 | 0.0960 | 1.37465 | 2.30 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.3279 | 1028.13 | 0.1200 | 1.37170 | 1.44 | 00-022-1235; 01-085-0930 |
| 71.3612 | 533.31 | 0.3360 | 1.32067 | 0.75 | 01-075-1620; 01-080-2173; 01-086-2270 |

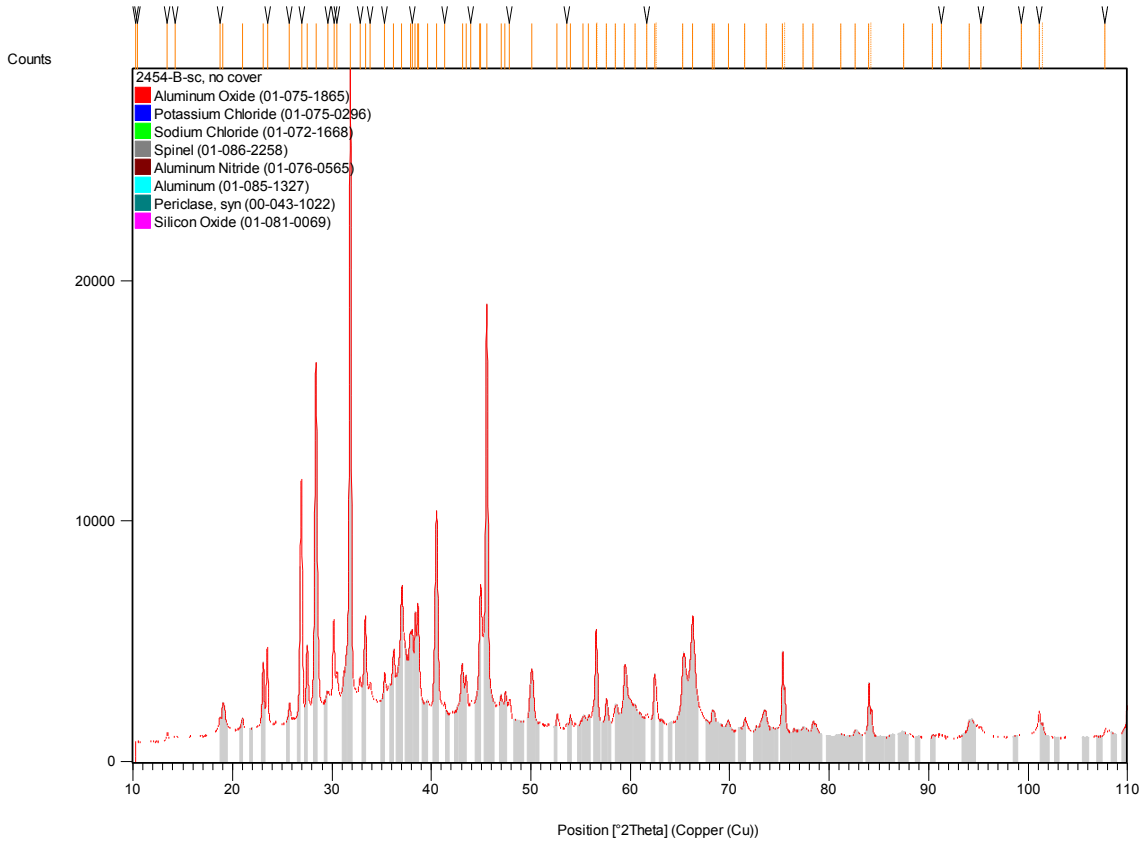
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 72.9777 | 579.10 | 0.1200 | 1.29535 | 0.81 | 01-077-2064; 01-086-2270 |
| 73.6559 | 1876.08 | 0.1920 | 1.28509 | 2.63 | 00-004-0587; 00-022-1235 |
| 73.8772 | 1603.46 | 0.1440 | 1.28497 | 2.25 | |
| 75.2014 | 8497.75 | 0.1560 | 1.26247 | 11.93 | 01-077-2064 |
| 75.4178 | 4816.08 | 0.1080 | 1.26251 | 6.76 | |
| 76.8160 | 657.98 | 0.1200 | 1.23990 | 0.92 | 01-075-1862 |
| 77.3334 | 1163.95 | 0.1680 | 1.23289 | 1.63 | 01-074-1132 |
| 78.5381 | 431.31 | 0.6720 | 1.21697 | 0.61 | 01-074-1132; 00-043-1022; 01-086-2270 |
| 80.6147 | 188.64 | 0.1440 | 1.19078 | 0.26 | 01-075-1862 |
| 82.5810 | 781.08 | 0.1920 | 1.16734 | 1.10 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.8916 | 5373.96 | 0.1440 | 1.15241 | 7.54 | 01-077-2064; 01-085-0930 |
| 84.1223 | 2972.52 | 0.1560 | 1.15269 | 4.17 | |
| 85.7082 | 222.27 | 0.2400 | 1.13257 | 0.31 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 87.5936 | 432.41 | 0.2400 | 1.11298 | 0.61 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 88.9296 | 152.29 | 0.1920 | 1.09969 | 0.21 | 01-075-1862 |
| 90.3108 | 399.76 | 0.1200 | 1.08642 | 0.56 | 00-022-1235 |
| 91.1115 | 561.85 | 0.1920 | 1.07895 | 0.79 | |
| 94.0246 | 1456.77 | 0.1200 | 1.05304 | 2.04 | 00-043-1022; 00-004-0864 |
| 94.4253 | 1733.85 | 0.1440 | 1.05223 | 2.43 | |
| 95.1920 | 797.04 | 0.1680 | 1.04319 | 1.12 | |
| 99.2739 | 646.35 | 0.2400 | 1.01095 | 0.91 | |
| 101.0309 | 1804.01 | 0.1680 | 0.99806 | 2.53 | |
| 101.3775 | 1421.00 | 0.0960 | 0.99806 | 1.99 | |
| 107.6507 | 599.29 | 0.1680 | 0.95426 | 0.84 | |
| 108.4939 | 506.49 | 0.2400 | 0.94918 | 0.71 | 00-004-0587 |
| 109.9051 | 2986.73 | 0.0600 | 0.94091 | 4.19 | 00-043-1022 |

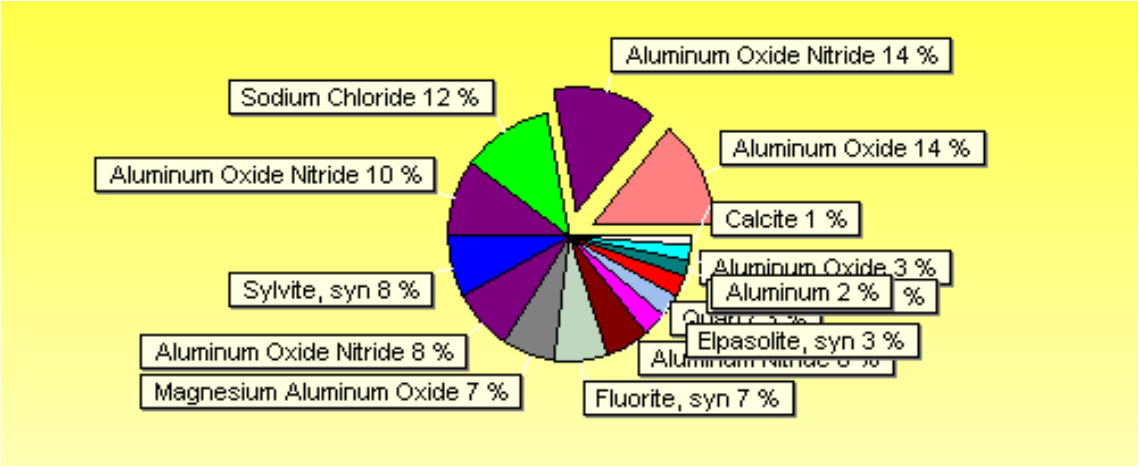
## 1.2: 2454-B-SC

### 1. 2.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2454-B-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2454-B-sc, no cover.sd |
| Sample Identification | 2454-B-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by todd in project Ft. Devens. | |
| Measurement Date / Time | 9/6/2010 1:22:00 PM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

**1.2.2: Main Graphics, Analyze View of 2454-B**



**1.2.3: Pattern List of 2454-B**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 31 | Aluminum Oxide | Al2 O3 | 3 | 12 | 0 |
| 00-004-0587 | 34 | Sylvite, syn | K Cl | 8 | 8 | 0 |
| 01-077-2064 | 50 | Sodium Chloride | Na Cl | 12 | 9 | 0 |
| 01-074-1132 | 42 | Magnesium Aluminum Oxide | Mg Al2 O4 | 7 | 12 | 0 |
| 01-075-1620 | 40 | Aluminum Nitride | Al N | 6 | 9 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 14 | 11 | 0 |
| 01-080-2172 | 15 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 10 | 8 | 0 |
| 01-080-2173 | 15 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 8 | 8 | 0 |
| 01-085-1327 | 36 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 31 | Periclase, syn | Mg O | 2 | 6 | 0 |
| 00-004-0864 | 18 | Fluorite, syn | Ca F2 | 7 | 5 | 0 |
| 00-022-1235 | 19 | Elpasolite, syn | K2 Na Al F6 | 3 | 12 | 0 |
| 01-080-1385 | 18 | Aluminum Oxide | Al2.667 O4 | 14 | 11 | 0 |
| 01-086-2339 | 11 | Calcite | Ca ( C O3 ) | 1 | 18 | 0 |
| 01-085-0930 | 19 | Quartz | Si O2 | 3 | 19 | 0 |

**1.2.4: Peak List of 2454-B**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.2784 | 390.75 | 0.0394 | 8.60653 | 1.40 | |
| 10.4044 | 185.69 | 0.2362 | 8.50257 | 0.67 | |
| 13.4431 | 339.24 | 0.0984 | 6.58669 | 1.22 | |
| 14.2128 | 176.64 | 0.1968 | 6.23170 | 0.63 | |
| 18.7440 | 787.33 | 0.1378 | 4.73422 | 2.83 | |
| 18.9789 | 1203.08 | 0.1574 | 4.67613 | 4.32 | 01-074-1132; 00-022-1235 |
| 21.0267 | 708.16 | 0.1082 | 4.22513 | 2.54 | 01-085-0930 |
| 23.0794 | 3062.55 | 0.1181 | 3.85378 | 11.00 | 01-086-2339 |
| 23.4920 | 3644.38 | 0.1476 | 3.78703 | 13.09 | |
| 25.7348 | 1359.51 | 0.1378 | 3.46184 | 4.88 | |
| 26.9236 | 10617.56 | 0.1574 | 3.31163 | 38.13 | |
| 27.5135 | 3776.52 | 0.1771 | 3.24195 | 13.56 | 01-077-2064 |
| 28.4217 | 14884.73 | 0.1870 | 3.14039 | 53.45 | 00-004-0587; 00-004-0864 |
| 29.5465 | 1847.40 | 0.1574 | 3.02335 | 6.63 | |
| 30.1848 | 4804.33 | 0.1673 | 2.96085 | 17.25 | |
| 30.5222 | 2648.80 | 0.1181 | 2.92889 | 9.51 | |
| 31.8431 | 27845.93 | 0.1870 | 2.81035 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 32.8052 | 2430.35 | 0.1574 | 2.73009 | 8.73 | |
| 33.3599 | 4986.85 | 0.0886 | 2.68595 | 17.91 | 01-075-1620 |
| 33.8444 | 2233.43 | 0.1574 | 2.64860 | 8.02 | |
| 35.2872 | 2610.30 | 0.0689 | 2.54355 | 9.37 | |
| 36.1919 | 3577.15 | 0.1574 | 2.48202 | 12.85 | 01-075-1620 |
| 37.0224 | 6185.25 | 0.1771 | 2.42822 | 22.21 | 00-043-1022 |
| 37.8560 | 4278.89 | 0.1378 | 2.37665 | 15.37 | 01-075-1862; 01-075-1620 |
| 38.0985 | 4302.12 | 0.1181 | 2.36208 | 15.45 | |
| 38.3765 | 5174.65 | 0.0590 | 2.34561 | 18.58 | 01-085-1327; 00-022-1235 |
| 38.6382 | 5509.81 | 0.0720 | 2.32839 | 19.79 | 01-074-1132; 01-085-1327 |
| 38.7162 | 4859.92 | 0.0787 | 2.32581 | 17.45 | 01-074-1132 |
| 39.5989 | 1495.30 | 0.1968 | 2.27597 | 5.37 | 01-085-0930 |
| 40.5539 | 8990.61 | 0.2165 | 2.22455 | 32.29 | 00-004-0587 |
| 41.3321 | 1316.22 | 0.1771 | 2.18445 | 4.73 | |
| 43.0894 | 3007.54 | 0.1968 | 2.09935 | 10.80 | 01-086-2339 |
| 43.4685 | 2529.91 | 0.1378 | 2.08191 | 9.09 | 01-075-1862 |
| 43.9575 | 1452.44 | 0.1574 | 2.05988 | 5.22 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 44.8756 | 5583.01 | 0.1200 | 2.01818 | 20.05 | 01-074-1132; 01-085-1327 |
| 44.9739 | 6267.31 | 0.0787 | 2.01566 | 22.51 | 01-074-1132 |
| 45.5400 | 17662.02 | 0.1476 | 1.99192 | 63.43 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 46.9979 | 1732.91 | 0.0984 | 1.93347 | 6.22 | 00-004-0864; 01-086-2339 |
| 47.3992 | 1747.98 | 0.1181 | 1.91803 | 6.28 | 01-086-2339 |
| 47.8035 | 1502.86 | 0.1968 | 1.90275 | 5.40 | |
| 50.0933 | 2787.10 | 0.1968 | 1.82101 | 10.01 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.6538 | 901.62 | 0.1181 | 1.73833 | 3.24 | 01-075-1862 |
| 53.5981 | 551.35 | 0.2165 | 1.70991 | 1.98 | |
| 53.9522 | 886.86 | 0.0984 | 1.69953 | 3.18 | 01-077-2064 |
| 55.2167 | 809.27 | 0.2362 | 1.66356 | 2.91 | 00-022-1235; 01-085-0930 |
| 55.7756 | 864.83 | 0.1968 | 1.64821 | 3.11 | 01-074-1132; 00-004-0864 |
| 56.5532 | 4448.87 | 0.1560 | 1.62604 | 15.98 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385; 01-086-2339 |
| 56.7183 | 2722.19 | 0.0960 | 1.62572 | 9.78 | |
| 57.6014 | 1580.00 | 0.1680 | 1.59891 | 5.67 | 01-075-1862; 01-086-2339 |
| 58.4981 | 1312.51 | 0.3840 | 1.57652 | 4.71 | 00-004-0587 |
| 59.4039 | 2759.75 | 0.0960 | 1.55463 | 9.91 | 01-074-1132; 01-075-1620 |
| 60.4525 | 1320.53 | 0.3360 | 1.53014 | 4.74 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 61.6659 | 935.47 | 0.2880 | 1.50292 | 3.36 | |
| 62.4354 | 2598.96 | 0.1080 | 1.48623 | 9.33 | 00-043-1022 |
| 62.6176 | 1972.68 | 0.0960 | 1.48602 | 7.08 | |
| 65.3023 | 3305.02 | 0.1920 | 1.42774 | 11.87 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 66.2862 | 5041.53 | 0.1440 | 1.40892 | 18.11 | 01-074-1132; 01-086-2339; 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171 |
| 68.2234 | 1106.38 | 0.1320 | 1.37355 | 3.97 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.4538 | 995.48 | 0.1440 | 1.36949 | 3.57 | 01-074-1132; 01-085-0930 |
| 69.8436 | 689.60 | 0.2400 | 1.34560 | 2.48 | 01-074-1132; 01-075-1620; 01-080-2171; 01-080-2172; 01-080-1385 |
| 71.5024 | 746.29 | 0.2400 | 1.31840 | 2.68 | 01-075-1620 |
| 73.6157 | 1026.75 | 0.5280 | 1.28569 | 3.69 | 00-004-0587; 00-022-1235; 01-086-2339; 01-085-0930 |
| 75.3213 | 3569.44 | 0.1440 | 1.26075 | 12.82 | 01-077-2064 |
| 75.5442 | 2055.08 | 0.0840 | 1.26071 | 7.38 | |
| 77.3920 | 399.60 | 0.5760 | 1.23211 | 1.44 | 01-074-1132 |
| 78.3673 | 613.64 | 0.1920 | 1.21920 | 2.20 | 01-074-1132; 01-085-1327; 01-086-2339 |
| 81.1542 | 90.45 | 0.7680 | 1.18422 | 0.32 | 01-075-1620; 01-086-2339; 01-085-0930 |
| 82.6366 | 262.04 | 0.4800 | 1.16669 | 0.94 | 01-074-1132; 01-085-1327 |
| 83.9921 | 2248.31 | 0.1440 | 1.15128 | 8.07 | 01-077-2064; 01-086-2339; 01-085-0930 |
| 84.2693 | 1128.10 | 0.1440 | 1.15105 | 4.05 | |
| 87.4461 | 185.23 | 0.4800 | 1.11448 | 0.67 | 00-004-0587; 01-080-2171; 00-004-0864; 01-080-1385; 01-085-0930 |
| 90.3922 | 126.99 | 0.1920 | 1.08566 | 0.46 | 00-022-1235 |
| 91.2532 | 108.72 | 0.5760 | 1.07765 | 0.39 | |
| 94.0614 | 724.19 | 0.1920 | 1.05273 | 2.60 | 00-043-1022; 00-004-0864 |
| 95.2223 | 442.30 | 0.2880 | 1.04294 | 1.59 | |

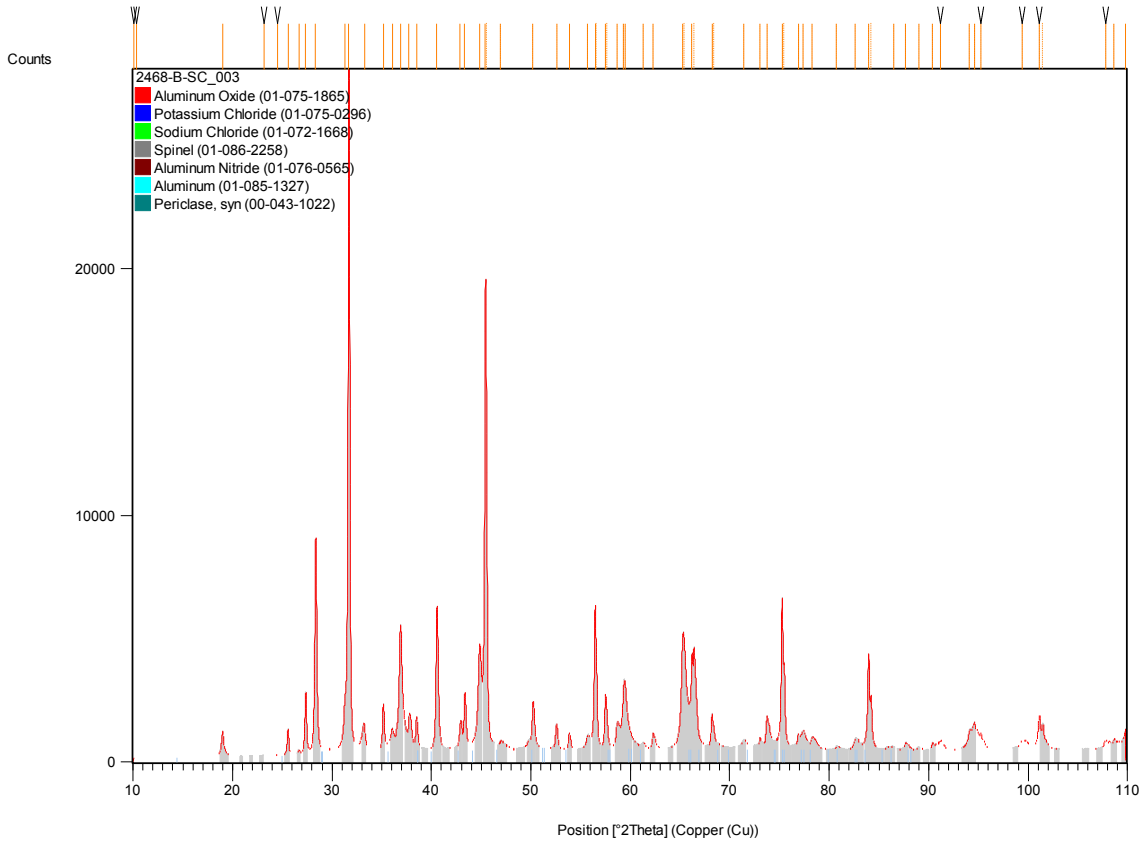
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 99.3158 | 225.82 | 0.6720 | 1.01064 | 0.81 | |
| 101.1450 | 1057.89 | 0.1680 | 0.99724 | 3.80 | |
| 101.4681 | 565.82 | 0.1440 | 0.99741 | 2.03 | |
| 107.7364 | 329.63 | 0.1440 | 0.95374 | 1.18 | |

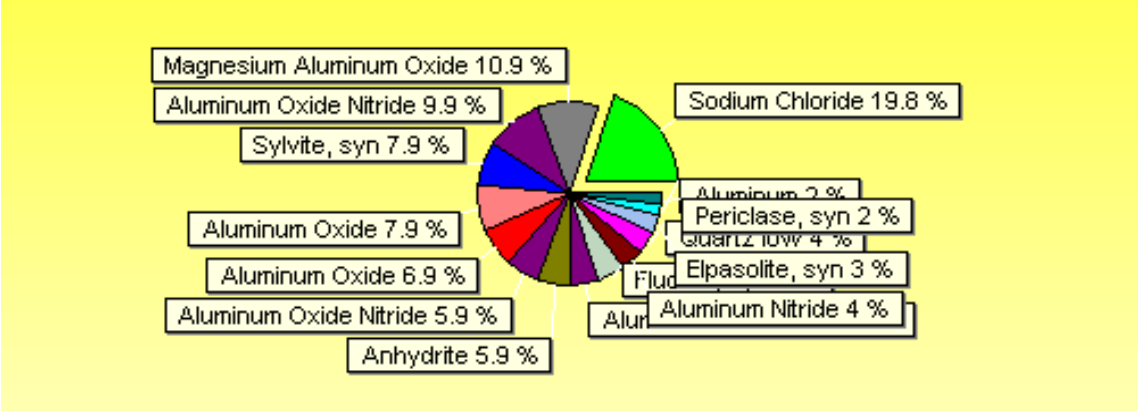
## 1.3: 2468-B-SC

### 1.3.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2468-B-SC_003 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2468-B-SC_003.sd |
| Sample Identification | 2468-B-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 7/29/2010 3:37:00 PM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 30 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 1.3.2: Main Graphics, Analyze View of 2468-B


Counts
2468-B-SC_003
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Magnesium Aluminum Oxide 10.9 %
Aluminum Oxide Nitride 9.9 %
Sylvite, syn 7.9 %
Sodium Chloride 19.8 %
Periclase, syn 2 %
Aluminum Oxide 7.9 %
Elpasolite, syn 3 %
Aluminum Oxide 6.9 %
Aluminum Nitride 4 %
Aluminum Oxide Nitride 5.9 %
Anhydrite 5.9 %

**1.3.3: Pattern List of 2468-B**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 50 | Aluminum Oxide | Al2 O3 | 7 | 20 | 0 |
| 00-004-0587 | 49 | Sylvite, syn | K Cl | 8 | 10 | 0 |
| 01-077-2064 | 49 | Sodium Chloride | Na Cl | 20 | 9 | 0 |
| 01-074-1132 | 51 | Magnesium Aluminum Oxide | Mg Al2 O4 | 11 | 14 | 0 |
| 01-075-1620 | 32 | Aluminum Nitride | Al N | 4 | 7 | 0 |
| 01-080-2171 | 11 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 10 | 12 | 0 |
| 01-080-2172 | 14 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 17 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 12 | 0 |
| 01-085-1327 | 37 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 31 | Periclase, syn | Mg O | 2 | 7 | 0 |
| 00-004-0864 | 19 | Fluorite, syn | Ca F2 | 5 | 5 | 0 |
| 00-022-1235 | 28 | Elpasolite, syn | K2 Na Al F6 | 3 | 16 | 0 |
| 01-086-2270 | 20 | Anhydrite | Ca ( S O4 ) | 6 | 36 | 0 |
| 01-080-1385 | 15 | Aluminum Oxide | Al2.667 O4 | 8 | 12 | 0 |
| 01-085-0335 | 15 | Quartz low | Si O2 | 4 | 16 | 0 |

**1.3.4: Peak List of 2468-B**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.0403 | 10.52 | 0.0492 | 8.81012 | 0.04 | |
| 10.3123 | 3.46 | 0.3149 | 8.57829 | 0.01 | |
| 19.0004 | 1029.62 | 0.0689 | 4.67090 | 3.77 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.1552 | 42.46 | 0.3149 | 3.84133 | 0.16 | |
| 24.5151 | 60.76 | 0.2362 | 3.63125 | 0.22 | |
| 25.5733 | 1071.03 | 0.0787 | 3.48334 | 3.92 | 01-075-1862; 01-086-2270 |
| 26.6567 | 199.97 | 0.1574 | 3.34417 | 0.73 | 01-085-0335 |
| 27.3508 | 2516.35 | 0.1082 | 3.26086 | 9.20 | 01-077-2064 |
| 28.3610 | 8719.33 | 0.1673 | 3.14697 | 31.89 | 00-004-0587; 00-004-0864 |
| 31.2664 | 2050.53 | 0.1181 | 2.86085 | 7.50 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 31.6901 | 27339.12 | 0.1673 | 2.82357 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.2422 | 1165.07 | 0.2165 | 2.69519 | 4.26 | 01-075-1620 |
| 35.1549 | 1985.50 | 0.1279 | 2.55282 | 7.26 | 01-075-1862 |
| 36.0909 | 983.72 | 0.1378 | 2.48873 | 3.60 | 01-075-1620 |
| 36.8766 | 5135.61 | 0.0886 | 2.43749 | 18.78 | 01-074-1132; 00-043-1022 |
| 37.7567 | 1474.16 | 0.1082 | 2.38267 | 5.39 | 01-075-1862; 01-075-1620 |
| 38.5389 | 1403.17 | 0.0984 | 2.33609 | 5.13 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 40.5365 | 5717.19 | 0.1574 | 2.22547 | 20.91 | 00-004-0587 |
| 42.8944 | 1085.38 | 0.2165 | 2.10844 | 3.97 | 00-043-1022 |
| 43.3314 | 2139.68 | 0.1082 | 2.08818 | 7.83 | 01-075-1862; 01-086-2270 |
| 44.8351 | 4219.70 | 0.1574 | 2.02158 | 15.43 | 01-074-1132; 01-085-1327 |
| 45.4287 | 18574.98 | 0.1560 | 1.99488 | 67.94 | 01-077-2064; 01-080-2171; 01-086-2270 |
| 45.5795 | 10356.30 | 0.0960 | 1.99358 | 37.88 | |
| 46.9633 | 326.66 | 0.2880 | 1.93321 | 1.19 | 00-004-0864; 01-086-2270 |
| 50.2171 | 1888.06 | 0.1320 | 1.81531 | 6.91 | 00-004-0587; 01-080-2173; 00-022-1235; 01-085-0335 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 52.5787 | 1008.67 | 0.1320 | 1.73920 | 3.69 | 01-075-1862 |
| 53.8436 | 642.34 | 0.1440 | 1.70129 | 2.35 | 01-077-2064 |
| 55.7076 | 559.72 | 0.3360 | 1.64870 | 2.05 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4772 | 5828.70 | 0.1440 | 1.62804 | 21.32 | 01-077-2064; 01-080-2171 |
| 56.6219 | 3318.28 | 0.0960 | 1.62826 | 12.14 | |
| 57.4625 | 1955.35 | 0.1200 | 1.60245 | 7.15 | 01-075-1862 |
| 57.6666 | 1403.51 | 0.0720 | 1.60123 | 5.13 | |
| 58.6533 | 1081.97 | 0.1680 | 1.57272 | 3.96 | 00-004-0587 |
| 59.3408 | 2605.25 | 0.1440 | 1.55613 | 9.53 | 01-074-1132; 01-075-1620 |
| 59.5164 | 2553.63 | 0.1440 | 1.55196 | 9.34 | 01-075-1862; 01-074-1132 |
| 61.3029 | 281.52 | 0.3840 | 1.51094 | 1.03 | 01-075-1862 |
| 62.3138 | 636.22 | 0.1680 | 1.48884 | 2.33 | 00-043-1022; 01-086-2270 |
| 65.2416 | 4354.30 | 0.2400 | 1.42892 | 15.93 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 65.4325 | 4408.67 | 0.1440 | 1.42876 | 16.13 | |
| 66.1994 | 3858.98 | 0.0720 | 1.41055 | 14.12 | 00-004-0587; 01-077-2064; 01-075-1620 |
| 66.3933 | 4154.84 | 0.0720 | 1.41040 | 15.20 | |
| 68.2203 | 1451.22 | 0.1560 | 1.37361 | 5.31 | 01-075-1862; 00-022-1235; 01-085-0335 |
| 68.4160 | 898.64 | 0.0720 | 1.37356 | 3.29 | |
| 71.4037 | 410.79 | 0.1920 | 1.31998 | 1.50 | 01-075-1620; 01-080-2173; 01-086-2270 |
| 73.0495 | 464.25 | 0.0960 | 1.29425 | 1.70 | 01-077-2064; 01-086-2270 |
| 73.7501 | 1338.86 | 0.1680 | 1.28368 | 4.90 | 00-004-0587; 00-022-1235 |
| 75.2812 | 6154.45 | 0.1560 | 1.26132 | 22.51 | 01-077-2064 |
| 75.4880 | 3432.17 | 0.1320 | 1.26151 | 12.55 | |
| 76.8860 | 643.57 | 0.1200 | 1.23895 | 2.35 | 01-075-1862; 01-086-2270 |
| 77.3997 | 770.13 | 0.2880 | 1.23200 | 2.82 | 01-074-1132; 01-086-2270 |
| 78.2905 | 511.20 | 0.1920 | 1.22020 | 1.87 | 01-074-1132; 01-085-1327 |

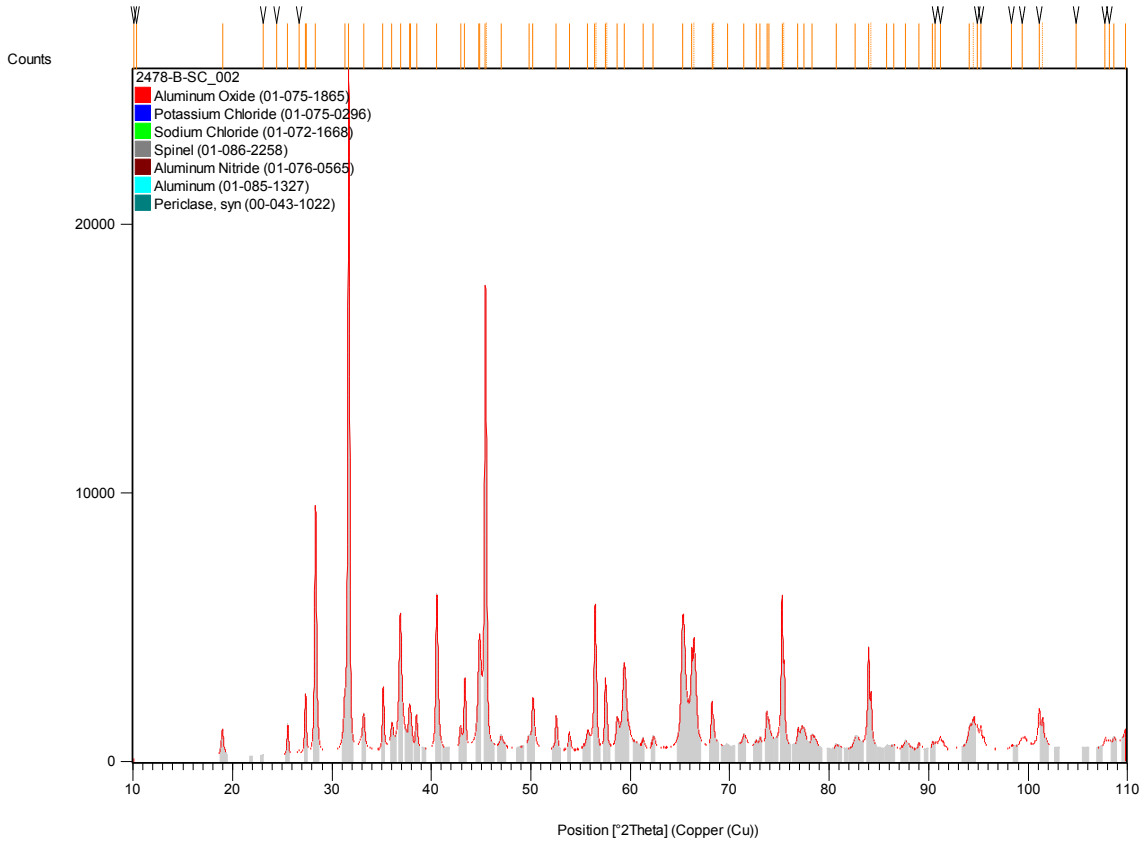
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 80.6919 | 156.67 | 0.1440 | 1.18983 | 0.57 | 01-075-1862 |
| 82.6461 | 461.30 | 0.2880 | 1.16658 | 1.69 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.9430 | 3797.68 | 0.1920 | 1.15183 | 13.89 | 01-077-2064; 01-085-0335 |
| 84.2356 | 2159.89 | 0.1320 | 1.15143 | 7.90 | |
| 86.4409 | 126.42 | 0.5760 | 1.12484 | 0.46 | 01-075-1862 |
| 87.6940 | 294.07 | 0.1920 | 1.11197 | 1.08 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 89.0359 | 123.12 | 0.1680 | 1.09865 | 0.45 | 01-075-1862 |
| 90.3862 | 301.15 | 0.1200 | 1.08571 | 1.10 | 00-022-1235 |
| 91.2057 | 364.76 | 0.1920 | 1.07808 | 1.33 | |
| 94.1166 | 780.22 | 0.2400 | 1.05225 | 2.85 | 00-043-1022; 00-004-0864 |
| 94.6062 | 1123.76 | 0.1920 | 1.04810 | 4.11 | 00-004-0587 |
| 95.2340 | 659.80 | 0.2160 | 1.04284 | 2.41 | |
| 99.3576 | 357.01 | 0.2880 | 1.01032 | 1.31 | |
| 101.1117 | 1385.90 | 0.1080 | 0.99748 | 5.07 | |
| 101.4810 | 1020.68 | 0.1680 | 0.99732 | 3.73 | |
| 107.7887 | 434.32 | 0.1200 | 0.95342 | 1.59 | |
| 108.6402 | 404.41 | 0.2400 | 0.94831 | 1.48 | 00-004-0587 |
| 109.8065 | 980.25 | 0.0600 | 0.94148 | 3.59 | 00-043-1022 |

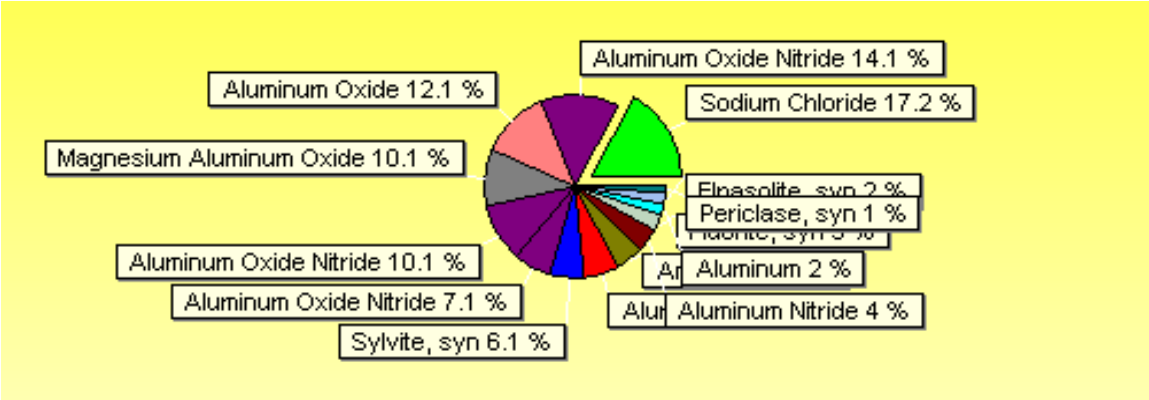
## 1.4: 2478-B-SC

### 1.4.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2478-B-SC_002 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2478-B-SC_002.sd |
| Sample Identification | 2478-B-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 7/30/2010 8:25:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 30 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

**1.4.2: Main Graphics, Analyze View of 2478-B**



**1.4.3: Pattern List of 2478-B**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 42 | Aluminum Oxide | Al2 O3 | 6 | 18 | 0 |
| 00-004-0587 | 43 | Sylvite, syn | K Cl | 6 | 10 | 0 |
| 01-077-2064 | 57 | Sodium Chloride | Na Cl | 17 | 9 | 0 |
| 01-074-1132 | 54 | Magnesium Aluminum Oxide | Mg Al2 O4 | 10 | 15 | 0 |
| 01-075-1620 | 42 | Aluminum Nitride | Al N | 4 | 11 | 0 |
| 01-080-2171 | 11 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 14 | 12 | 0 |
| 01-080-2172 | 13 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 10 | 11 | 0 |
| 01-080-2173 | 14 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 7 | 12 | 0 |
| 01-085-1327 | 32 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 30 | Periclase, syn | Mg O | 1 | 8 | 0 |
| 00-004-0864 | 29 | Fluorite, syn | Ca F2 | 3 | 7 | 0 |
| 00-022-1235 | 19 | Elpasolite, syn | K2 Na Al F6 | 2 | 16 | 0 |
| 01-086-2270 | 17 | Anhydrite | Ca ( S O4 ) | 5 | 36 | 0 |
| 01-080-1385 | 12 | Aluminum Oxide | Al2.667 O4 | 12 | 12 | 0 |

**1.4.4: Peak List of 2478-B**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.0576 | 15.45 | 0.0492 | 8.79502 | 0.06 | |
| 10.3307 | 14.95 | 0.4723 | 8.56306 | 0.06 | |
| 19.0022 | 1000.33 | 0.0886 | 4.67047 | 3.90 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0859 | 46.04 | 0.2362 | 3.85271 | 0.18 | |
| 24.4631 | 67.98 | 0.2362 | 3.63885 | 0.27 | |
| 25.5476 | 1184.48 | 0.0590 | 3.48679 | 4.62 | 01-075-1862; 01-086-2270 |
| 26.6517 | 224.65 | 0.1378 | 3.34480 | 0.88 | |
| 27.3192 | 2288.32 | 0.0720 | 3.26186 | 8.93 | 01-077-2064 |
| 27.3765 | 2188.93 | 0.0492 | 3.25786 | 8.54 | 01-077-2064 |
| 28.3336 | 9200.78 | 0.1476 | 3.14995 | 35.90 | 00-004-0587; 00-004-0864 |
| 31.2777 | 2346.68 | 0.0984 | 2.85985 | 9.16 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 31.6818 | 25630.47 | 0.1870 | 2.82429 | 100.00 | 01-077-2064; 01-080-2171; 01-080-1385 |
| 33.2245 | 1503.35 | 0.1968 | 2.69659 | 5.87 | 01-075-1620 |
| 35.1243 | 2452.23 | 0.0984 | 2.55497 | 9.57 | 01-075-1862 |
| 36.0356 | 1200.77 | 0.1378 | 2.49242 | 4.68 | 01-075-1620 |
| 36.8612 | 5264.69 | 0.0984 | 2.43847 | 20.54 | 01-074-1132; 00-043-1022 |
| 37.7721 | 1849.22 | 0.1440 | 2.37977 | 7.21 | 01-075-1862; 01-075-1620 |
| 37.8979 | 1720.76 | 0.0984 | 2.37412 | 6.71 | 01-075-1620 |
| 38.4850 | 1460.94 | 0.1082 | 2.33924 | 5.70 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.4934 | 5517.34 | 0.2165 | 2.22774 | 21.53 | 00-004-0587 |
| 42.9084 | 1056.78 | 0.1771 | 2.10778 | 4.12 | 00-043-1022 |
| 43.3102 | 2630.53 | 0.1279 | 2.08915 | 10.26 | 01-075-1862; 01-086-2270 |
| 44.7724 | 4014.61 | 0.0840 | 2.02259 | 15.66 | 01-074-1132; 01-085-1327 |
| 44.8594 | 4428.71 | 0.0787 | 2.02054 | 17.28 | 01-074-1132; 01-085-1327 |
| 45.4150 | 17245.00 | 0.1440 | 1.99545 | 67.28 | 01-077-2064; 01-086-2270 |
| 45.5569 | 9699.77 | 0.1080 | 1.99451 | 37.84 | |
| 47.0521 | 686.54 | 0.2160 | 1.92977 | 2.68 | 00-004-0864 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 49.8053 | 709.19 | 0.1920 | 1.82935 | 2.77 | 01-075-1620; 01-080-2171 |
| 50.1790 | 1979.75 | 0.1680 | 1.81660 | 7.72 | 00-004-0587; 01-080-2173; 00-022-1235 |
| 52.5176 | 1300.32 | 0.0960 | 1.74108 | 5.07 | 01-075-1862 |
| 53.8433 | 766.04 | 0.1200 | 1.70130 | 2.99 | 01-077-2064 |
| 55.7136 | 798.80 | 0.2880 | 1.64853 | 3.12 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4235 | 5318.79 | 0.1320 | 1.62946 | 20.75 | 01-077-2064 |
| 56.6069 | 3140.71 | 0.1200 | 1.62866 | 12.25 | |
| 57.4679 | 2656.67 | 0.1200 | 1.60231 | 10.37 | 01-075-1862 |
| 57.6502 | 1706.46 | 0.1200 | 1.60164 | 6.66 | |
| 58.6449 | 1242.70 | 0.1440 | 1.57292 | 4.85 | 00-004-0587 |
| 59.3613 | 3234.40 | 0.2160 | 1.55564 | 12.62 | 01-074-1132; 01-075-1620 |
| 61.2953 | 525.96 | 0.0720 | 1.51111 | 2.05 | 01-075-1862 |
| 62.3003 | 614.05 | 0.1200 | 1.48913 | 2.40 | 00-043-1022; 01-086-2270 |
| 65.2546 | 5043.88 | 0.0840 | 1.42867 | 19.68 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 66.1827 | 3839.14 | 0.0720 | 1.41087 | 14.98 | 01-077-2064; 01-075-1620 |
| 66.3965 | 4203.73 | 0.0840 | 1.41034 | 16.40 | |
| 68.2085 | 1879.13 | 0.1080 | 1.37382 | 7.33 | 01-075-1862; 00-022-1235 |
| 68.3884 | 1167.39 | 0.0720 | 1.37405 | 4.55 | |
| 69.7442 | 268.80 | 0.2880 | 1.34727 | 1.05 | 01-074-1132; 01-075-1620 |
| 71.4048 | 656.44 | 0.2880 | 1.31996 | 2.56 | 01-075-1620; 01-080-2173; 01-086-2270 |
| 72.6353 | 389.42 | 0.1920 | 1.30061 | 1.52 | 01-075-1620 |
| 73.0361 | 537.91 | 0.1200 | 1.29445 | 2.10 | 01-077-2064; 01-086-2270 |
| 73.7098 | 1471.58 | 0.1200 | 1.28428 | 5.74 | 00-004-0587; 00-022-1235 |
| 73.9494 | 1079.30 | 0.1440 | 1.28071 | 4.21 | 01-074-1132 |
| 75.2526 | 5767.23 | 0.1440 | 1.26173 | 22.50 | 01-077-2064 |
| 75.4612 | 3373.95 | 0.0720 | 1.26189 | 13.16 | |
| 76.8496 | 863.49 | 0.1680 | 1.23945 | 3.37 | 01-075-1862 |
| 77.4320 | 845.76 | 0.6240 | 1.23157 | 3.30 | 01-074-1132; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 78.2499 | 607.37 | 0.1920 | 1.22073 | 2.37 | 01-074-1132; 01-085-1327 |
| 80.6851 | 264.00 | 0.1920 | 1.18992 | 1.03 | 01-075-1862 |
| 82.6306 | 540.28 | 0.2880 | 1.16676 | 2.11 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.9763 | 3855.69 | 0.1440 | 1.15146 | 15.04 | 01-077-2064 |
| 84.2204 | 2151.68 | 0.1320 | 1.15160 | 8.40 | |
| 85.7975 | 175.76 | 0.2880 | 1.13162 | 0.69 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 86.4419 | 183.07 | 0.3840 | 1.12483 | 0.71 | 01-075-1862 |
| 87.6478 | 366.95 | 0.1200 | 1.11243 | 1.43 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 88.9804 | 253.60 | 0.1680 | 1.09919 | 0.99 | 01-075-1862 |
| 90.3980 | 333.79 | 0.1680 | 1.08560 | 1.30 | 00-022-1235 |
| 90.6708 | 292.57 | 0.1440 | 1.08305 | 1.14 | |
| 91.1740 | 461.77 | 0.1920 | 1.07838 | 1.80 | |
| 94.1149 | 875.08 | 0.2400 | 1.05227 | 3.41 | 00-043-1022; 00-004-0864 |
| 94.5452 | 1196.74 | 0.1200 | 1.05122 | 4.67 | |
| 94.8575 | 883.34 | 0.1440 | 1.04598 | 3.45 | |
| 95.2504 | 844.09 | 0.1920 | 1.04270 | 3.29 | |
| 98.3305 | 113.66 | 0.2880 | 1.01811 | 0.44 | |
| 99.3856 | 428.34 | 0.2880 | 1.01011 | 1.67 | |
| 101.1231 | 1480.69 | 0.1920 | 0.99740 | 5.78 | |
| 101.4726 | 1129.09 | 0.1440 | 0.99738 | 4.41 | |
| 104.8618 | 110.31 | 0.5760 | 0.97184 | 0.43 | |
| 107.7264 | 641.49 | 0.1440 | 0.95380 | 2.50 | |
| 108.1340 | 608.11 | 0.1440 | 0.95133 | 2.37 | |
| 108.6025 | 724.27 | 0.2400 | 0.94853 | 2.83 | 00-004-0587 |
| 109.7705 | 999.16 | 0.0480 | 0.94168 | 3.90 | 00-043-1022 |

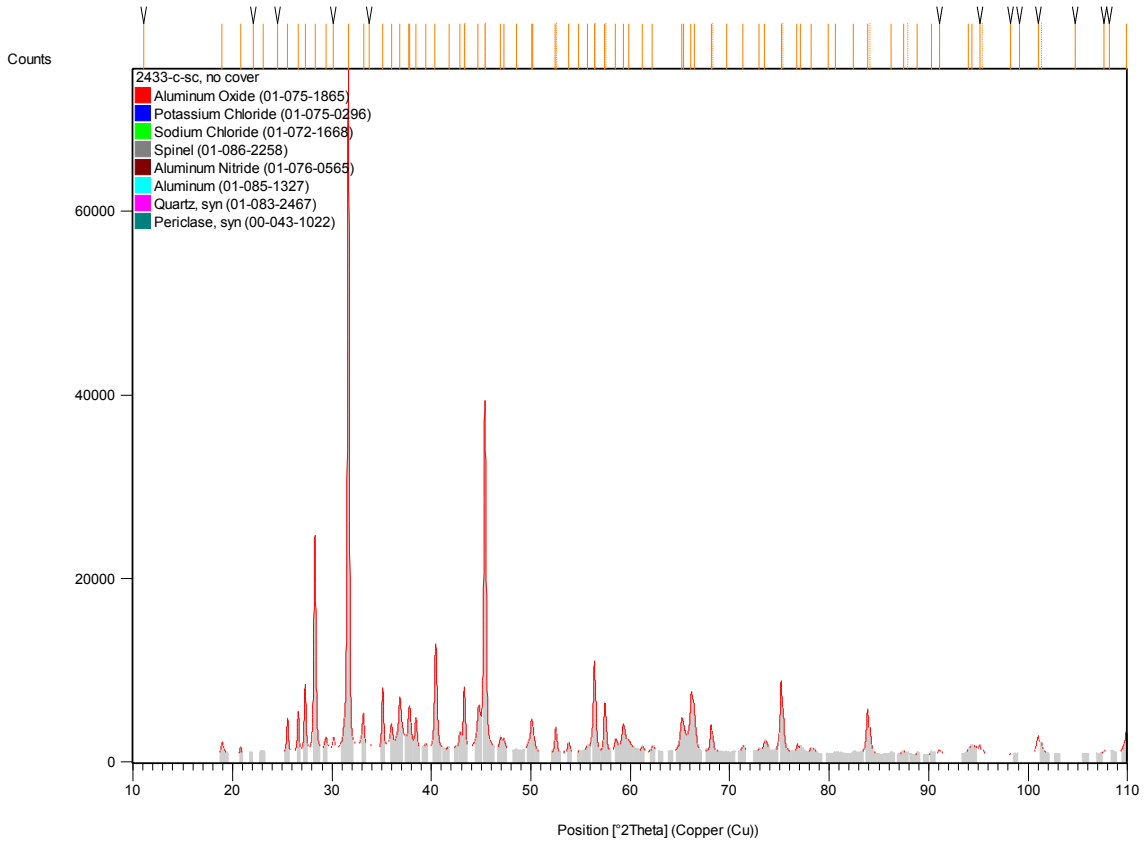
# FACILITY C

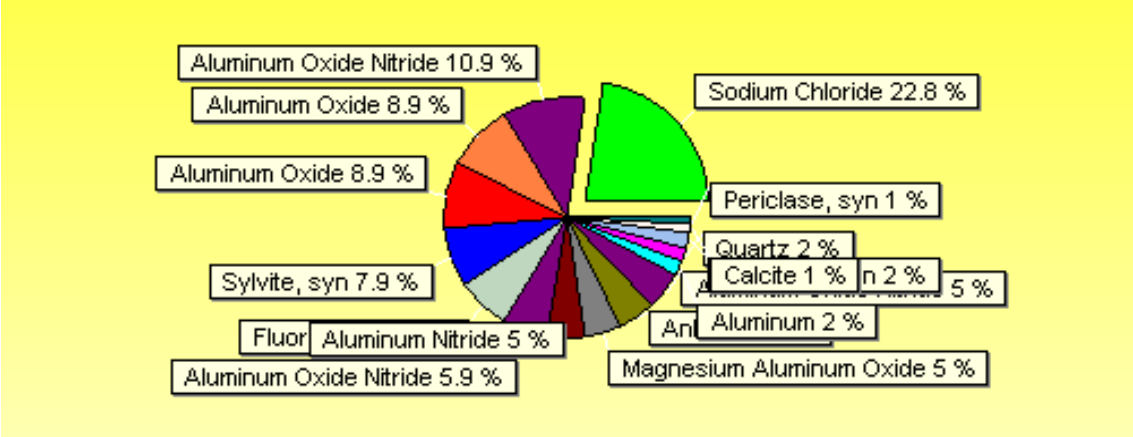
## 2.1: 2433-C-SC

### 2.1.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2433-C-SC, no cover |
| File name | D:\SAP\2433-c-sc, no cover.sd |
| Sample Identification | 2433-C-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by todd in project Ft. Devens. | |
| Measurement Date / Time | 9/7/2010 6:35:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 2.1.1: Main Graphics, Analyze View of 2433-C


Counts
2433-c-sc, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Quartz, syn (01-083-2467)
Periclase, syn (00-043-1022)
60000
40000
20000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Aluminum Oxide Nitride 10.9 %
Aluminum Oxide 8.9 %
Sodium Chloride 22.8 %
Aluminum Oxide 8.9 %
Periclase, syn 1 %
Quartz 2 %
Calcite 1 %
Sylvite, syn 7.9 %
Aluminum 2 %
Aluminum Nitride 5 %
Aluminum Oxide Nitride 5.9 %
Magnesium Aluminum Oxide 5 %

**2.1.3: Pattern List of 2433-C**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 59 | Aluminum Oxide | Al2 O3 | 9 | 20 | 0 |
| 00-004-0587 | 39 | Sylvite, syn | K Cl | 8 | 10 | 0 |
| 01-077-2064 | 48 | Sodium Chloride | Na Cl | 23 | 9 | 0 |
| 01-074-1132 | 45 | Magnesium Aluminum Oxide | Mg Al2 O4 | 5 | 13 | 0 |
| 01-075-1620 | 39 | Aluminum Nitride | Al N | 5 | 10 | 0 |
| 01-080-2171 | 20 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 11 | 13 | 0 |
| 01-080-2172 | 18 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 20 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 13 | 0 |
| 01-085-1327 | 39 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 29 | Periclase, syn | Mg O | 1 | 6 | 0 |
| 00-004-0864 | 24 | Fluorite, syn | Ca F2 | 7 | 7 | 0 |
| 00-022-1235 | 20 | Elpasolite, syn | K2 Na Al F6 | 2 | 14 | 0 |
| 01-086-2270 | 22 | Anhydrite | Ca ( S O4 ) | 5 | 37 | 0 |
| 01-080-1385 | 18 | Aluminum Oxide | Al2.667 O4 | 9 | 12 | 0 |
| 01-086-2340 | 17 | Calcite | Ca ( C O3 ) | 1 | 18 | 0 |
| 01-085-0930 | 32 | Quartz | Si O2 | 2 | 22 | 0 |

**2.1.4: Peak List of 2433-C**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 11.0641 | 181.83 | 0.3936 | 7.99706 | 0.24 | |
| 18.9670 | 1342.79 | 0.1574 | 4.67904 | 1.79 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.8140 | 855.31 | 0.1181 | 4.26783 | 1.14 | 01-085-0930 |
| 22.0522 | 309.92 | 0.4723 | 4.03092 | 0.41 | |
| 23.1096 | 388.96 | 0.1574 | 3.84881 | 0.52 | 01-086-2340 |
| 24.4977 | 399.19 | 0.1771 | 3.63378 | 0.53 | |
| 25.5120 | 3939.31 | 0.0984 | 3.49157 | 5.25 | 01-075-1862; 01-086-2270 |
| 26.5836 | 4731.94 | 0.1181 | 3.35320 | 6.31 | 01-085-0930 |
| 27.3204 | 7548.82 | 0.1279 | 3.26442 | 10.06 | 01-077-2064 |
| 28.2767 | 23928.78 | 0.1968 | 3.15616 | 31.90 | 00-004-0587; 00-004-0864 |
| 29.4178 | 1835.06 | 0.1771 | 3.03629 | 2.45 | 01-086-2340 |
| 30.1487 | 1939.02 | 0.1574 | 2.96432 | 2.58 | |
| 31.6455 | 75016.20 | 0.1771 | 2.82745 | 100.00 | 01-077-2064; 01-080-2171 |
| 33.1579 | 4517.37 | 0.0787 | 2.70186 | 6.02 | 01-075-1620 |
| 33.7175 | 1233.90 | 0.2362 | 2.65829 | 1.64 | |
| 35.0699 | 7041.67 | 0.0886 | 2.55881 | 9.39 | 01-075-1862 |
| 35.9821 | 3395.74 | 0.1574 | 2.49601 | 4.53 | 01-075-1620; 01-086-2340 |
| 36.8228 | 6273.39 | 0.1181 | 2.44093 | 8.36 | 01-074-1132; 00-043-1022 |
| 37.7185 | 5185.56 | 0.0840 | 2.38303 | 6.91 | 01-075-1862; 01-080-2173 |
| 37.8212 | 5140.08 | 0.1574 | 2.37876 | 6.85 | 01-075-1862; 01-075-1620 |
| 38.4372 | 4036.61 | 0.0886 | 2.34204 | 5.38 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 39.4031 | 1275.59 | 0.0984 | 2.28683 | 1.70 | 01-080-2172; 01-080-2173; 01-080-1385; 01-086-2340; 01-085-0930 |
| 40.3643 | 10370.73 | 0.1771 | 2.23457 | 13.82 | 00-004-0587; 01-085-0930 |
| 41.7470 | 831.25 | 0.3149 | 2.16370 | 1.11 | 01-075-1862 |
| 42.8567 | 2506.25 | 0.1574 | 2.11020 | 3.34 | 00-043-1022 |
| 43.2972 | 7443.45 | 0.1800 | 2.08802 | 9.92 | 01-075-1862; 01-086-2270; 01-086-2340 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 43.3942 | 5128.43 | 0.0720 | 2.08876 | 6.84 | |
| 44.7004 | 5009.68 | 0.1080 | 2.02568 | 6.68 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3509 | 37722.89 | 0.1680 | 1.99812 | 50.29 | 01-077-2064; 01-086-2270 |
| 45.4687 | 27805.09 | 0.0960 | 1.99818 | 37.07 | |
| 46.9494 | 1985.17 | 0.0960 | 1.93375 | 2.65 | 00-004-0864; 01-086-2270 |
| 47.2601 | 1839.34 | 0.0960 | 1.92176 | 2.45 | 01-086-2270; 01-086-2340 |
| 48.5777 | 610.99 | 0.3360 | 1.87267 | 0.81 | 01-086-2270 |
| 50.0608 | 3914.59 | 0.0720 | 1.82061 | 5.22 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 50.1814 | 3384.93 | 0.1440 | 1.81652 | 4.51 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-085-0930 |
| 52.4661 | 2901.20 | 0.1680 | 1.74267 | 3.87 | 01-075-1862 |
| 52.6227 | 1912.50 | 0.0720 | 1.74217 | 2.55 | |
| 53.7838 | 1421.99 | 0.1680 | 1.70304 | 1.90 | 01-077-2064 |
| 54.8111 | 468.27 | 0.1680 | 1.67352 | 0.62 | 01-085-0930 |
| 55.6566 | 1045.30 | 0.2400 | 1.65009 | 1.39 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.3906 | 10042.72 | 0.1080 | 1.63034 | 13.39 | 01-077-2064 |
| 56.5291 | 6334.87 | 0.0960 | 1.63071 | 8.44 | |
| 57.3875 | 5341.81 | 0.1320 | 1.60436 | 7.12 | 01-075-1862; 01-086-2340; 01-085-0930 |
| 57.5805 | 3682.21 | 0.0480 | 1.60342 | 4.91 | |
| 58.5122 | 1710.64 | 0.1680 | 1.57618 | 2.28 | 00-004-0587 |
| 59.2981 | 3321.62 | 0.2400 | 1.55715 | 4.43 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 59.8563 | 1548.59 | 0.1920 | 1.54395 | 2.06 | 01-075-1862; 01-085-0930 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 61.2098 | 986.67 | 0.1200 | 1.51302 | 1.32 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 62.2137 | 990.60 | 0.1440 | 1.49099 | 1.32 | 00-043-1022; 01-086-2270 |
| 65.1601 | 3887.30 | 0.1920 | 1.43051 | 5.18 | 01-074-1132; 01-085-1327 |
| 65.3249 | 3634.10 | 0.1680 | 1.42730 | 4.84 | 01-074-1132; 01-086-2270 |
| 66.0913 | 6417.80 | 0.1440 | 1.41260 | 8.56 | 01-077-2064; 01-075-1620 |
| 66.4297 | 5276.45 | 0.1680 | 1.40622 | 7.03 | 01-075-1862; 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 68.1221 | 3304.34 | 0.1320 | 1.37535 | 4.40 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.3018 | 2052.88 | 0.1200 | 1.37558 | 2.74 | |
| 69.6623 | 372.69 | 0.2880 | 1.34866 | 0.50 | 01-074-1132; 01-075-1620; 01-086-2340 |
| 71.3348 | 995.09 | 0.2400 | 1.32109 | 1.33 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270 |
| 72.9440 | 757.38 | 0.1440 | 1.29586 | 1.01 | 01-077-2064; 01-086-2270 |
| 73.4951 | 1553.32 | 0.1200 | 1.28750 | 2.07 | 01-086-2340; 01-085-0930 |
| 75.1575 | 8043.10 | 0.1560 | 1.26309 | 10.72 | 01-077-2064 |
| 75.3628 | 4998.66 | 0.1320 | 1.26329 | 6.66 | |
| 76.7639 | 1140.09 | 0.1680 | 1.24062 | 1.52 | 01-075-1862; 01-086-2340 |
| 77.0589 | 973.82 | 0.2400 | 1.23660 | 1.30 | 01-075-1862; 01-086-2270 |
| 78.1945 | 844.03 | 0.2160 | 1.22146 | 1.13 | 01-085-1327 |
| 79.8570 | 154.35 | 0.3840 | 1.20016 | 0.21 | 01-080-2171; 01-080-2172; 01-086-2270; 01-080-1385; |
| 80.5810 | 360.82 | 0.1440 | 1.19119 | 0.48 | 01-075-1862; 01-086-2340 |

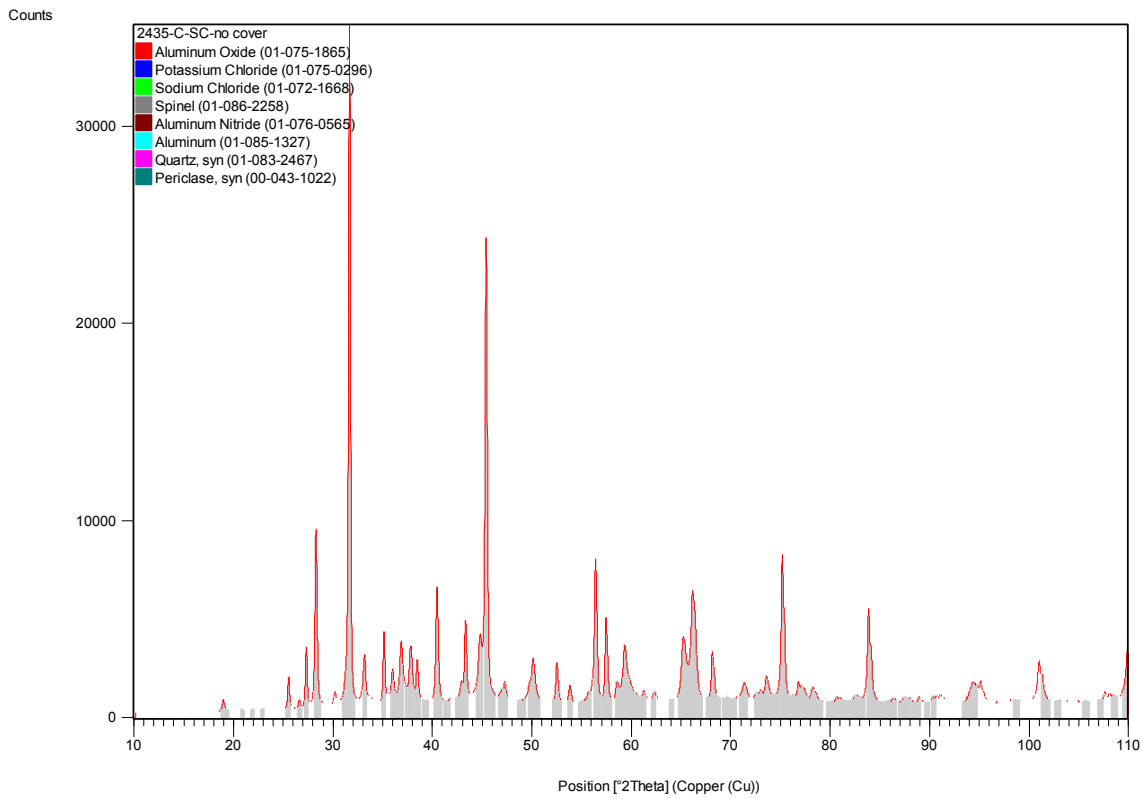
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 82.4323 | 346.99 | 0.3840 | 1.16907 | 0.46 | 01-074-1132; 01-085-1327 |
| 83.8759 | 4926.52 | 0.1320 | 1.15258 | 6.57 | 01-077-2064; 01-086-2340; 01-085-0930 |
| 84.1108 | 2868.13 | 0.1200 | 1.15282 | 3.82 | |
| 86.2547 | 259.32 | 0.2880 | 1.12679 | 0.35 | 01-075-1862; 01-086-2340 |
| 87.4406 | 404.64 | 0.4320 | 1.11454 | 0.54 | 00-004-0587; 01-080-2171; 01-080-2172; 00-004-0864; 01-080-1385; 01-085-0930 |
| 87.9660 | 257.58 | 0.1440 | 1.11199 | 0.34 | |
| 88.8703 | 310.22 | 0.1920 | 1.10027 | 0.41 | 01-075-1862; 01-086-2270 |
| 90.2620 | 353.61 | 0.1920 | 1.08689 | 0.47 | 00-022-1235 |
| 91.0518 | 538.49 | 0.1440 | 1.07950 | 0.72 | |
| 93.9474 | 632.05 | 0.1440 | 1.05370 | 0.84 | 00-043-1022 |
| 94.3057 | 1077.79 | 0.2400 | 1.05064 | 1.44 | 00-004-0587; 00-004-0864 |
| 95.1346 | 1145.67 | 0.1920 | 1.04367 | 1.53 | |
| 95.4626 | 495.48 | 0.1680 | 1.04353 | 0.66 | |
| 98.2217 | 147.61 | 0.2880 | 1.01894 | 0.20 | |
| 99.1206 | 215.41 | 0.4800 | 1.01210 | 0.29 | |
| 100.9826 | 2056.43 | 0.0840 | 0.99841 | 2.74 | |
| 101.3586 | 1176.78 | 0.1920 | 0.99819 | 1.57 | |
| 104.6909 | 35.03 | 0.5760 | 0.97296 | 0.05 | |
| 107.6213 | 316.21 | 0.1920 | 0.95444 | 0.42 | |
| 108.1660 | 160.99 | 0.4800 | 0.95114 | 0.21 | |
| 109.8493 | 1397.73 | 0.1080 | 0.94123 | 1.86 | 00-043-1022 |

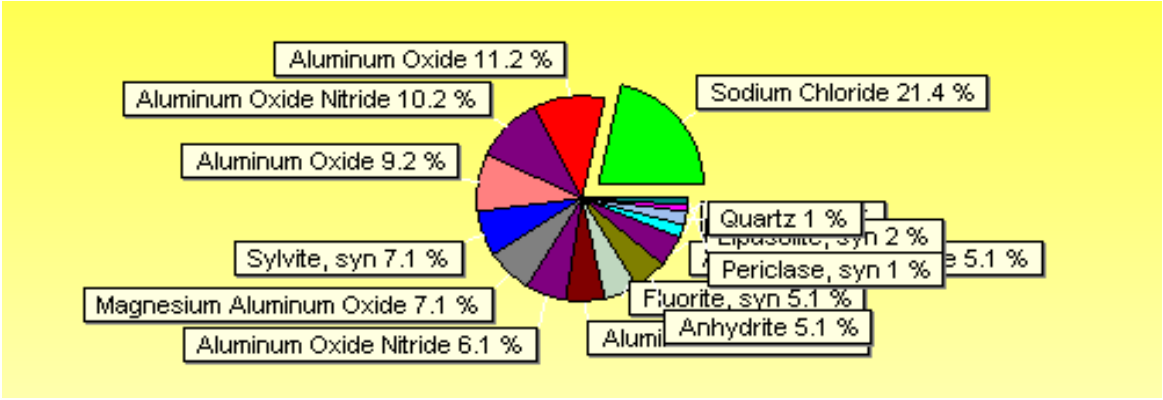
## 2.2: 2435-C-SC

### 2.2.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2435-C-SC-no cover |
| File name | D:\SAP\2435-C-SC-no cover.sd |
| Sample Identification | 2435-C-SC-no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 7/10/2010 5:54:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

**2.2.2: Main Graphics, Analyze View of 2435-C**




**2.2.3: Pattern List of 2435-C**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 59 | Aluminum Oxide | Al2 O3 | 11 | 20 | 0 |
| 00-004-0587 | 42 | Sylvite, syn | K Cl | 7 | 10 | 0 |
| 01-077-2064 | 44 | Sodium Chloride | Na Cl | 21 | 9 | 0 |
| 01-074-1132 | 42 | Magnesium Aluminum Oxide | Mg Al2 O4 | 7 | 13 | 0 |
| 01-075-1620 | 38 | Aluminum Nitride | Al N | 6 | 10 | 0 |
| 01-080-2171 | 14 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 10 | 12 | 0 |
| 01-080-2172 | 16 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 19 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 12 | 0 |
| 01-085-1327 | 41 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 29 | Periclase, syn | Mg O | 1 | 6 | 0 |
| 00-004-0864 | 14 | Fluorite, syn | Ca F2 | 5 | 5 | 0 |
| 00-022-1235 | 23 | Elpasolite, syn | K2 Na Al F6 | 2 | 12 | 0 |
| 01-086-2270 | 17 | Anhydrite | Ca ( S O4 ) | 5 | 36 | 0 |
| 01-080-1385 | 17 | Aluminum Oxide | Al2.667 O4 | 9 | 12 | 0 |
| 01-085-0930 | 9 | Quartz | Si O2 | 1 | 17 | 0 |

**2.2.4: Peak List of 2435-C**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.1523 | 105.29 | 0.0492 | 8.71319 | 0.30 | |
| 10.7137 | 29.86 | 0.9446 | 8.25782 | 0.09 | |
| 18.9908 | 612.16 | 0.0886 | 4.67324 | 1.76 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.8139 | 114.88 | 0.1181 | 4.26785 | 0.33 | 01-085-0930 |
| 23.0997 | 131.34 | 0.2362 | 3.85045 | 0.38 | 01-086-2270 |
| 24.4843 | 167.49 | 0.1968 | 3.63574 | 0.48 | |
| 25.5721 | 1687.94 | 0.1378 | 3.48349 | 4.84 | 01-075-1862; 01-086-2270 |
| 26.5893 | 595.77 | 0.1771 | 3.35249 | 1.71 | 01-085-0930 |
| 27.3429 | 3207.29 | 0.1378 | 3.26179 | 9.20 | 01-077-2064 |
| 28.2919 | 9209.78 | 0.1968 | 3.15450 | 26.40 | 00-004-0587; 00-004-0864 |
| 30.1574 | 909.86 | 0.1771 | 2.96348 | 2.61 | |
| 31.6610 | 34880.69 | 0.1968 | 2.82609 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.1801 | 2800.47 | 0.1279 | 2.70009 | 8.03 | 01-075-1620 |
| 35.1157 | 3938.07 | 0.1279 | 2.55557 | 11.29 | 01-075-1862 |
| 35.9872 | 2009.85 | 0.1968 | 2.49567 | 5.76 | 01-075-1620 |
| 36.8284 | 3352.82 | 0.0886 | 2.44057 | 9.61 | 01-074-1132; 00-043-1022 |
| 37.7503 | 3069.79 | 0.2362 | 2.38306 | 8.80 | 01-075-1862; 01-075-1620 |
| 38.4910 | 2470.43 | 0.1968 | 2.33889 | 7.08 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.3895 | 5096.87 | 0.0840 | 2.23138 | 14.61 | 00-004-0587; 01-085-0930 |
| 40.4531 | 6198.69 | 0.1082 | 2.22986 | 17.77 | 00-004-0587; 01-085-0930 |
| 41.7896 | 557.55 | 0.3149 | 2.16159 | 1.60 | 01-075-1862 |
| 42.9212 | 1418.03 | 0.1574 | 2.10718 | 4.07 | 00-043-1022 |
| 43.2738 | 4015.35 | 0.1574 | 2.09083 | 11.51 | 01-075-1862; 01-086-2270 |
| 44.7209 | 3380.55 | 0.1082 | 2.02648 | 9.69 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3709 | 23160.42 | 0.1378 | 1.99895 | 66.40 | 01-077-2064; 01-086-2270 |
| 47.2497 | 1340.12 | 0.0787 | 1.92375 | 3.84 | 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 49.7409 | 1296.75 | 0.1181 | 1.83309 | 3.72 | 01-075-1620 |
| 50.1076 | 2555.80 | 0.1082 | 1.82053 | 7.33 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.4953 | 2245.80 | 0.1378 | 1.74321 | 6.44 | 01-075-1862 |
| 53.8073 | 1117.94 | 0.0984 | 1.70376 | 3.21 | 01-077-2064 |
| 55.6462 | 761.60 | 0.1574 | 1.65174 | 2.18 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.3927 | 7541.18 | 0.1320 | 1.63028 | 21.62 | 01-077-2064 |
| 56.5586 | 4492.07 | 0.0720 | 1.62993 | 12.88 | |
| 57.4383 | 4494.55 | 0.1560 | 1.60306 | 12.89 | 01-075-1862; 01-085-0930 |
| 57.6076 | 2900.02 | 0.0840 | 1.60273 | 8.31 | |
| 58.5182 | 1263.14 | 0.1920 | 1.57603 | 3.62 | 00-004-0587 |
| 59.3019 | 3093.38 | 0.1680 | 1.55706 | 8.87 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 61.2766 | 772.69 | 0.1680 | 1.51153 | 2.22 | 01-075-1862; 01-086-2270 |
| 62.2622 | 732.36 | 0.3360 | 1.48995 | 2.10 | 00-043-1022; 01-086-2270 |
| 65.1796 | 3358.10 | 0.3120 | 1.43013 | 9.63 | 01-074-1132; 01-085-1327 |
| 66.1244 | 5760.32 | 0.1320 | 1.41197 | 16.51 | 01-077-2064; 01-075-1620 |
| 66.4736 | 3893.54 | 0.2640 | 1.40540 | 11.16 | 01-075-1862; 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 68.1494 | 2771.55 | 0.1680 | 1.37486 | 7.95 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.3403 | 1723.68 | 0.1200 | 1.37489 | 4.94 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 69.6883 | 429.92 | 0.2880 | 1.34822 | 1.23 | 01-074-1132; 01-075-1620 |
| 71.3729 | 1151.18 | 0.2160 | 1.32048 | 3.30 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270 |
| 72.5367 | 598.53 | 0.2880 | 1.30213 | 1.72 | 01-075-1620 |
| 72.9402 | 767.17 | 0.1680 | 1.29914 | 2.20 | |
| 73.5442 | 1400.34 | 0.3360 | 1.28676 | 4.01 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 75.1640 | 7405.85 | 0.1920 | 1.26300 | 21.23 | 01-077-2064 |
| 75.4254 | 4115.02 | 0.1440 | 1.26240 | 11.80 | |
| 76.7411 | 1050.95 | 0.1560 | 1.24093 | 3.01 | 01-075-1862 |
| 77.2211 | 896.79 | 0.3840 | 1.23441 | 2.57 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.2042 | 835.51 | 0.2400 | 1.22133 | 2.40 | 01-074-1132; 01-085-1327 |
| 80.6353 | 399.33 | 0.1920 | 1.19052 | 1.14 | 01-075-1862 |
| 82.5522 | 452.94 | 0.3840 | 1.16767 | 1.30 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.8380 | 4680.91 | 0.1440 | 1.15301 | 13.42 | 01-077-2064; 01-085-0930 |
| 84.1307 | 2785.04 | 0.1200 | 1.15260 | 7.98 | |
| 86.2338 | 244.17 | 0.2880 | 1.12701 | 0.70 | 01-075-1862 |
| 87.4526 | 315.99 | 0.3360 | 1.11441 | 0.91 | 00-004-0587; 01-080-2171; 01-080-2172; 00-004-0864; 01-080-1385; 01-085-0930 |
| 87.9569 | 277.14 | 0.1440 | 1.11208 | 0.79 | |
| 88.8949 | 298.87 | 0.1920 | 1.10003 | 0.86 | 01-075-1862; 01-086-2270 |
| 90.2658 | 344.33 | 0.1440 | 1.08685 | 0.99 | 00-022-1235 |
| 90.5912 | 315.47 | 0.1440 | 1.08379 | 0.90 | 00-022-1235 |
| 91.1070 | 466.74 | 0.1920 | 1.07899 | 1.34 | |
| 94.3196 | 1061.20 | 0.1920 | 1.05052 | 3.04 | 00-004-0587; 00-004-0864 |
| 95.1079 | 1077.91 | 0.1680 | 1.04389 | 3.09 | |
| 98.2183 | 191.12 | 0.1440 | 1.01897 | 0.55 | |
| 99.1956 | 239.18 | 0.4800 | 1.01154 | 0.69 | |
| 101.0254 | 2080.65 | 0.1080 | 0.99810 | 5.97 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 101.3506 | 1379.02 | 0.1200 | 0.99825 | 3.95 | |
| 103.2407 | 71.20 | 0.5760 | 0.98263 | 0.20 | |
| 104.6543 | 63.05 | 0.5760 | 0.97320 | 0.18 | |
| 107.6227 | 328.91 | 0.1920 | 0.95443 | 0.94 | |

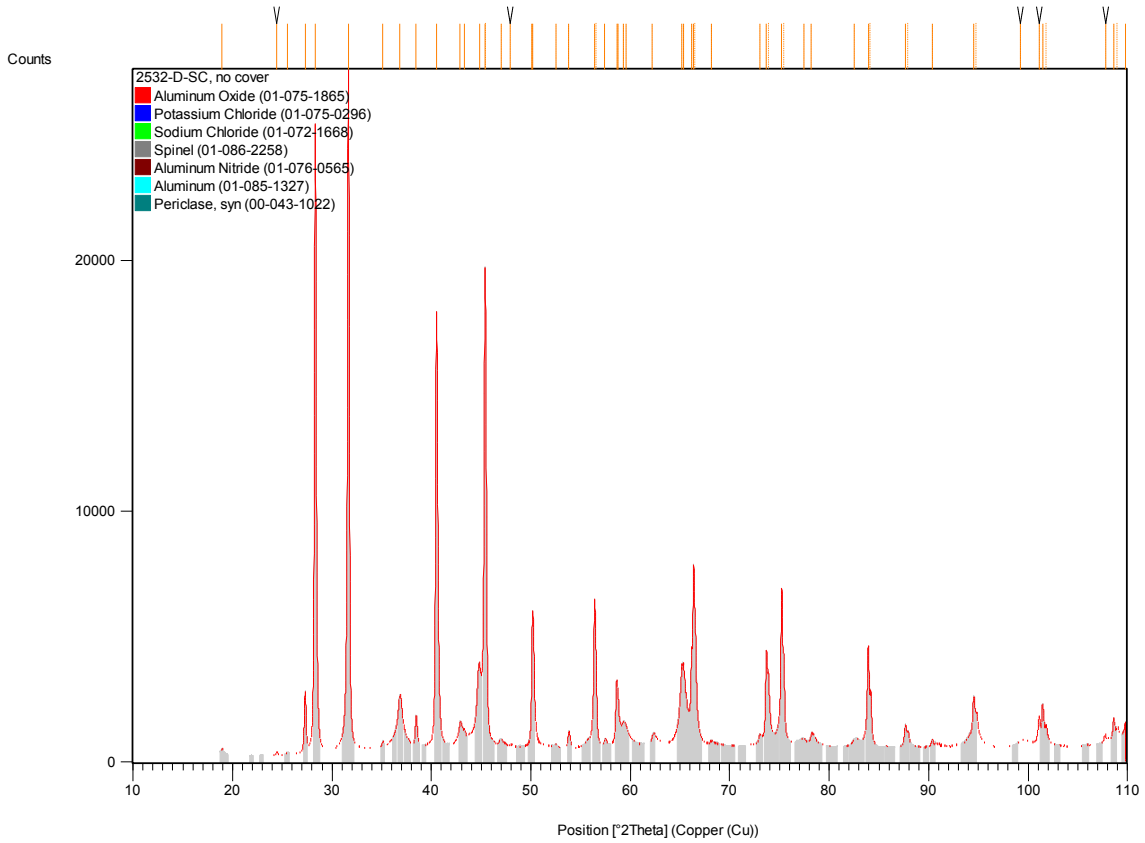
## FACILITY D

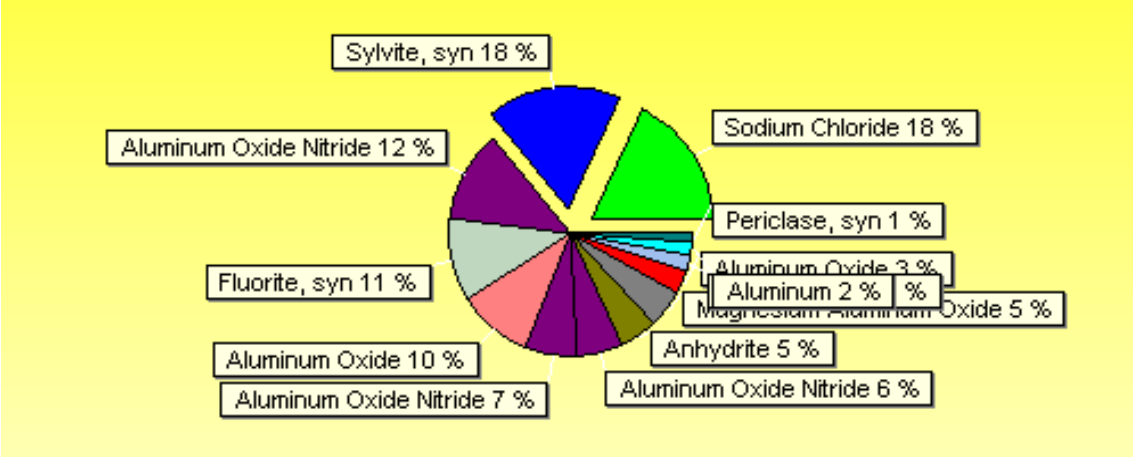
## 3.1: 2532-D-SC

### 3.1:1: Measurement Conditions

Dataset Name 2532-D-SC, no cover
**File name C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no** cover\2532-D-SC, no cover.udf
Sample Identification 2532-D-SC
Comment Exported by X'Pert SW
Generated by Ford in project SAP.
Raw Data Origin PHILIPS-ASCII (.UDF)
Scan Axis Gonio
Start Position [°2Th.] 10.0000
End Position [°2Th.] 110.0000
Step Size [°2Th.] 0.0100
Scan Step Time [s] 5.0000
Scan Type Pre-set time
Offset [°2Th.] 0.0000
Divergence Slit Type Automatic
Irradiated Length [mm] 10.00
Specimen Length [mm] 10.00
Receiving Slit Size [mm] 2.0000
Measurement Temperature [°C] 25.00
Anode Material Cu
K-Alpha1 [Å] 1.54060
K-Alpha2 [Å] 1.54443
K-Beta [Å] 1.39225
K-A2 / K-A1 Ratio 0.50000
Generator Settings 40 mA, 45 kV
Diffractometer Type XPERT
Diffractometer Number 1
Goniometer Radius [mm] 240.00
Dist. Focus-Diverg. Slit [mm] 91.00
Incident Beam Monochromator No
Spinning No

### 3.1.2: Main Graphics, Analyze View of 2532-D


Counts
2532-D-SC, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Sylvite, syn 18 %
Sodium Chloride 18 %
Aluminum Oxide Nitride 12 %
Periclase, syn 1 %
Fluorite, syn 11 %
Aluminum 2 %
Anhydrite 5 %
Aluminum Oxide 10 %
Aluminum Oxide Nitride 7 %
Aluminum Oxide Nitride 6 %

**3.1.3: Pattern List of 2532-D**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 27 | Aluminum Oxide | Al2 O3 | 3 | 15 | 0 |
| 00-004-0587 | 59 | Sylvite, syn | K Cl | 18 | 10 | 0 |
| 01-077-2064 | 52 | Sodium Chloride | Na Cl | 18 | 9 | 0 |
| 01-074-1132 | 43 | Magnesium Aluminum Oxide | Mg Al2 O4 | 5 | 12 | 0 |
| 01-080-2171 | 13 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 12 | 11 | 0 |
| 01-080-2172 | 14 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 7 | 11 | 0 |
| 01-080-2173 | 15 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 6 | 11 | 0 |
| 01-085-1327 | 39 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 31 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 24 | Fluorite, syn | Ca F2 | 11 | 6 | 0 |
| 00-022-1235 | 21 | Elpasolite, syn | K2 Na Al F6 | 2 | 14 | 0 |
| 01-086-2270 | 12 | Anhydrite | Ca ( S O4 ) | 5 | 30 | 0 |
| 01-080-1385 | 15 | Aluminum Oxide | Al2.667 O4 | 10 | 11 | 0 |

**3.1.4: Peak List of 2532-D**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9620 | 283.00 | 0.1378 | 4.68028 | 1.07 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 24.4706 | 122.09 | 0.1378 | 3.63776 | 0.46 | |
| 25.5090 | 88.77 | 0.1574 | 3.49197 | 0.34 | 01-075-1862; 01-086-2270 |
| 27.3010 | 2409.85 | 0.1082 | 3.26671 | 9.11 | 01-077-2064 |
| 28.3018 | 24409.16 | 0.1771 | 3.15342 | 92.24 | 00-004-0587; 00-004-0864 |
| 31.6318 | 26462.19 | 0.1378 | 2.82863 | 100.00 | 01-077-2064; 01-080-2171 |
| 35.1231 | 307.84 | 0.1181 | 2.55506 | 1.16 | 01-075-1862 |
| 36.7827 | 2074.55 | 0.1378 | 2.44349 | 7.84 | 01-074-1132; 00-043-1022 |
| 38.4670 | 1312.56 | 0.0787 | 2.34030 | 4.96 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.4879 | 17045.68 | 0.1181 | 2.22803 | 64.42 | 00-004-0587 |
| 42.8771 | 1056.54 | 0.1378 | 2.10925 | 3.99 | 00-043-1022 |
| 43.3329 | 736.89 | 0.1181 | 2.08811 | 2.78 | 01-075-1862; 01-086-2270 |
| 44.8077 | 3414.93 | 0.2755 | 2.02275 | 12.90 | 01-074-1132; 01-085-1327 |
| 45.3865 | 19102.73 | 0.1440 | 1.99664 | 72.19 | 01-077-2064; 01-086-2270 |
| 45.5086 | 12176.62 | 0.0960 | 1.99652 | 46.02 | |
| 46.9751 | 362.82 | 0.2400 | 1.93276 | 1.37 | 00-004-0864; 01-086-2270 |
| 47.9542 | 145.28 | 0.2880 | 1.89555 | 0.55 | |
| 50.1295 | 4763.70 | 0.0600 | 1.81828 | 18.00 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 50.1790 | 5467.11 | 0.0960 | 1.81660 | 20.66 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235 |
| 52.5190 | 126.74 | 0.2880 | 1.74104 | 0.48 | 01-075-1862 |
| 53.8148 | 694.99 | 0.1440 | 1.70213 | 2.63 | 01-077-2064 |
| 56.3843 | 5528.13 | 0.1680 | 1.63050 | 20.89 | 01-077-2064 |
| 56.5722 | 3411.56 | 0.1200 | 1.62957 | 12.89 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 57.4467 | 339.56 | 0.2880 | 1.60285 | 1.28 | 01-075-1862 |
| 58.6285 | 2657.62 | 0.1320 | 1.57332 | 10.04 | 00-004-0587 |
| 58.7671 | 2213.99 | 0.1200 | 1.56994 | 8.37 | 00-004-0587 |
| 59.2988 | 1026.67 | 0.2160 | 1.55713 | 3.88 | 01-074-1132; 01-086-2270 |
| 59.5350 | 885.53 | 0.3840 | 1.55152 | 3.35 | 01-075-1862; 01-074-1132 |
| 62.2337 | 490.00 | 0.3840 | 1.49056 | 1.85 | 00-043-1022; 01-086-2270 |
| 65.1837 | 3174.83 | 0.1920 | 1.43005 | 12.00 | 01-074-1132; 01-085-1327 |
| 65.3070 | 3311.24 | 0.1680 | 1.42765 | 12.51 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 66.1492 | 3633.61 | 0.0960 | 1.41150 | 13.73 | 01-077-2064 |
| 66.3612 | 7242.26 | 0.1320 | 1.40750 | 27.37 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 66.5690 | 4347.90 | 0.1080 | 1.40710 | 16.43 | |
| 68.1812 | 236.29 | 0.1920 | 1.37430 | 0.89 | 01-075-1862; 00-022-1235 |
| 73.0062 | 436.85 | 0.1920 | 1.29491 | 1.65 | 01-077-2064; 01-086-2270 |
| 73.6878 | 3786.38 | 0.1320 | 1.28461 | 14.31 | 00-004-0587; 00-022-1235 |
| 73.9073 | 2754.43 | 0.1200 | 1.28452 | 10.41 | |
| 75.2245 | 6272.04 | 0.1680 | 1.26213 | 23.70 | 01-077-2064 |
| 75.4641 | 3381.13 | 0.1200 | 1.26185 | 12.78 | |
| 77.4327 | 297.82 | 0.4800 | 1.23156 | 1.13 | 01-074-1132; 01-086-2270 |
| 78.2188 | 537.54 | 0.1920 | 1.22114 | 2.03 | 01-074-1132; 01-085-1327 |
| 82.4724 | 298.68 | 0.2880 | 1.16860 | 1.13 | 01-074-1132; 01-085-1327 |
| 83.9441 | 4015.57 | 0.1440 | 1.15182 | 15.17 | 01-077-2064 |
| 84.1675 | 2293.14 | 0.0840 | 1.15219 | 8.67 | |
| 87.6582 | 908.79 | 0.1440 | 1.11233 | 3.43 | 00-004-0587; 01-080-2171; |

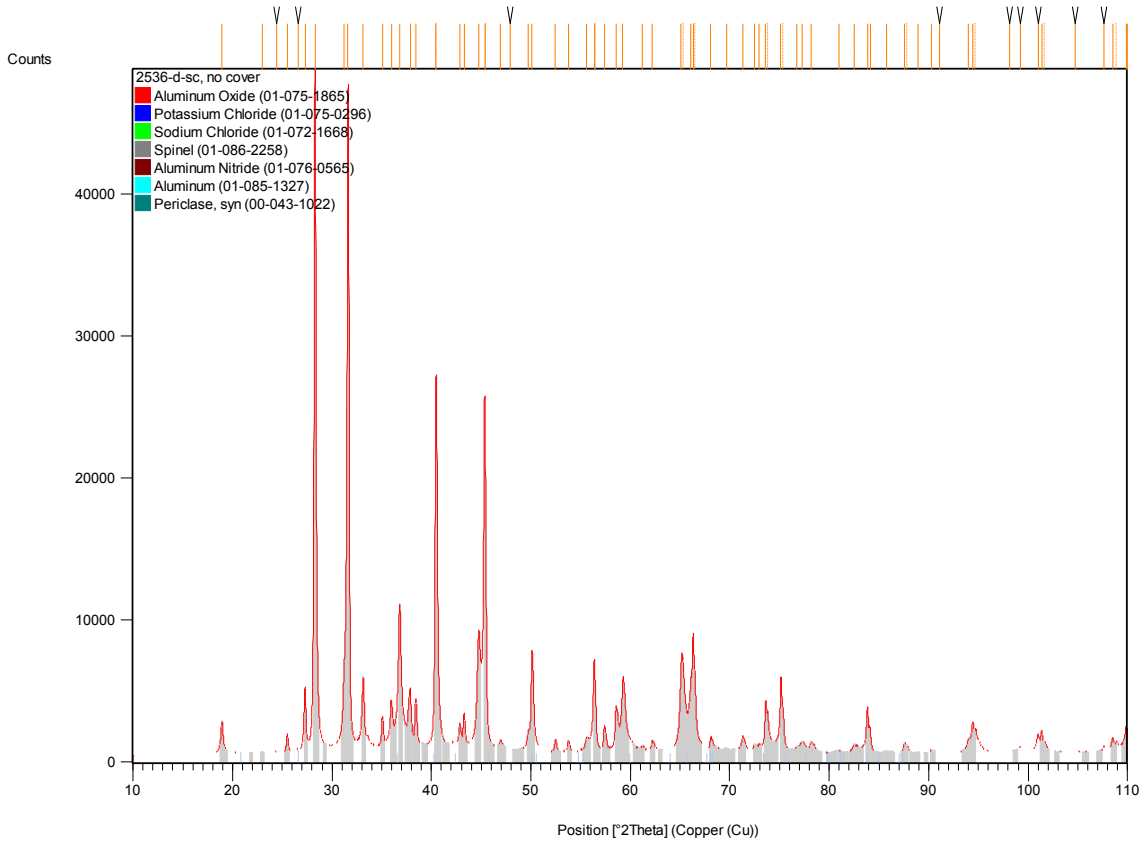
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| | | | | | 01-080-2172; 01-080-2173; 01-080-1385 |
| 87.9440 | 620.76 | 0.1440 | 1.11221 | 2.35 | |
| 90.3685 | 304.75 | 0.1440 | 1.08588 | 1.15 | 00-022-1235 |
| 94.4877 | 1867.63 | 0.0960 | 1.04910 | 7.06 | 00-004-0587; 00-004-0864 |
| 94.8264 | 1349.97 | 0.1440 | 1.04884 | 5.10 | |
| 99.2268 | 216.51 | 0.7680 | 1.01130 | 0.82 | |
| 101.0832 | 1140.37 | 0.0840 | 0.99768 | 4.31 | |
| 101.4460 | 1675.18 | 0.1680 | 0.99510 | 6.33 | 00-004-0587 |
| 101.8271 | 804.55 | 0.1920 | 0.99487 | 3.04 | |
| 107.7595 | 618.28 | 0.1920 | 0.95360 | 2.34 | |
| 108.6038 | 1397.59 | 0.0960 | 0.94852 | 5.28 | 00-004-0587 |
| 108.9826 | 1107.36 | 0.1920 | 0.94863 | 4.18 | |
| 109.7756 | 1443.59 | 0.0480 | 0.94165 | 5.46 | 00-043-1022 |

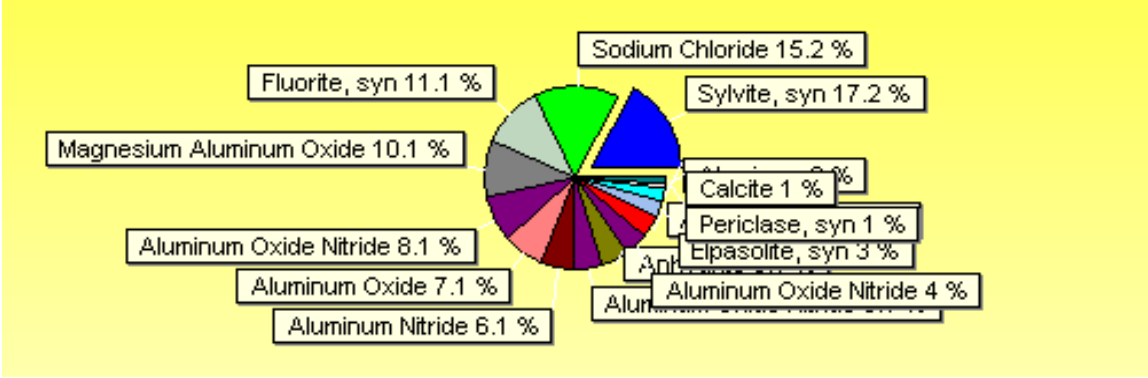
## 3.2: 2536-D-SC

### 3.2.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2536-D-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2536-d-sc, no cover.udf |
| Sample Identification | 2536-D-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by todd in project Ft. Devens. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 3.2.2: Main Graphics, Analyze View of 2536-D


Counts
2536-d-sc, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
40000
30000
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Sodium Chloride 15.2 %
Fluorite, syn 11.1 %
Sylvite, syn 17.2 %
Magnesium Aluminum Oxide 10.1 %
Calcite 1 %
Periclase, syn 1 %
Aluminum Oxide Nitride 8.1 %
Elpasolite, syn 3 %
Aluminum Oxide 7.1 %
Aluminum Oxide Nitride 4 %
Aluminum Nitride 6.1 %

**3.2.3: Pattern List of 2536-D**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 45 | Aluminum Oxide | Al2 O3 | 4 | 18 | 0 |
| 00-004-0587 | 52 | Sylvite, syn | K Cl | 17 | 10 | 0 |
| 01-077-2064 | 47 | Sodium Chloride | Na Cl | 15 | 9 | 0 |
| 01-074-1132 | 49 | Magnesium Aluminum Oxide | Mg Al2 O4 | 10 | 15 | 0 |
| 01-075-1620 | 46 | Aluminum Nitride | Al N | 6 | 11 | 0 |
| 01-080-2171 | 16 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 8 | 13 | 0 |
| 01-080-2172 | 17 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 5 | 12 | 0 |
| 01-080-2173 | 17 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 4 | 12 | 0 |
| 01-085-1327 | 34 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 31 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 22 | Fluorite, syn | Ca F2 | 11 | 6 | 0 |
| 00-022-1235 | 27 | Elpasolite, syn | K2 Na Al F6 | 3 | 17 | 0 |
| 01-086-2270 | 20 | Anhydrite | Ca ( S O4 ) | 5 | 35 | 0 |
| 01-080-1385 | 18 | Aluminum Oxide | Al2.667 O4 | 7 | 12 | 0 |
| 01-086-2340 | 9 | Calcite | Ca ( C O3 ) | 1 | 14 | 0 |

**3.2.4: Peak List of 2536-D**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9648 | 2326.35 | 0.0492 | 4.67959 | 4.88 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0226 | 84.84 | 0.2362 | 3.86317 | 0.18 | 01-086-2270; 01-086-2340 |
| 24.4323 | 237.65 | 0.1574 | 3.64336 | 0.50 | |
| 25.4958 | 1324.73 | 0.0787 | 3.49375 | 2.78 | 01-075-1862; 01-086-2270 |
| 26.5895 | 456.64 | 0.1574 | 3.35247 | 0.96 | |
| 27.2825 | 4671.33 | 0.1574 | 3.26888 | 9.79 | 01-077-2064 |
| 28.3136 | 47715.23 | 0.1476 | 3.15213 | 100.00 | 00-004-0587; 00-004-0864 |
| 31.1963 | 5728.80 | 0.0787 | 2.86712 | 12.01 | 01-074-1132; 00-022-1235; 01-086-2270; 01-086-2340 |
| 31.6035 | 46034.18 | 0.1771 | 2.83110 | 96.48 | 01-077-2064 |
| 33.1246 | 5396.51 | 0.1082 | 2.70450 | 11.31 | 01-075-1620 |
| 35.0680 | 2643.60 | 0.0590 | 2.55894 | 5.54 | 01-075-1862 |
| 35.9822 | 3739.81 | 0.1771 | 2.49600 | 7.84 | 01-075-1620; 01-086-2340 |
| 36.7945 | 10538.25 | 0.0590 | 2.44274 | 22.09 | 01-074-1132; 00-043-1022 |
| 37.8534 | 4563.95 | 0.1082 | 2.37681 | 9.56 | 01-075-1862; 01-075-1620 |
| 38.4538 | 3798.12 | 0.0689 | 2.34107 | 7.96 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.4392 | 25620.43 | 0.1800 | 2.22875 | 53.69 | 00-004-0587 |
| 40.5475 | 21963.80 | 0.0720 | 2.22857 | 46.03 | |
| 42.8443 | 2058.79 | 0.2160 | 2.10904 | 4.31 | 00-043-1022 |
| 43.2691 | 2815.07 | 0.1680 | 2.08931 | 5.90 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 44.7702 | 8676.52 | 0.1200 | 2.02268 | 18.18 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3504 | 25190.44 | 0.1560 | 1.99815 | 52.79 | 01-077-2064; 01-086-2270 |
| 45.4570 | 17604.60 | 0.0600 | 1.99866 | 36.90 | |
| 46.9514 | 933.14 | 0.2880 | 1.93367 | 1.96 | 00-004-0864; 01-086-2270 |
| 47.9232 | 349.55 | 0.2880 | 1.89671 | 0.73 | |
| 49.7134 | 1513.63 | 0.1440 | 1.83252 | 3.17 | 01-075-1620 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 50.0670 | 6114.06 | 0.0600 | 1.82040 | 12.81 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 50.1136 | 7203.32 | 0.0480 | 1.82334 | 15.10 | |
| 52.4402 | 999.57 | 0.1440 | 1.74346 | 2.09 | 01-075-1862; 01-086-2270 |
| 53.7472 | 891.88 | 0.1440 | 1.70411 | 1.87 | 01-077-2064 |
| 55.5928 | 1068.06 | 0.2880 | 1.65183 | 2.24 | 01-074-1132; 01-086-2270 |
| 56.3777 | 6536.13 | 0.1320 | 1.63068 | 13.70 | 01-077-2064 |
| 56.5263 | 3831.04 | 0.0960 | 1.63079 | 8.03 | |
| 57.4123 | 2011.73 | 0.0720 | 1.60373 | 4.22 | 01-075-1862; 01-086-2340 |
| 58.5508 | 3167.49 | 0.0960 | 1.57523 | 6.64 | 00-004-0587 |
| 59.2293 | 5045.98 | 0.2160 | 1.55879 | 10.58 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 61.1997 | 492.80 | 0.3840 | 1.51324 | 1.03 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 62.2188 | 831.05 | 0.1680 | 1.49088 | 1.74 | 00-043-1022; 01-086-2270 |
| 65.1245 | 6427.84 | 0.2640 | 1.43121 | 13.47 | 01-074-1132; 01-085-1327 |
| 65.3501 | 5999.05 | 0.0960 | 1.43036 | 12.57 | |
| 66.1005 | 5358.49 | 0.0720 | 1.41242 | 11.23 | 01-077-2064; 01-075-1620 |
| 66.3045 | 8312.54 | 0.1440 | 1.40857 | 17.42 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171 |
| 66.4853 | 5689.75 | 0.1200 | 1.40867 | 11.92 | |
| 68.0903 | 1191.87 | 0.1440 | 1.37591 | 2.50 | 01-075-1862; 00-022-1235 |
| 69.6438 | 396.47 | 0.2400 | 1.34897 | 0.83 | 01-074-1132; 01-075-1620; 01-086-2340 |
| 71.3081 | 1211.83 | 0.1920 | 1.32152 | 2.54 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270 |
| 72.4984 | 589.27 | 0.2400 | 1.30273 | 1.23 | 01-075-1620 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 72.9586 | 644.45 | 0.1440 | 1.29564 | 1.35 | 01-077-2064; 01-086-2270 |
| 73.5986 | 3539.41 | 0.1320 | 1.28595 | 7.42 | 00-004-0587; 00-022-1235; 01-086-2340 |
| 73.8301 | 2774.28 | 0.1200 | 1.28567 | 5.81 | |
| 75.1386 | 5372.07 | 0.1320 | 1.26336 | 11.26 | 01-077-2064 |
| 75.3800 | 3065.32 | 0.0720 | 1.26305 | 6.42 | |
| 76.7518 | 501.40 | 0.1920 | 1.24078 | 1.05 | 01-075-1862 |
| 77.2363 | 831.44 | 0.2880 | 1.23420 | 1.74 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.1901 | 831.94 | 0.1920 | 1.22152 | 1.74 | 01-085-1327 |
| 80.9528 | 170.81 | 0.3840 | 1.18666 | 0.36 | 01-075-1620 |
| 82.5211 | 651.59 | 0.1920 | 1.16803 | 1.37 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.8290 | 3135.95 | 0.0840 | 1.15311 | 6.57 | 01-077-2064; 01-086-2340 |
| 84.1277 | 1668.49 | 0.1440 | 1.14977 | 3.50 | 01-075-1862; 01-077-2064; 01-080-2171; 01-086-2270 |
| 85.7247 | 142.97 | 0.3840 | 1.13239 | 0.30 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 87.5423 | 755.11 | 0.2160 | 1.11350 | 1.58 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385 |
| 87.8473 | 537.56 | 0.1440 | 1.11318 | 1.13 | |
| 88.8869 | 82.77 | 0.3840 | 1.10011 | 0.17 | 01-075-1862; 01-086-2270 |
| 90.2701 | 263.32 | 0.1680 | 1.08681 | 0.55 | 00-022-1235 |
| 91.0829 | 293.16 | 0.3840 | 1.07922 | 0.61 | |
| 93.9785 | 975.10 | 0.1680 | 1.05343 | 2.04 | 00-043-1022 |
| 94.4035 | 2085.79 | 0.1680 | 1.04981 | 4.37 | 00-004-0587; 00-004-0864 |
| 94.7157 | 1610.23 | 0.1440 | 1.04977 | 3.37 | |
| 98.1470 | 147.54 | 0.2880 | 1.01952 | 0.31 | |
| 99.1917 | 475.19 | 0.1440 | 1.01157 | 1.00 | |
| 100.9868 | 1278.47 | 0.1920 | 0.99838 | 2.68 | |
| 101.3529 | 1538.47 | 0.1920 | 0.99576 | 3.22 | 00-004-0587 |

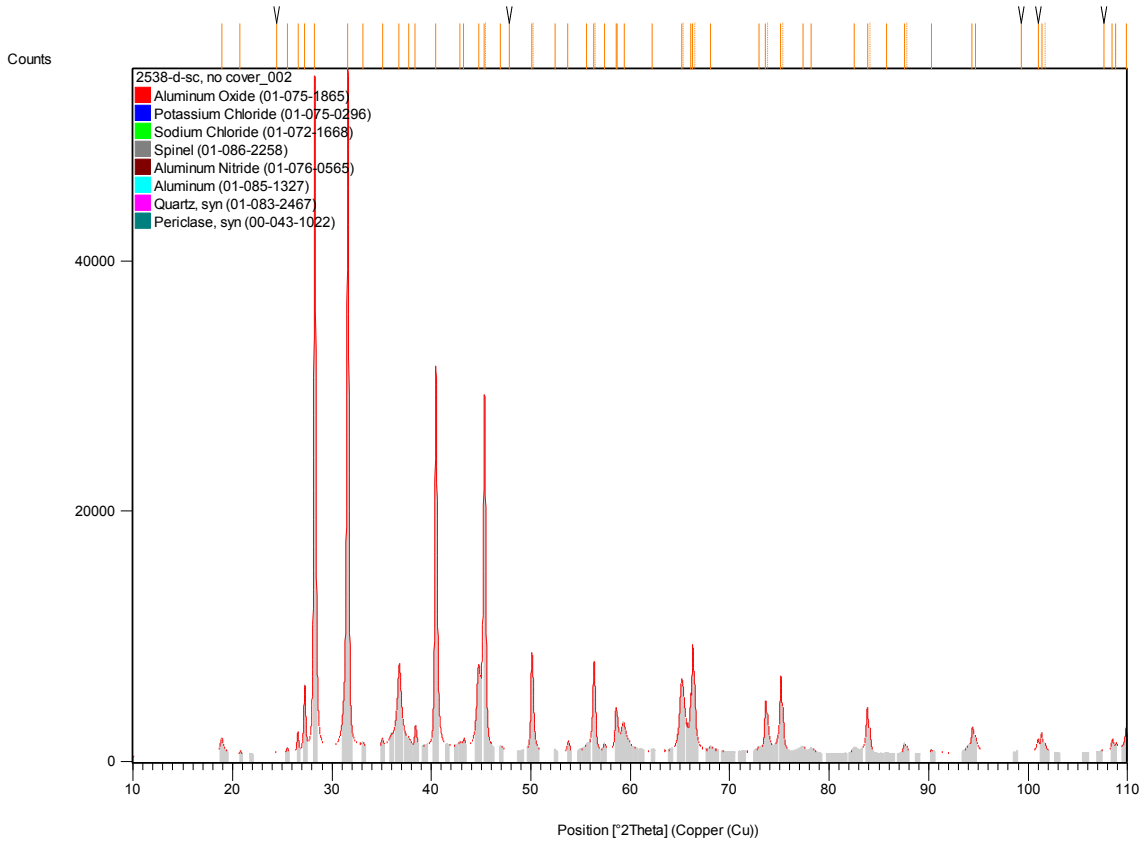
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 101.6689 | 769.34 | 0.1440 | 0.99599 | 1.61 | |
| 104.7063 | 85.99 | 0.4800 | 0.97286 | 0.18 | |
| 107.6206 | 400.24 | 0.1920 | 0.95444 | 0.84 | |
| 108.4821 | 987.04 | 0.2160 | 0.94925 | 2.07 | 00-004-0587 |
| 108.8723 | 747.80 | 0.1920 | 0.94929 | 1.57 | |
| 109.8756 | 1873.64 | 0.1080 | 0.94108 | 3.93 | 00-043-1022 |
| 109.9382 | 1599.01 | 0.0360 | 0.94072 | 3.35 | 00-043-1022 |

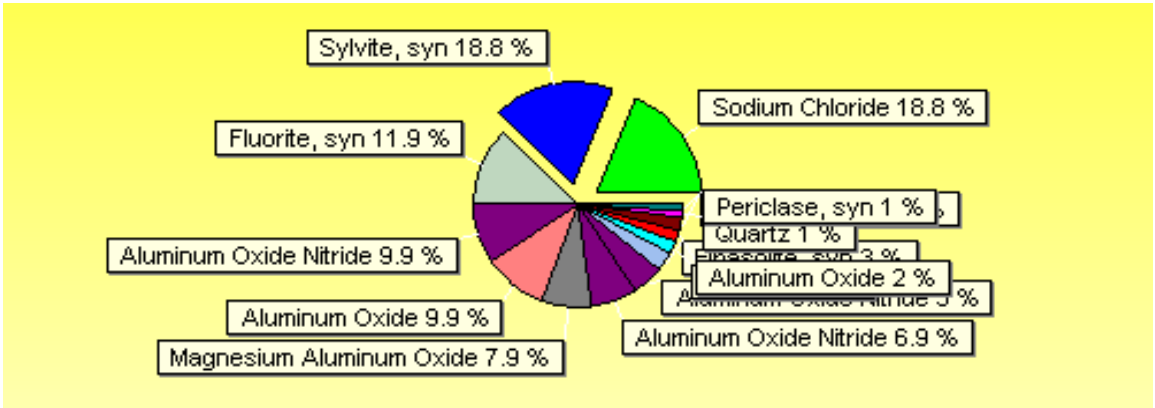
## 3.3: 2538-D-SC

### 3.3.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2538-D-SC, no cover_002 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2538-d-sc, no cover_002.udf |
| Sample Identification | 2538-D-SC, no cover |
| Comment | Exported by X'Pert SW Generated by todd in project Ft. Devens. |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 3.3.2: Main Graphics, Analyze View of 2538-D



**3.3.3: Pattern List of 2538-D**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 36 | Aluminum Oxide | Al2 O3 | 2 | 15 | 0 |
| 00-004-0587 | 61 | Sylvite, syn | K Cl | 19 | 10 | 0 |
| 01-077-2064 | 56 | Sodium Chloride | Na Cl | 19 | 9 | 0 |
| 01-074-1132 | 48 | Magnesium Aluminum Oxide | Mg Al2 O4 | 8 | 14 | 0 |
| 01-075-1620 | 22 | Aluminum Nitride | Al N | 2 | 7 | 0 |
| 01-080-2171 | 18 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 10 | 11 | 0 |
| 01-080-2172 | 19 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 7 | 11 | 0 |
| 01-080-2173 | 20 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 11 | 0 |
| 01-085-1327 | 29 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 21 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 24 | Fluorite, syn | Ca F2 | 12 | 7 | 0 |
| 00-022-1235 | 24 | Elpasolite, syn | K2 Na Al F6 | 3 | 15 | 0 |
| 01-080-1385 | 20 | Aluminum Oxide | Al2.667 O4 | 10 | 11 | 0 |
| 01-085-0930 | 26 | Quartz | Si O2 | 1 | 19 | 0 |

**3.3.4: Peak List of 2538-D:**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9438 | 1231.34 | 0.2558 | 4.68473 | 2.26 | 01-074-1132; 00-022-1235 |
| 20.7704 | 257.58 | 0.1181 | 4.27669 | 0.47 | 01-085-0930 |
| 24.4085 | 210.45 | 0.1378 | 3.64686 | 0.39 | |
| 25.5050 | 310.69 | 0.1181 | 3.49251 | 0.57 | 01-075-1862 |
| 26.5997 | 1330.72 | 0.0689 | 3.35122 | 2.44 | 01-085-0930 |
| 27.2737 | 5273.95 | 0.0787 | 3.26991 | 9.69 | 01-077-2064 |
| 28.2561 | 52942.88 | 0.1968 | 3.15842 | 97.26 | 00-004-0587; 00-004-0864 |
| 31.6053 | 54434.18 | 0.1574 | 2.83095 | 100.00 | 01-077-2064 |
| 33.1502 | 424.05 | 0.1378 | 2.70246 | 0.78 | 01-075-1620 |
| 35.0525 | 783.02 | 0.1378 | 2.56004 | 1.44 | 01-075-1862 |
| 36.7631 | 6715.17 | 0.2165 | 2.44475 | 12.34 | 01-074-1132; 00-043-1022 |
| 37.6912 | 901.41 | 0.1968 | 2.38666 | 1.66 | 01-075-1862; 01-080-2172; 01-080-2173 |
| 38.3839 | 1862.09 | 0.0984 | 2.34517 | 3.42 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.4439 | 30658.58 | 0.1378 | 2.23035 | 56.32 | 00-004-0587; 01-085-0930 |
| 42.8426 | 662.64 | 0.1968 | 2.11087 | 1.22 | 00-043-1022 |
| 43.2395 | 875.46 | 0.1378 | 2.09241 | 1.61 | 01-075-1862 |
| 44.7347 | 6804.80 | 0.2558 | 2.02588 | 12.50 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3114 | 27833.49 | 0.1920 | 1.99978 | 51.13 | 01-077-2064 |
| 45.4595 | 16862.22 | 0.0840 | 1.99856 | 30.98 | |
| 46.9476 | 387.51 | 0.3840 | 1.93382 | 0.71 | 00-004-0864 |
| 47.8585 | 183.54 | 0.1920 | 1.89912 | 0.34 | |
| 50.0744 | 7577.27 | 0.1200 | 1.82015 | 13.92 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 50.2556 | 4956.66 | 0.0840 | 1.81852 | 9.11 | |
| 52.4082 | 204.57 | 0.1440 | 1.74445 | 0.38 | 01-075-1862 |
| 53.7391 | 843.02 | 0.0960 | 1.70435 | 1.55 | 01-077-2064 |
| 55.5823 | 639.63 | 0.2880 | 1.65212 | 1.18 | 01-074-1132; 00-004-0864 |
| 56.3258 | 7178.94 | 0.1560 | 1.63206 | 13.19 | 01-077-2064 |
| 56.5058 | 4251.19 | 0.0960 | 1.63133 | 7.81 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 57.3704 | 702.70 | 0.1920 | 1.60480 | 1.29 | 01-075-1862; 01-085-0930 |
| 58.5603 | 3572.44 | 0.1080 | 1.57500 | 6.56 | 00-004-0587 |
| 58.6915 | 2971.99 | 0.1200 | 1.57179 | 5.46 | 00-004-0587 |
| 59.3577 | 2284.94 | 0.1440 | 1.55573 | 4.20 | 01-074-1132; 01-075-1620 |
| 62.2232 | 227.60 | 0.2880 | 1.49079 | 0.42 | 00-043-1022 |
| 65.1813 | 5821.19 | 0.3120 | 1.43010 | 10.69 | 01-074-1132; 01-085-1327 |
| 65.3088 | 5183.02 | 0.1680 | 1.43116 | 9.52 | |
| 66.0710 | 4505.38 | 0.1200 | 1.41298 | 8.28 | 01-077-2064; 01-075-1620 |
| 66.2792 | 8559.06 | 0.1080 | 1.40905 | 15.72 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171 |
| 66.4828 | 5252.83 | 0.1200 | 1.40872 | 9.65 | |
| 68.0791 | 503.49 | 0.1920 | 1.37611 | 0.92 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 72.9172 | 460.53 | 0.1440 | 1.29627 | 0.85 | 01-077-2064 |
| 73.6113 | 4124.26 | 0.1320 | 1.28575 | 7.58 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 75.1321 | 6081.39 | 0.1680 | 1.26346 | 11.17 | 01-077-2064 |
| 75.3609 | 3473.34 | 0.0960 | 1.26332 | 6.38 | |
| 77.3320 | 486.45 | 0.5760 | 1.23291 | 0.89 | 01-075-1862; 01-074-1132 |
| 78.1427 | 443.77 | 0.2400 | 1.22214 | 0.82 | 01-085-1327; 00-022-1235 |
| 82.5149 | 464.18 | 0.5280 | 1.16810 | 0.85 | 01-074-1132; 01-085-1327 |
| 83.8531 | 3658.78 | 0.1200 | 1.15284 | 6.72 | 01-077-2064; 01-085-0930 |
| 84.0919 | 2017.07 | 0.1440 | 1.15303 | 3.71 | |
| 85.7202 | 48.86 | 0.5760 | 1.13244 | 0.09 | 01-074-1132; 01-075-1620 |
| 87.5360 | 869.56 | 0.1440 | 1.11357 | 1.60 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 87.8267 | 585.14 | 0.1200 | 1.11339 | 1.07 | |
| 90.2347 | 273.20 | 0.1440 | 1.08714 | 0.50 | 00-022-1235 |

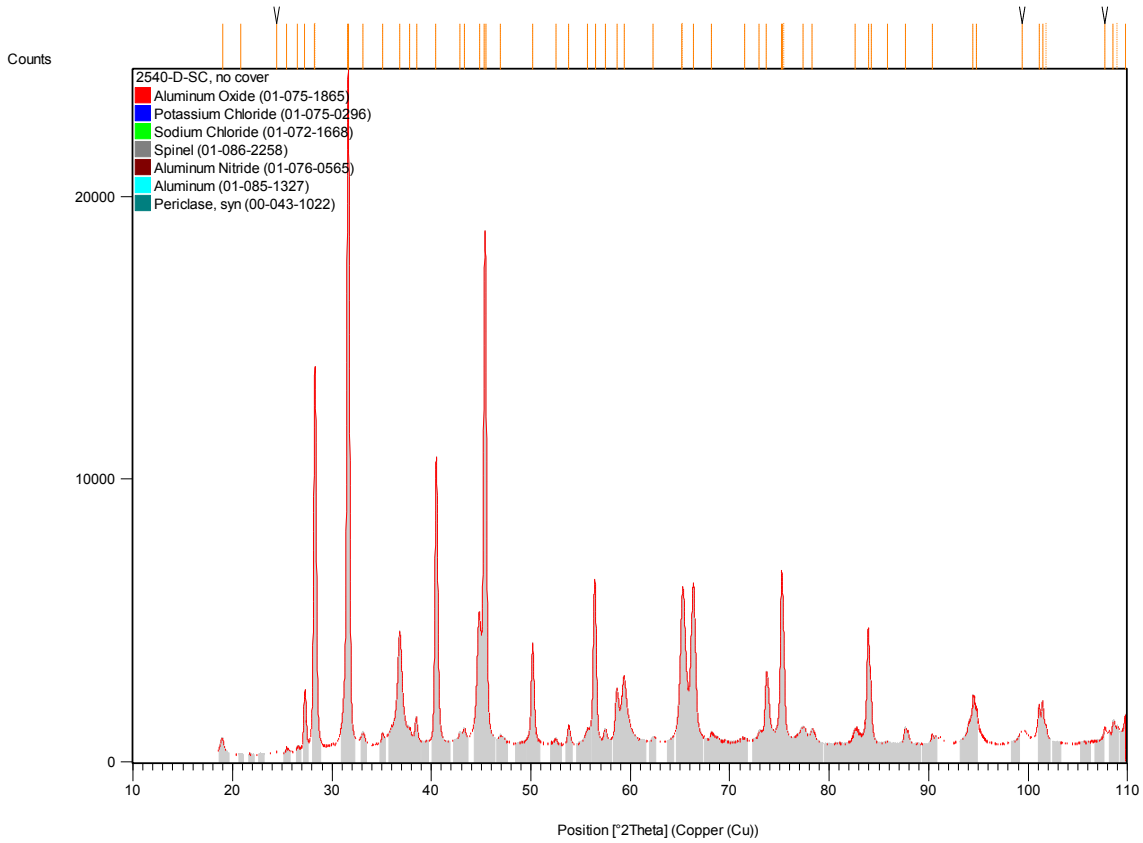
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 94.3880 | 2060.87 | 0.0840 | 1.04994 | 3.79 | 00-004-0587; 00-004-0864 |
| 94.7214 | 1321.76 | 0.1920 | 1.04712 | 2.43 | 00-004-0587 |
| 99.2790 | 362.75 | 0.8640 | 1.01091 | 0.67 | |
| 100.9866 | 1100.77 | 0.1440 | 0.99838 | 2.02 | |
| 101.3540 | 1632.38 | 0.1200 | 0.99575 | 3.00 | 00-004-0587 |
| 101.7437 | 739.52 | 0.1440 | 0.99546 | 1.36 | |
| 107.5972 | 398.67 | 0.2400 | 0.95459 | 0.73 | |
| 108.4551 | 1007.98 | 0.1680 | 0.94941 | 1.85 | 00-004-0587 |
| 108.8391 | 772.45 | 0.1920 | 0.94713 | 1.42 | 00-004-0587 |
| 109.9171 | 1666.59 | 0.0600 | 0.94084 | 3.06 | 00-043-1022 |

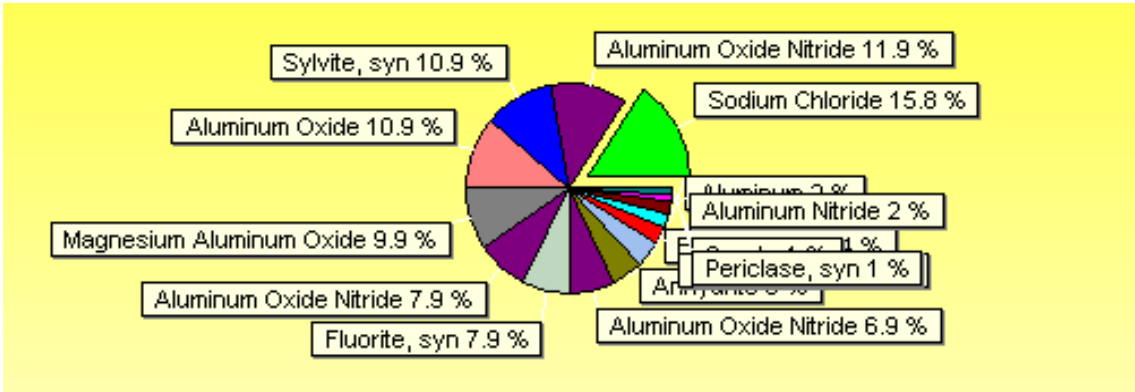
## 3.4: 2540-D-SC

### 3.4.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2540-D-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2540-D-SC, no cover.udf |
| Sample Identification | 2540-D-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 3.4.2: Main Graphics, Analyze View of 2540-D




**3.4.3: Pattern List of 2540-D**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 35 | Aluminum Oxide | Al2 O3 | 3 | 15 | 0 |
| 00-004-0587 | 55 | Sylvite, syn | K Cl | 11 | 10 | 0 |
| 01-077-2064 | 56 | Sodium Chloride | Na Cl | 16 | 9 | 0 |
| 01-074-1132 | 54 | Magnesium Aluminum Oxide | Mg Al2 O4 | 10 | 14 | 0 |
| 01-075-1620 | 26 | Aluminum Nitride | Al N | 2 | 8 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 12 | 11 | 0 |
| 01-080-2172 | 17 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 8 | 11 | 0 |
| 01-080-2173 | 19 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 7 | 11 | 0 |
| 01-085-1327 | 27 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 21 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 22 | Fluorite, syn | Ca F2 | 8 | 7 | 0 |
| 00-022-1235 | 27 | Elpasolite, syn | K2 Na Al F6 | 4 | 17 | 0 |
| 01-086-2270 | 25 | Anhydrite | Ca ( S O4 ) | 5 | 35 | 0 |
| 01-080-1385 | 19 | Aluminum Oxide | Al2.667 O4 | 11 | 11 | 0 |
| 01-085-0930 | 6 | Quartz | Si O2 | 1 | 19 | 0 |

**3.4.4: Peak List of 2540-D**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9959 | 592.28 | 0.3149 | 4.67200 | 2.48 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.8595 | 27.22 | 0.4723 | 4.25862 | 0.11 | 01-085-0930 |
| 24.4649 | 31.89 | 0.7872 | 3.63859 | 0.13 | |
| 25.4704 | 138.62 | 0.1574 | 3.49718 | 0.58 | 01-075-1862; 01-086-2270 |
| 26.5081 | 130.71 | 0.2362 | 3.36258 | 0.55 | 01-085-0930 |
| 27.2415 | 1928.81 | 0.0787 | 3.27370 | 8.06 | 01-077-2064 |
| 28.2125 | 10839.66 | 0.1320 | 3.16058 | 45.30 | 00-004-0587; 00-004-0864 |
| 28.2798 | 13482.11 | 0.0480 | 3.16104 | 56.35 | |
| 28.3219 | 13170.16 | 0.0960 | 3.14862 | 55.04 | 00-004-0587; 00-004-0864; 01-086-2270 |
| 31.5645 | 21506.33 | 0.1560 | 2.83217 | 89.88 | 01-077-2064; 01-080-2171; 01-086-2270; 01-080-1385 |
| 31.6435 | 23927.71 | 0.1800 | 2.82528 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 33.1508 | 458.02 | 0.3840 | 2.70018 | 1.91 | 01-075-1620 |
| 35.0589 | 429.67 | 0.1200 | 2.55747 | 1.80 | 01-075-1862 |
| 36.8349 | 3990.17 | 0.3360 | 2.43814 | 16.68 | 01-074-1132; 00-043-1022; 01-085-0930 |
| 37.7911 | 601.90 | 0.2880 | 2.37861 | 2.52 | 01-075-1862; 01-075-1620; 01-080-2172; 01-080-2173 |
| 38.5195 | 942.09 | 0.2880 | 2.33529 | 3.94 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.4530 | 9813.46 | 0.3000 | 2.22802 | 41.01 | 00-004-0587; 01-085-0930 |
| 42.8461 | 433.76 | 0.2400 | 2.10896 | 1.81 | 00-043-1022 |
| 43.3227 | 571.45 | 0.2880 | 2.08685 | 2.39 | 01-075-1862; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 44.8161 | 4661.75 | 0.3120 | 2.02072 | 19.48 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.2848 | 13747.59 | 0.0840 | 2.00089 | 57.45 | 01-077-2064; 01-086-2270 |
| 45.4432 | 17065.36 | 0.1920 | 1.99428 | 71.32 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 46.9174 | 294.86 | 0.3840 | 1.93500 | 1.23 | 00-004-0864; 01-086-2270 |
| 50.1702 | 3537.08 | 0.0720 | 1.81690 | 14.78 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.5524 | 182.01 | 0.3360 | 1.74001 | 0.76 | 01-075-1862; 01-086-2270 |
| 53.8012 | 644.38 | 0.1680 | 1.70253 | 2.69 | 01-077-2064 |
| 55.6695 | 513.70 | 0.2880 | 1.64973 | 2.15 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4889 | 5305.53 | 0.1200 | 1.62773 | 22.17 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 57.4671 | 519.70 | 0.3360 | 1.60233 | 2.17 | 01-075-1862; 01-085-0930 |
| 58.6422 | 1937.51 | 0.2880 | 1.57299 | 8.10 | 00-004-0587 |
| 59.3717 | 2314.01 | 0.2160 | 1.55539 | 9.67 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 62.3105 | 187.70 | 0.3840 | 1.48891 | 0.78 | 00-043-1022; 01-086-2270 |
| 65.1584 | 4817.07 | 0.2160 | 1.43055 | 20.13 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 65.2785 | 5470.85 | 0.1200 | 1.43175 | 22.86 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 66.3890 | 5430.32 | 0.3840 | 1.40698 | 22.69 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 68.1319 | 337.34 | 0.2880 | 1.37517 | 1.41 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 71.4634 | 156.25 | 0.6720 | 1.31903 | 0.65 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270 |
| 72.9819 | 421.88 | 0.2400 | 1.29528 | 1.76 | 01-077-2064; 01-086-2270 |
| 73.6844 | 2493.45 | 0.1440 | 1.28466 | 10.42 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 75.2201 | 6098.28 | 0.1920 | 1.26220 | 25.49 | 01-077-2064 |
| 75.2935 | 5760.19 | 0.0960 | 1.26115 | 24.07 | 01-077-2064; 01-080-2171; 01-080-1385 |
| 75.5022 | 2835.23 | 0.1440 | 1.26131 | 11.85 | |
| 77.3433 | 603.41 | 0.5760 | 1.23276 | 2.52 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.2884 | 524.70 | 0.3840 | 1.22023 | 2.19 | 01-074-1132; 01-085-1327; 00-043-1022; 00-022-1235; 01-086-2270 |
| 82.5959 | 505.60 | 0.3360 | 1.16716 | 2.11 | 01-074-1132; 01-085-1327; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 83.9310 | 4080.90 | 0.1080 | 1.15196 | 17.06 | 01-075-1862; 01-077-2064; 01-086-2270; 01-085-0930 |
| 84.2426 | 1841.67 | 0.1920 | 1.14850 | 7.70 | 01-075-1862; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 85.8171 | 77.16 | 0.5760 | 1.13141 | 0.32 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 87.6250 | 607.09 | 0.1680 | 1.11267 | 2.54 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 90.3502 | 331.65 | 0.2400 | 1.08605 | 1.39 | 00-022-1235 |
| 94.4590 | 1600.49 | 0.1200 | 1.04934 | 6.69 | 00-004-0587; 00-004-0864 |
| 94.8377 | 1127.45 | 0.2160 | 1.04615 | 4.71 | 00-004-0587 |
| 99.3814 | 449.38 | 0.8640 | 1.01014 | 1.88 | |
| 101.0748 | 1311.73 | 0.1920 | 0.99775 | 5.48 | 00-004-0587 |
| 101.4645 | 1473.39 | 0.1920 | 0.99497 | 6.16 | 00-004-0587; 00-022-1235 |
| 101.8343 | 575.51 | 0.1440 | 0.99482 | 2.41 | |
| 107.6740 | 658.20 | 0.1920 | 0.95412 | 2.75 | |
| 108.5525 | 1091.08 | 0.2160 | 0.94883 | 4.56 | 00-004-0587 |
| 108.9883 | 995.15 | 0.1440 | 0.94860 | 4.16 | |
| 109.7606 | 1510.33 | 0.0480 | 0.94174 | 6.31 | 00-043-1022 |

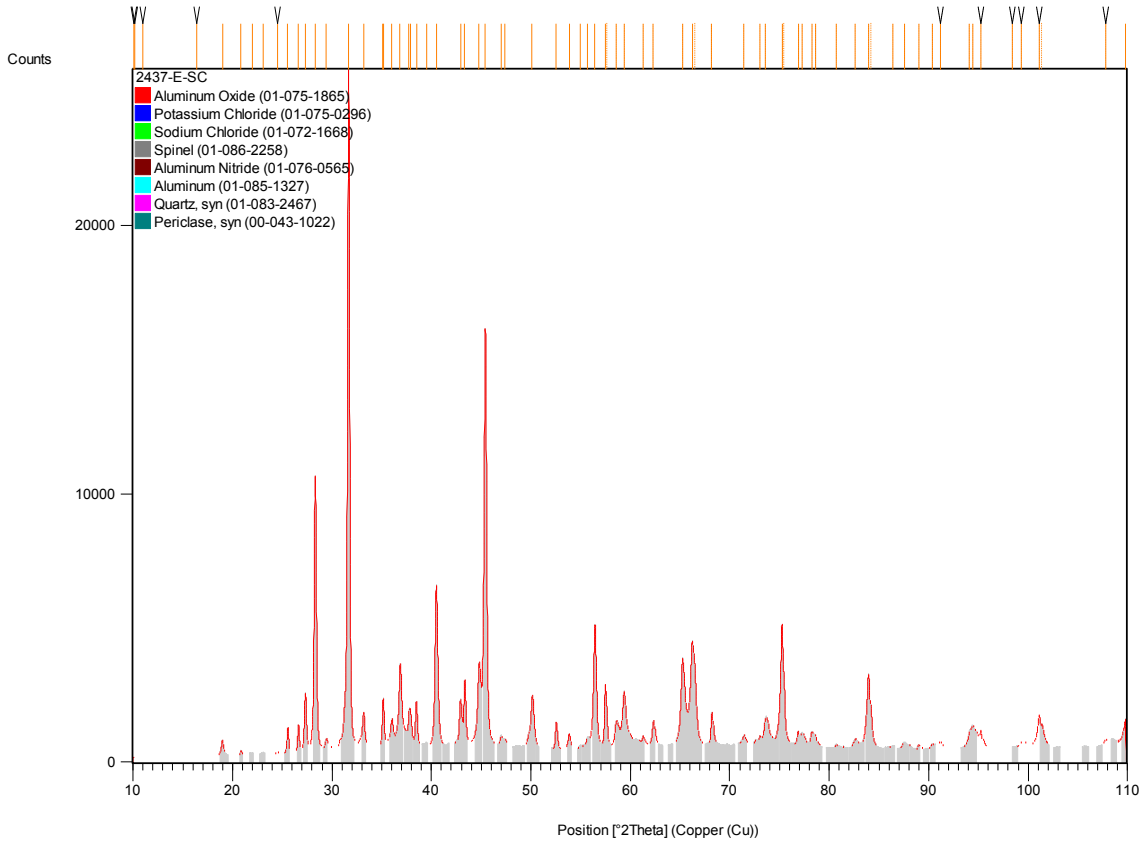
# FACILITY E

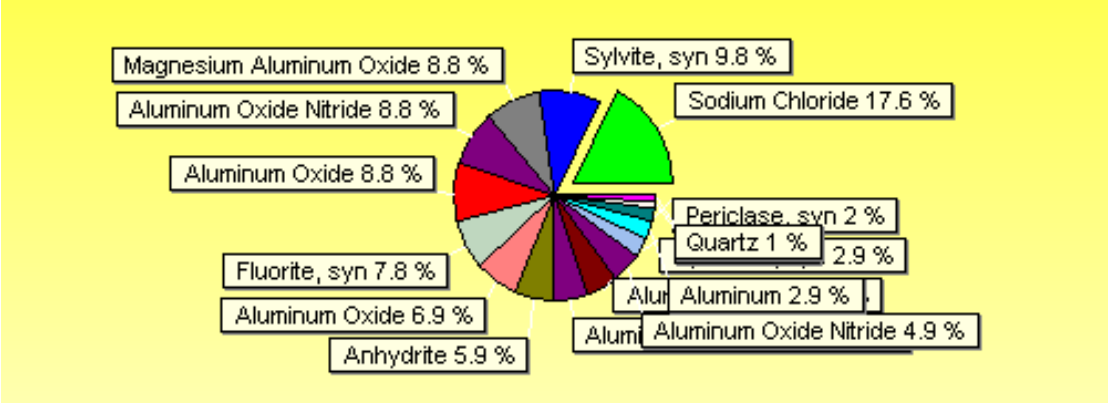
## 4.1: 2437-E-SC

### 4.1.1: Measurement Conditions

| | |
|---|---|
| Dataset Name | 2437-E-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2437-E-SC.sd |
| Sample Identification | 2437-E-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 8/1/2010 10:50:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 30 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 4.1.2: Main Graphics, Analyze View




**4.1.3: Pattern List of 2437-E-SC**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 52 | Aluminum Oxide | Al2 O3 | 9 | 19 | 0 |
| 00-004-0587 | 34 | Sylvite, syn | K Cl | 10 | 9 | 0 |
| 01-077-2064 | 39 | Sodium Chloride | Na Cl | 18 | 9 | 0 |
| 01-074-1132 | 39 | Magnesium Aluminum Oxide | Mg Al2 O4 | 9 | 13 | 0 |
| 01-075-1620 | 32 | Aluminum Nitride | Al N | 5 | 8 | 0 |
| 01-080-2171 | 12 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 9 | 11 | 0 |
| 01-080-2172 | 12 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 11 | 0 |
| 01-080-2173 | 14 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 10 | 0 |
| 01-085-1327 | 34 | Aluminum | Al | 3 | 5 | 0 |
| 00-043-1022 | 35 | Periclase, syn | Mg O | 2 | 7 | 0 |
| 00-004-0864 | 16 | Fluorite, syn | Ca F2 | 8 | 5 | 0 |
| 00-022-1235 | 25 | Elpasolite, syn | K2 Na Al F6 | 3 | 14 | 0 |
| 01-086-2270 | 23 | Anhydrite | Ca ( S O4 ) | 6 | 33 | 0 |
| 01-080-1385 | 13 | Aluminum Oxide | Al2.667 O4 | 7 | 11 | 0 |
| 01-086-2340 | 20 | Calcite | Ca ( C O3 ) | 1 | 14 | 0 |
| 01-085-0930 | 20 | Quartz | Si O2 | 1 | 18 | 0 |

**4.1.4: Peak List of 2437-E-SC**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.0318 | 9.82 | 0.0394 | 8.81757 | 0.04 | |
| 10.1612 | 41.32 | 0.2362 | 8.70550 | 0.16 | |
| 10.9525 | 77.56 | 0.6298 | 8.07833 | 0.30 | |
| 16.3556 | 34.02 | 0.2362 | 5.41975 | 0.13 | |
| 18.9770 | 616.76 | 0.1771 | 4.67661 | 2.41 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.8380 | 237.92 | 0.1378 | 4.26296 | 0.93 | 01-085-0930 |
| 21.9560 | 132.00 | 0.6298 | 4.04836 | 0.52 | 00-022-1235 |
| 23.0907 | 154.82 | 0.2362 | 3.85192 | 0.60 | 01-086-2270; 01-086-2340 |
| 24.4887 | 186.25 | 0.1574 | 3.63510 | 0.73 | |
| 25.5399 | 1054.86 | 0.1082 | 3.48782 | 4.12 | 01-075-1862; 01-086-2270 |
| 26.6231 | 1162.05 | 0.1082 | 3.34832 | 4.54 | 01-085-0930 |
| 27.3109 | 2322.63 | 0.1771 | 3.26554 | 9.07 | 01-077-2064 |
| 28.3037 | 10298.15 | 0.2263 | 3.15322 | 40.22 | 00-004-0587; 00-004-0864 |
| 29.4108 | 654.94 | 0.1574 | 3.03699 | 2.56 | 01-086-2340 |
| 31.6833 | 25604.78 | 0.1968 | 2.82416 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 33.2117 | 1590.32 | 0.0886 | 2.69760 | 6.21 | 01-075-1620 |
| 35.1041 | 1927.60 | 0.0720 | 2.55428 | 7.53 | 01-075-1862 |
| 35.1712 | 2126.79 | 0.0787 | 2.55167 | 8.31 | 01-075-1862 |
| 35.9923 | 1341.19 | 0.1771 | 2.49532 | 5.24 | 01-075-1620; 01-086-2340 |
| 36.8398 | 3370.86 | 0.0984 | 2.43984 | 13.16 | 01-074-1132; 00-043-1022 |
| 37.7502 | 1670.03 | 0.1680 | 2.38109 | 6.52 | 01-075-1862; 01-075-1620 |
| 37.9015 | 1610.50 | 0.1378 | 2.37390 | 6.29 | 01-075-1862; 01-075-1620 |
| 38.4849 | 1976.15 | 0.0886 | 2.33925 | 7.72 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 39.4859 | 484.77 | 0.1181 | 2.28222 | 1.89 | 01-086-2340; 01-085-0930 |
| 40.4900 | 6334.31 | 0.2066 | 2.22791 | 24.74 | 00-004-0587; 01-085-0930 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 42.9106 | 2046.33 | 0.0984 | 2.10768 | 7.99 | 00-043-1022 |
| 43.3508 | 2777.44 | 0.1279 | 2.08729 | 10.85 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 44.7959 | 3386.45 | 0.2362 | 2.02326 | 13.23 | 01-074-1132; 01-085-1327 |
| 45.4155 | 15822.34 | 0.1968 | 1.99709 | 61.79 | 01-077-2064; 01-080-2171; 01-086-2270 |
| 46.9861 | 678.58 | 0.1181 | 1.93393 | 2.65 | 00-004-0864; 01-086-2270; 01-086-2340 |
| 47.3652 | 565.44 | 0.1968 | 1.91933 | 2.21 | 01-086-2270 |
| 50.1310 | 2156.88 | 0.0886 | 1.81973 | 8.42 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.5158 | 1131.31 | 0.1082 | 1.74257 | 4.42 | 01-075-1862 |
| 53.8351 | 716.61 | 0.1181 | 1.70295 | 2.80 | 01-077-2064 |
| 54.9371 | 285.78 | 0.2362 | 1.67136 | 1.12 | 01-085-0930 |
| 55.6850 | 619.24 | 0.2362 | 1.65068 | 2.42 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4437 | 4794.50 | 0.1181 | 1.63028 | 18.73 | 01-077-2064 |
| 57.4859 | 2541.21 | 0.1440 | 1.60185 | 9.92 | 01-075-1862; 01-086-2340 |
| 57.6648 | 1471.31 | 0.0960 | 1.60127 | 5.75 | |
| 58.5688 | 1128.72 | 0.3360 | 1.57479 | 4.41 | 00-004-0587 |
| 59.3647 | 2267.61 | 0.2160 | 1.55556 | 8.86 | 01-074-1132; 01-075-1620 |
| 61.2844 | 595.85 | 0.1920 | 1.51135 | 2.33 | 01-075-1862; 01-086-2270 |
| 62.3040 | 1154.72 | 0.1920 | 1.48905 | 4.51 | 00-043-1022; 01-086-2270 |
| 65.2679 | 3525.04 | 0.1680 | 1.42841 | 13.77 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 66.2187 | 4146.39 | 0.0960 | 1.41019 | 16.19 | 00-004-0587; 01-077-2064; 01-075-1620 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 66.5178 | 3018.82 | 0.0960 | 1.40806 | 11.79 | |
| 68.1776 | 1445.79 | 0.1320 | 1.37436 | 5.65 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 71.4170 | 604.40 | 0.2400 | 1.31977 | 2.36 | 01-075-1620; 01-080-2173; 01-086-2270 |
| 73.0332 | 546.22 | 0.1440 | 1.29450 | 2.13 | 01-077-2064; 01-086-2270 |
| 73.6077 | 1240.28 | 0.3840 | 1.28581 | 4.84 | 00-004-0587; 00-022-1235; 01-086-2340; 01-085-0930 |
| 75.2518 | 4716.62 | 0.1320 | 1.26174 | 18.42 | 01-077-2064 |
| 75.4977 | 2488.47 | 0.1200 | 1.26137 | 9.72 | |
| 76.8688 | 735.69 | 0.1200 | 1.23918 | 2.87 | 01-075-1862; 01-086-2270 |
| 77.2708 | 668.20 | 0.3360 | 1.23374 | 2.61 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.2542 | 729.98 | 0.1200 | 1.22068 | 2.85 | 01-074-1132; 01-085-1327 |
| 78.6060 | 546.37 | 0.2400 | 1.21609 | 2.13 | 01-074-1132; 00-043-1022; 01-086-2270; 01-086-2340 |
| 80.6712 | 222.99 | 0.1920 | 1.19009 | 0.87 | 01-075-1862 |
| 82.6334 | 420.55 | 0.1920 | 1.16673 | 1.64 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.9763 | 2829.54 | 0.2040 | 1.15146 | 11.05 | 01-077-2064; 01-086-2340; 01-085-0930 |
| 84.2243 | 1619.23 | 0.0960 | 1.15155 | 6.32 | |
| 86.4287 | 132.28 | 0.3840 | 1.12497 | 0.52 | 01-075-1862 |
| 87.5641 | 281.31 | 0.3840 | 1.11328 | 1.10 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 88.9726 | 170.65 | 0.1920 | 1.09927 | 0.67 | 01-075-1862 |
| 90.3570 | 202.48 | 0.1920 | 1.08599 | 0.79 | 00-022-1235 |
| 91.1930 | 294.13 | 0.1320 | 1.07820 | 1.15 | |

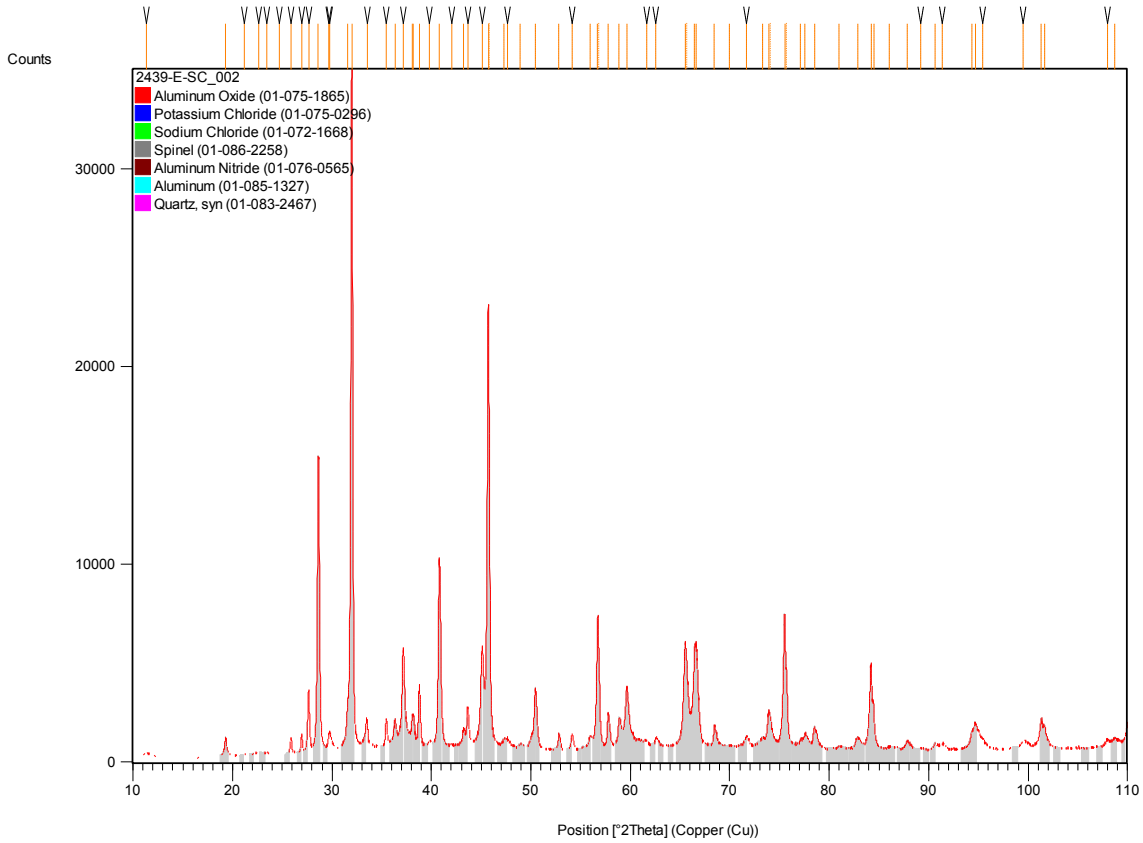
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 94.0951 | 661.18 | 0.1680 | 1.05244 | 2.58 | 00-043-1022; 00-004-0864 |
| 94.4141 | 873.67 | 0.1920 | 1.04972 | 3.41 | 00-004-0587; 00-004-0864 |
| 95.2360 | 654.09 | 0.1200 | 1.04282 | 2.55 | |
| 98.3857 | 107.36 | 0.2880 | 1.01768 | 0.42 | |
| 99.3261 | 261.23 | 0.6720 | 1.01056 | 1.02 | |
| 101.1123 | 1201.07 | 0.2400 | 0.99748 | 4.69 | |
| 101.4025 | 862.12 | 0.1680 | 0.99788 | 3.37 | |
| 107.7911 | 160.43 | 0.1440 | 0.95341 | 0.63 | |
| 109.8167 | 961.47 | 0.0720 | 0.94142 | 3.76 | 00-043-1022 |

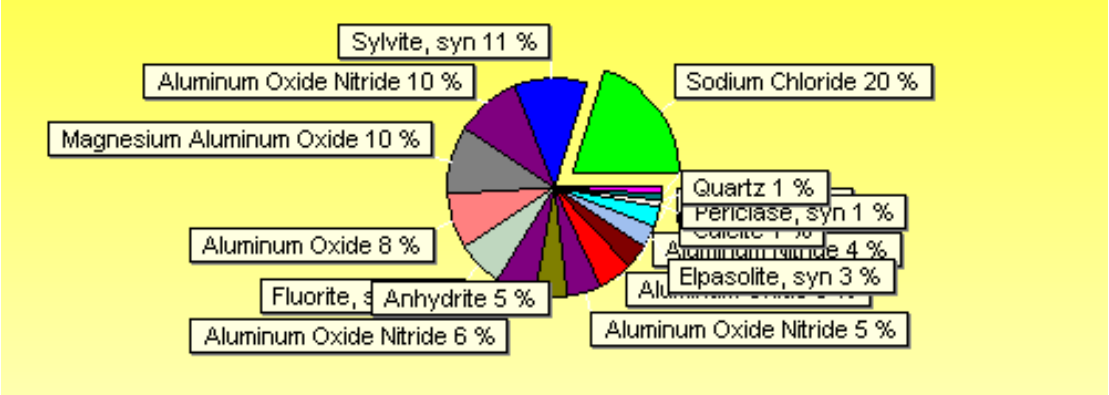
## 4.2: 2439-E-SC

### 4.2.1: Measurement Conditions of 2439-E-SC

| | |
|---|---|
| Dataset Name | 2439-E-SC_002 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2439-E-SC_002.udf |
| Sample Identification | 2439-E-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 4.2.2: Main Graphics, Analyze View of 2439-E


Counts
2439-E-SC_002
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Quartz, syn (01-083-2467)
30000
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Sylvite, syn 11 %
Aluminum Oxide Nitride 10 %
Sodium Chloride 20 %
Magnesium Aluminum Oxide 10 %
Quartz 1 %
Periclase, syn 1 %
Aluminum Oxide 8 %
Elpasolite, syn 3 %
Anhydrite 5 %
Aluminum Oxide Nitride 6 %
Aluminum Oxide Nitride 5 %

**4.2.3: Pattern List of 2439-E**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 52 | Aluminum Oxide | Al2 O3 | 5 | 19 | 0 |
| 00-004-0587 | 46 | Sylvite, syn | K Cl | 11 | 10 | 0 |
| 01-077-2064 | 48 | Sodium Chloride | Na Cl | 20 | 9 | 0 |
| 01-074-1132 | 52 | Magnesium Aluminum Oxide | Mg Al2 O4 | 10 | 15 | 0 |
| 01-075-1620 | 38 | Aluminum Nitride | Al N | 4 | 9 | 0 |
| 01-080-2171 | 14 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 10 | 12 | 0 |
| 01-080-2172 | 15 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 16 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 12 | 0 |
| 01-085-1327 | 40 | Aluminum | Al | 3 | 5 | 0 |
| 00-043-1022 | 31 | Periclase, syn | Mg O | 1 | 6 | 0 |
| 00-004-0864 | 18 | Fluorite, syn | Ca F2 | 7 | 6 | 0 |
| 00-022-1235 | 27 | Elpasolite, syn | K2 Na Al F6 | 3 | 15 | 0 |
| 01-086-2270 | 20 | Anhydrite | Ca ( S O4 ) | 5 | 35 | 0 |
| 01-080-1385 | 15 | Aluminum Oxide | Al2.667 O4 | 8 | 12 | 0 |
| 01-086-2340 | 23 | Calcite | Ca ( C O3 ) | 1 | 15 | 0 |
| 01-085-0930 | 18 | Quartz | Si O2 | 1 | 18 | 0 |

**4.2.4: Peak List of 2439-E**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 11.3009 | 197.85 | 0.2755 | 7.83002 | 0.58 | |
| 19.3118 | 980.34 | 0.1378 | 4.59627 | 2.86 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 21.1856 | 176.61 | 0.1181 | 4.19381 | 0.51 | |
| 22.5859 | 228.51 | 0.2362 | 3.93685 | 0.67 | |
| 23.4176 | 218.79 | 0.1968 | 3.79889 | 0.64 | |
| 24.7397 | 162.77 | 0.2362 | 3.59879 | 0.47 | |
| 25.8924 | 887.58 | 0.1378 | 3.44112 | 2.59 | |
| 26.9383 | 1087.54 | 0.1378 | 3.30986 | 3.17 | |
| 27.6698 | 3277.24 | 0.1378 | 3.22399 | 9.55 | |
| 28.6289 | 15226.77 | 0.1476 | 3.11813 | 44.38 | 01-086-2270 |
| 29.6613 | 1067.13 | 0.0960 | 3.00941 | 3.11 | |
| 29.7636 | 1179.17 | 0.1181 | 3.00179 | 3.44 | |
| 31.5592 | 2618.79 | 0.0787 | 2.83498 | 7.63 | 01-077-2064 |
| 31.9782 | 34307.43 | 0.1673 | 2.79878 | 100.00 | 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 33.5187 | 1816.80 | 0.1968 | 2.67359 | 5.30 | |
| 35.4407 | 1836.06 | 0.0886 | 2.53288 | 5.35 | |
| 36.3405 | 1821.14 | 0.1574 | 2.47221 | 5.31 | 01-086-2270 |
| 37.1794 | 5434.55 | 0.0590 | 2.41833 | 15.84 | |
| 38.0545 | 1843.79 | 0.0960 | 2.36275 | 5.37 | 01-075-1620 |
| 38.2011 | 1983.77 | 0.1378 | 2.35597 | 5.78 | 00-022-1235 |
| 38.8084 | 3475.57 | 0.0984 | 2.32049 | 10.13 | 01-086-2270 |
| 39.7562 | 701.17 | 0.2362 | 2.26733 | 2.04 | |
| 40.7717 | 9357.56 | 0.1082 | 2.21317 | 27.28 | 01-086-2270 |
| 42.0489 | 515.68 | 0.3149 | 2.14886 | 1.50 | |
| 43.2399 | 1288.18 | 0.1968 | 2.09239 | 3.75 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 43.6370 | 2353.04 | 0.1378 | 2.07427 | 6.86 | |
| 45.1144 | 5437.69 | 0.2558 | 2.00971 | 15.85 | |
| 45.7058 | 22632.19 | 0.1800 | 1.98343 | 65.97 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0930 |
| 45.8585 | 11620.57 | 0.0960 | 1.98210 | 33.87 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 47.2822 | 775.24 | 0.1920 | 1.92092 | 2.26 | 01-086-2270; 01-086-2340 |
| 47.6316 | 804.24 | 0.2880 | 1.90764 | 2.34 | |
| 48.9087 | 473.62 | 0.3840 | 1.86077 | 1.38 | 01-074-1132; 00-022-1235 |
| 50.4424 | 3211.73 | 0.2160 | 1.80773 | 9.36 | 01-085-0930 |
| 52.8169 | 982.45 | 0.1440 | 1.73191 | 2.86 | 01-086-2270 |
| 54.1304 | 984.36 | 0.2160 | 1.69295 | 2.87 | |
| 55.9550 | 858.92 | 0.2160 | 1.64199 | 2.50 | 00-004-0864 |
| 56.6904 | 6536.55 | 0.0840 | 1.62242 | 19.05 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-086-2340 |
| 56.8832 | 4000.24 | 0.0960 | 1.62140 | 11.66 | |
| 57.7574 | 1912.57 | 0.0840 | 1.59496 | 5.57 | 01-086-2270 |
| 58.8821 | 1770.73 | 0.1440 | 1.56715 | 5.16 | 00-022-1235; 01-086-2270 |
| 59.6329 | 3295.55 | 0.2160 | 1.54920 | 9.61 | 01-075-1862 |
| 61.6156 | 603.52 | 0.3840 | 1.50402 | 1.76 | |
| 62.5616 | 727.95 | 0.2400 | 1.48353 | 2.12 | |
| 65.5012 | 5426.07 | 0.2160 | 1.42389 | 15.82 | 01-086-2270 |
| 65.7179 | 4140.41 | 0.1440 | 1.42324 | 12.07 | |
| 66.4604 | 5270.22 | 0.1200 | 1.40564 | 15.36 | 01-075-1862; 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 66.6250 | 5441.04 | 0.1440 | 1.40257 | 15.86 | 01-075-1862; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 68.4518 | 1291.72 | 0.1920 | 1.36952 | 3.77 | 01-074-1132; 00-004-0864; 00-022-1235; 01-085-0930 |
| 69.9929 | 316.23 | 0.2880 | 1.34309 | 0.92 | 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385; 01-086-2340 |
| 71.6618 | 746.77 | 0.2400 | 1.31586 | 2.18 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 73.2794 | 699.67 | 0.1920 | 1.29076 | 2.04 | 01-085-0930 |
| 73.9159 | 2023.95 | 0.1920 | 1.28121 | 5.90 | 00-004-0587; 01-074-1132; 00-022-1235 |
| 75.5131 | 6945.03 | 0.1320 | 1.25803 | 20.24 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0930 |
| 75.7405 | 3740.07 | 0.0960 | 1.25793 | 10.90 | |
| 77.1153 | 615.77 | 0.1200 | 1.23584 | 1.79 | 01-075-1862; 01-086-2270 |
| 77.5716 | 836.49 | 0.1920 | 1.22970 | 2.44 | 01-074-1132; 01-086-2270; 01-085-0930 |
| 78.5302 | 1203.67 | 0.1680 | 1.21708 | 3.51 | 01-074-1132; 00-043-1022; 01-086-2270; 01-086-2340 |
| 80.9449 | 186.52 | 0.2880 | 1.18675 | 0.54 | 01-075-1620; 01-086-2340; 01-085-0930 |
| 82.8676 | 656.36 | 0.2400 | 1.16403 | 1.91 | 01-074-1132; 01-086-2270 |
| 84.2112 | 4297.52 | 0.0480 | 1.14884 | 12.53 | 01-075-1862; 01-080-2171; 01-080-2172; 01-086-2270; 01-080-1385 |
| 84.4539 | 2344.54 | 0.1440 | 1.14616 | 6.83 | 01-075-1862; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 86.0198 | 151.62 | 0.3840 | 1.12926 | 0.44 | 01-075-1862; 01-074-1132; 01-075-1620; 01-086-2270; 01-086-2340 |
| 87.8340 | 436.31 | 0.2400 | 1.11056 | 1.27 | 00-004-0587; 01-080-2172; 01-080-2173; 01-080-1385 |
| 90.6213 | 303.24 | 0.1440 | 1.08351 | 0.88 | 00-022-1235 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 91.3735 | 299.85 | 0.2400 | 1.07654 | 0.87 | |
| 94.3004 | 847.70 | 0.1440 | 1.05069 | 2.47 | 00-004-0587; 00-004-0864 |
| 94.6656 | 1308.46 | 0.2880 | 1.04759 | 3.81 | 00-004-0587 |
| 95.4417 | 587.83 | 0.1440 | 1.04112 | 1.71 | |
| 99.5062 | 372.11 | 0.2880 | 1.00921 | 1.08 | |
| 101.3281 | 1492.73 | 0.1680 | 0.99594 | 4.35 | 00-004-0587 |
| 101.6612 | 1125.89 | 0.2400 | 0.99357 | 3.28 | 00-004-0587; 00-022-1235 |
| 107.9463 | 417.63 | 0.2400 | 0.95247 | 1.22 | |
| 108.7062 | 470.33 | 0.2880 | 0.94792 | 1.37 | 00-004-0587 |

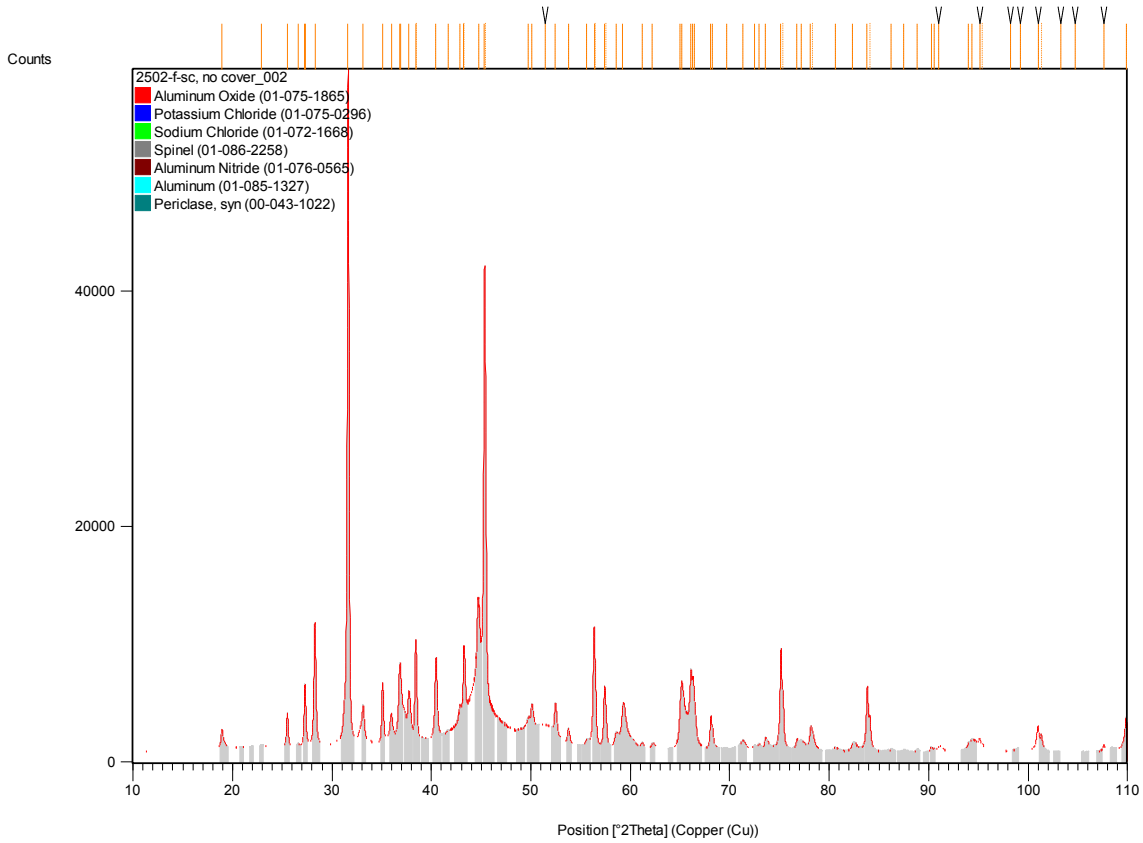
# FACILITY F

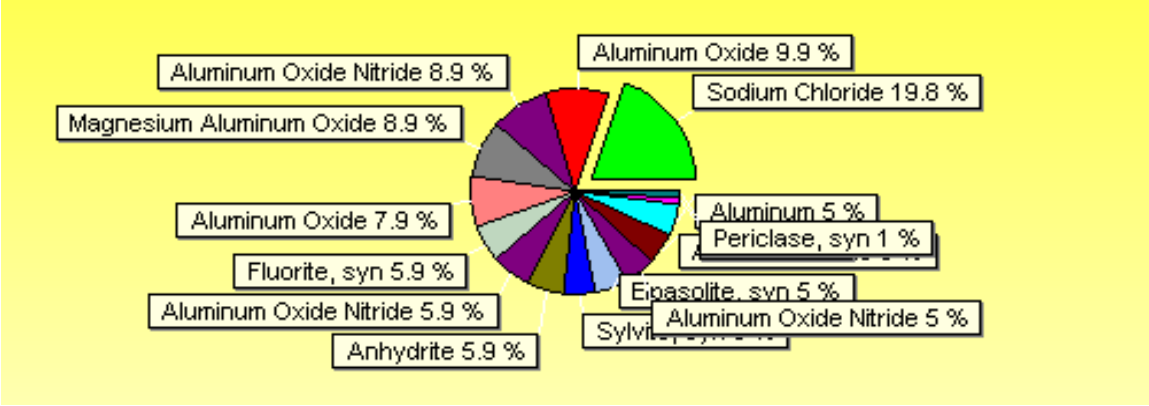
## 5.1: 2502-F-SC

### 5.1.1: Measurement Conditions of 2502-F

| | |
|---|---|
| Dataset Name | 2502-F-SC, no cover_002 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2502-f-sc, no cover_002.udf |
| Sample Identification | 2502-F-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by todd in project Ft. Devens. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 5.1.2: Main Graphics, Analyze View of 2502-F


Counts
2502-f-sc, no cover_002
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
40000
20000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Aluminum Oxide 9.9 %
Aluminum Oxide Nitride 8.9 %
Sodium Chloride 19.8 %
Magnesium Aluminum Oxide 8.9 %
Aluminum 5 %
Aluminum Oxide 7.9 %
Periclase, syn 1 %
Fluorite, syn 5.9 %
Aluminum Oxide Nitride 5.9 %
Aluminum Oxide Nitride 5 %
Anhydrite 5.9 %

**5.1.3: Pattern List of 2502-F**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 60 | Aluminum Oxide | Al2 O3 | 10 | 21 | 0 |
| 00-004-0587 | 47 | Sylvite, syn | K Cl | 5 | 10 | 0 |
| 01-077-2064 | 44 | Sodium Chloride | Na Cl | 20 | 9 | 0 |
| 01-074-1132 | 46 | Magnesium Aluminum Oxide | Mg Al2 O4 | 9 | 13 | 0 |
| 01-075-1620 | 35 | Aluminum Nitride | Al N | 5 | 9 | 0 |
| 01-080-2171 | 18 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 9 | 13 | 0 |
| 01-080-2172 | 20 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 21 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 12 | 0 |
| 01-085-1327 | 44 | Aluminum | Al | 5 | 5 | 0 |
| 00-043-1022 | 33 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 12 | Fluorite, syn | Ca F2 | 6 | 4 | 1 |
| 00-022-1235 | 26 | Elpasolite, syn | K2 Na Al F6 | 5 | 14 | 0 |
| 01-086-2270 | 19 | Anhydrite | Ca ( S O4 ) | 6 | 37 | 0 |
| 01-080-1385 | 20 | Aluminum Oxide | Al2.667 O4 | 8 | 13 | 0 |
| 01-085-0930 | 5 | Quartz | Si O2 | 1 | 17 | 0 |

**5.1.4: Peak List of 2502-F**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9355 | 1535.25 | 0.1771 | 4.68676 | 2.71 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 22.9354 | 120.75 | 0.4723 | 3.87766 | 0.21 | 01-086-2270 |
| 25.5104 | 2851.08 | 0.1181 | 3.49178 | 5.04 | 01-075-1862; 01-086-2270 |
| 26.5767 | 162.38 | 0.1181 | 3.35406 | 0.29 | 01-085-0930 |
| 27.2717 | 5159.69 | 0.0720 | 3.26743 | 9.11 | 01-077-2064 |
| 27.3281 | 4722.99 | 0.0492 | 3.26353 | 8.34 | 01-077-2064 |
| 28.3134 | 10004.57 | 0.1673 | 3.15216 | 17.67 | 00-004-0587; 00-004-0864 |
| 31.6155 | 56625.01 | 0.1870 | 2.83006 | 100.00 | 01-077-2064; 01-080-2171 |
| 33.1284 | 3174.59 | 0.0787 | 2.70419 | 5.61 | 01-075-1620 |
| 35.0772 | 5009.13 | 0.1080 | 2.55618 | 8.85 | 01-075-1862 |
| 35.1285 | 4464.54 | 0.0720 | 2.55890 | 7.88 | |
| 35.9703 | 2358.76 | 0.2160 | 2.49473 | 4.17 | 01-075-1620 |
| 36.7986 | 6468.07 | 0.1080 | 2.44045 | 11.42 | 01-074-1132; 00-043-1022 |
| 36.8811 | 6475.91 | 0.0840 | 2.43519 | 11.44 | 01-074-1132; 00-043-1022 |
| 37.6879 | 3925.06 | 0.1200 | 2.38489 | 6.93 | 01-075-1862; 01-080-2172; 01-080-2173; 01-080-1385 |
| 38.4071 | 8299.79 | 0.0960 | 2.34187 | 14.66 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 38.4887 | 7142.92 | 0.0600 | 2.34290 | 12.61 | |
| 40.4308 | 6438.96 | 0.1080 | 2.22919 | 11.37 | 00-004-0587; 01-085-0930 |
| 41.6723 | 210.67 | 0.3840 | 2.16561 | 0.37 | 01-075-1862 |
| 42.8563 | 2003.92 | 0.2160 | 2.10848 | 3.54 | 00-043-1022 |
| 43.2549 | 6993.65 | 0.0600 | 2.08996 | 12.35 | 01-075-1862; 01-086-2270 |
| 43.3146 | 6772.56 | 0.0600 | 2.09241 | 11.96 | |
| 44.7177 | 10702.21 | 0.2880 | 2.02494 | 18.90 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3368 | 38247.57 | 0.1560 | 1.99871 | 67.55 | 01-077-2064; 01-086-2270 |
| 45.4581 | 26512.33 | 0.1080 | 1.99862 | 46.82 | |
| 49.7408 | 1364.51 | 0.1920 | 1.83157 | 2.41 | 01-075-1620 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 50.1179 | 2619.87 | 0.0840 | 1.81867 | 4.63 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 51.4443 | 1130.33 | 0.8640 | 1.77485 | 2.00 | |
| 52.4605 | 3098.20 | 0.1920 | 1.74284 | 5.47 | 01-075-1862; 01-086-2270 |
| 53.7507 | 1256.15 | 0.1440 | 1.70401 | 2.22 | 01-077-2064 |
| 55.6203 | 511.15 | 0.2400 | 1.65108 | 0.90 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.3674 | 10106.26 | 0.1440 | 1.63095 | 17.85 | 01-077-2064 |
| 56.5129 | 5927.28 | 0.0960 | 1.63114 | 10.47 | |
| 57.4375 | 5105.47 | 0.1560 | 1.60309 | 9.02 | 01-075-1862; 01-085-0930 |
| 57.5874 | 2736.30 | 0.0960 | 1.60324 | 4.83 | |
| 58.5499 | 1214.15 | 0.2400 | 1.57525 | 2.14 | 00-004-0587 |
| 59.2490 | 3607.78 | 0.2640 | 1.55832 | 6.37 | 01-074-1132; 01-075-1620; 00-022-1235; 01-086-2270 |
| 62.2166 | 432.78 | 0.4320 | 1.49093 | 0.76 | 00-043-1022; 01-086-2270 |
| 65.0152 | 3955.67 | 0.1680 | 1.43335 | 6.99 | 01-085-1327; 00-022-1235 |
| 65.1495 | 5531.12 | 0.1440 | 1.43072 | 9.77 | 01-074-1132; 01-085-1327 |
| 66.0708 | 6629.24 | 0.0840 | 1.41299 | 11.71 | 01-077-2064; 01-075-1620 |
| 66.2878 | 6081.31 | 0.0960 | 1.40889 | 10.74 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-1385 |
| 66.4158 | 4969.95 | 0.1440 | 1.40648 | 8.78 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| | | | | | 01-080-2173; 01-080-1385 |
| 68.0817 | 2778.78 | 0.0600 | 1.37606 | 4.91 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.2816 | 1799.93 | 0.1200 | 1.37252 | 3.18 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 69.6490 | 175.95 | 0.2880 | 1.34888 | 0.31 | 01-074-1132; 01-075-1620 |
| 71.3457 | 852.85 | 0.4320 | 1.32091 | 1.51 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270 |
| 72.5028 | 392.15 | 0.1920 | 1.30266 | 0.69 | 01-075-1620 |
| 72.9214 | 622.60 | 0.1200 | 1.29621 | 1.10 | 01-077-2064; 01-086-2270 |
| 73.6102 | 1111.15 | 0.2160 | 1.28577 | 1.96 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 75.1289 | 8559.88 | 0.1560 | 1.26350 | 15.12 | 01-077-2064 |
| 75.3604 | 5136.95 | 0.0600 | 1.26333 | 9.07 | |
| 76.7686 | 1026.37 | 0.0960 | 1.24055 | 1.81 | 01-075-1862 |
| 77.2219 | 854.53 | 0.3840 | 1.23440 | 1.51 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.1288 | 2005.78 | 0.1920 | 1.22232 | 3.54 | 01-085-1327; 00-022-1235 |
| 78.3978 | 1378.01 | 0.1440 | 1.22183 | 2.43 | |
| 80.5924 | 306.32 | 0.1920 | 1.19105 | 0.54 | 01-075-1862 |
| 82.3223 | 673.90 | 0.1440 | 1.17035 | 1.19 | 01-085-1327; 00-022-1235 |
| 83.8206 | 5403.51 | 0.1560 | 1.15320 | 9.54 | 01-077-2064; 01-085-0930 |
| 84.1026 | 2912.46 | 0.1440 | 1.15291 | 5.14 | |
| 86.2473 | 224.65 | 0.1920 | 1.12687 | 0.40 | 01-075-1862 |
| 87.5224 | 176.39 | 0.2880 | 1.11371 | 0.31 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |

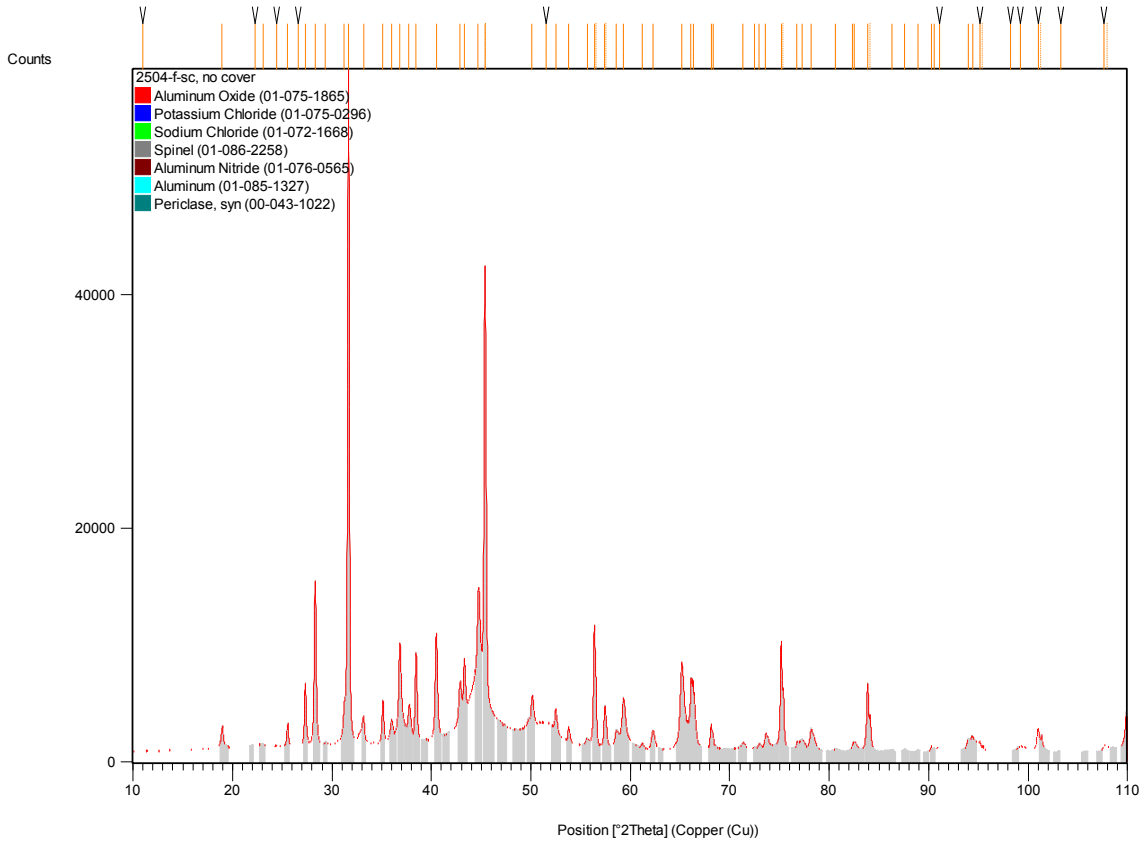
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 88.8694 | 270.30 | 0.1440 | 1.10028 | 0.48 | 01-075-1862; 01-086-2270 |
| 90.2402 | 434.74 | 0.1200 | 1.08709 | 0.77 | 00-022-1235 |
| 90.5555 | 320.04 | 0.1440 | 1.08412 | 0.57 | 00-022-1235 |
| 91.0248 | 531.21 | 0.1680 | 1.07975 | 0.94 | |
| 93.9934 | 766.82 | 0.1440 | 1.05331 | 1.35 | 00-043-1022; 00-004-0864 |
| 94.3802 | 1108.72 | 0.1920 | 1.05001 | 1.96 | 00-004-0587; 00-004-0864 |
| 95.1179 | 1142.63 | 0.1680 | 1.04381 | 2.02 | |
| 95.4192 | 675.46 | 0.1440 | 1.04389 | 1.19 | |
| 98.2164 | 203.25 | 0.2880 | 1.01899 | 0.36 | |
| 99.2413 | 444.14 | 0.4800 | 1.01119 | 0.78 | |
| 100.9998 | 2177.31 | 0.0960 | 0.99828 | 3.85 | |
| 101.3473 | 1379.23 | 0.1680 | 0.99827 | 2.44 | |
| 103.2726 | 59.51 | 0.5760 | 0.98242 | 0.11 | |
| 104.7221 | 48.89 | 0.7680 | 0.97275 | 0.09 | |
| 107.5886 | 549.22 | 0.1680 | 0.95464 | 0.97 | |
| 109.9198 | 2588.25 | 0.1440 | 0.94082 | 4.57 | 00-043-1022 |

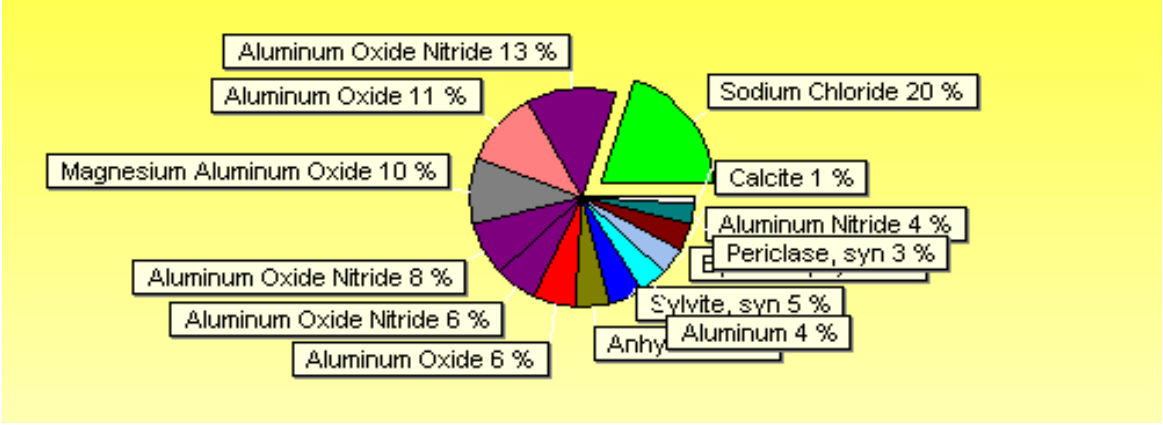
## 5.2: 2504-F-SC

### 5.2.1: Measurement Conditions of 2504-F

| | |
|---|---|
| Dataset Name | 2504-F-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2504-f-sc, no cover.udf |
| Sample Identification | 2504-F-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by todd in project Ft. Devens. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 5.2.2: Main Graphics, Analyze View of 2504-F


Counts
2504-f-sc, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
40000
20000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Aluminum Oxide Nitride 13 %
Aluminum Oxide 11 %
Sodium Chloride 20 %
Magnesium Aluminum Oxide 10 %
Calcite 1 %
Aluminum Nitride 4 %
Periclase, syn 3 %
Aluminum Oxide Nitride 8 %
Sylvite, syn 5 %
Aluminum Oxide Nitride 6 %
Aluminum 4 %
Aluminum Oxide 6 %

**5.2.3: Pattern List of 2504-F**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 53 | Aluminum Oxide | Al2 O3 | 6 | 21 | 0 |
| 00-004-0587 | 53 | Sylvite, syn | K Cl | 5 | 10 | 0 |
| 01-077-2064 | 59 | Sodium Chloride | Na Cl | 20 | 9 | 0 |
| 01-074-1132 | 53 | Magnesium Aluminum Oxide | Mg Al2 O4 | 10 | 14 | 0 |
| 01-075-1620 | 40 | Aluminum Nitride | Al N | 4 | 9 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 13 | 13 | 0 |
| 01-080-2172 | 17 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 8 | 13 | 0 |
| 01-080-2173 | 18 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 6 | 13 | 0 |
| 01-085-1327 | 42 | Aluminum | Al | 4 | 5 | 0 |
| 00-043-1022 | 35 | Periclase, syn | Mg O | 3 | 6 | 0 |
| 00-022-1235 | 26 | Elpasolite, syn | K2 Na Al F6 | 4 | 14 | 0 |
| 01-086-2270 | 21 | Anhydrite | Ca ( S O4 ) | 5 | 36 | 0 |
| 01-080-1385 | 17 | Aluminum Oxide | Al2.667 O4 | 11 | 13 | 0 |
| 01-086-2340 | 6 | Calcite | Ca ( C O3 ) | 1 | 15 | 0 |

**5.2.4: Peak List of 2504-F**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 11.0087 | 84.80 | 0.3149 | 8.03722 | 0.15 | |
| 18.9633 | 1759.03 | 0.1378 | 4.67995 | 3.04 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 22.2230 | 166.08 | 0.4723 | 4.00031 | 0.29 | |
| 23.0497 | 178.98 | 0.3149 | 3.85868 | 0.31 | 01-086-2270; 01-086-2340 |
| 24.4342 | 60.90 | 0.2362 | 3.64309 | 0.11 | |
| 25.5282 | 1881.51 | 0.0492 | 3.48939 | 3.25 | 01-075-1862; 01-086-2270 |
| 26.5840 | 193.11 | 0.1574 | 3.35316 | 0.33 | |
| 27.2932 | 4984.32 | 0.0984 | 3.26762 | 8.61 | 01-077-2064 |
| 28.2989 | 14010.89 | 0.1673 | 3.15374 | 24.21 | 00-004-0587 |
| 29.3337 | 238.44 | 0.1574 | 3.04480 | 0.41 | 01-086-2340 |
| 31.2467 | 3611.82 | 0.0984 | 2.86261 | 6.24 | 01-074-1132; 00-022-1235; 01-086-2270; 01-086-2340 |
| 31.6597 | 57871.07 | 0.1673 | 2.82621 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.1649 | 2354.74 | 0.0984 | 2.70130 | 4.07 | 01-075-1620 |
| 35.1161 | 3657.23 | 0.0886 | 2.55555 | 6.32 | 01-075-1862 |
| 36.0169 | 1949.76 | 0.1771 | 2.49367 | 3.37 | 01-075-1620; 01-086-2340 |
| 36.7808 | 7581.28 | 0.1082 | 2.44362 | 13.10 | 01-074-1132; 00-043-1022 |
| 37.7316 | 3031.16 | 0.0689 | 2.38420 | 5.24 | 01-075-1862; 01-075-1620; 01-080-2173 |
| 38.4293 | 7475.23 | 0.0984 | 2.34250 | 12.92 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.4803 | 8887.73 | 0.1560 | 2.22658 | 15.36 | 00-004-0587 |
| 40.5497 | 7755.84 | 0.0480 | 2.22845 | 13.40 | |
| 42.8434 | 4154.68 | 0.2400 | 2.10908 | 7.18 | 00-043-1022 |
| 43.3082 | 6202.02 | 0.1200 | 2.08752 | 10.72 | 01-075-1862; 01-086-2270; 01-086-2340 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 44.7095 | 11884.63 | 0.1440 | 2.02529 | 20.54 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3781 | 39493.24 | 0.1560 | 1.99699 | 68.24 | 01-077-2064; 01-086-2270 |
| 45.5036 | 24125.01 | 0.0600 | 1.99672 | 41.69 | |
| 50.0975 | 3044.90 | 0.3120 | 1.81936 | 5.26 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 51.5632 | 1221.75 | 0.7680 | 1.77104 | 2.11 | |
| 52.4806 | 2618.20 | 0.0840 | 1.74222 | 4.52 | 01-075-1862 |
| 53.7697 | 1311.76 | 0.0840 | 1.70345 | 2.27 | 01-077-2064 |
| 55.7150 | 603.32 | 0.2400 | 1.64850 | 1.04 | 01-074-1132; 01-086-2270 |
| 56.3734 | 10064.22 | 0.1560 | 1.63079 | 17.39 | 01-077-2064 |
| 56.5608 | 5275.79 | 0.1080 | 1.62987 | 9.12 | |
| 57.4045 | 3327.34 | 0.1200 | 1.60393 | 5.75 | 01-075-1862; 01-086-2340 |
| 57.5881 | 2078.26 | 0.1200 | 1.60322 | 3.59 | |
| 58.5670 | 1380.17 | 0.2160 | 1.57483 | 2.38 | 00-004-0587 |
| 59.2914 | 4193.09 | 0.0840 | 1.55731 | 7.25 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 61.2070 | 303.37 | 0.3360 | 1.51308 | 0.52 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 62.2420 | 1494.74 | 0.2880 | 1.49038 | 2.58 | 00-043-1022; 01-086-2270 |
| 65.1643 | 7281.14 | 0.3360 | 1.43043 | 12.58 | 01-074-1132; 01-085-1327 |
| 66.1085 | 6079.93 | 0.0480 | 1.41227 | 10.51 | 01-077-2064; 01-075-1620 |
| 66.3017 | 5750.86 | 0.1440 | 1.40862 | 9.94 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-1385 |
| 68.1184 | 2129.03 | 0.0600 | 1.37541 | 3.68 | 01-075-1862; 00-022-1235 |
| 68.2985 | 1286.18 | 0.1200 | 1.37222 | 2.22 | 01-075-1862; 00-022-1235 |
| 71.3502 | 577.73 | 0.3360 | 1.32084 | 1.00 | 01-075-1620; 01-080-2172; |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| | | | | | 01-080-2173; 01-086-2270 |
| 72.4565 | 203.02 | 0.2880 | 1.30338 | 0.35 | 01-075-1620 |
| 72.9630 | 585.30 | 0.1200 | 1.29557 | 1.01 | 01-077-2064; 01-086-2270 |
| 73.5878 | 1320.51 | 0.1680 | 1.28611 | 2.28 | 00-004-0587; 00-022-1235; 01-086-2340 |
| 75.1858 | 9276.18 | 0.1560 | 1.26269 | 16.03 | 01-077-2064 |
| 75.4205 | 4561.90 | 0.0480 | 1.26247 | 7.88 | |
| 76.7676 | 767.11 | 0.1200 | 1.24057 | 1.33 | 01-075-1862; 01-086-2340 |
| 77.2620 | 985.16 | 0.2400 | 1.23386 | 1.70 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.2043 | 1886.81 | 0.1680 | 1.22133 | 3.26 | 01-074-1132; 01-085-1327 |
| 80.5759 | 203.29 | 0.1920 | 1.19125 | 0.35 | 01-075-1862; 01-086-2340 |
| 82.3443 | 626.92 | 0.1920 | 1.17009 | 1.08 | 01-085-1327; 00-022-1235 |
| 82.5496 | 823.11 | 0.2400 | 1.16770 | 1.42 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.8615 | 5902.52 | 0.1320 | 1.15274 | 10.20 | 01-077-2064; 01-086-2340 |
| 84.1407 | 2930.52 | 0.0720 | 1.15248 | 5.06 | |
| 86.3399 | 158.23 | 0.3840 | 1.12590 | 0.27 | 01-075-1862 |
| 87.5435 | 244.90 | 0.2400 | 1.11349 | 0.42 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 88.8867 | 172.01 | 0.1440 | 1.10011 | 0.30 | 01-075-1862; 01-086-2270 |
| 90.2808 | 480.75 | 0.1200 | 1.08671 | 0.83 | 00-022-1235 |
| 90.5661 | 365.77 | 0.1440 | 1.08403 | 0.63 | 00-022-1235 |
| 91.0760 | 511.92 | 0.3840 | 1.07928 | 0.88 | |
| 93.9937 | 1120.94 | 0.2400 | 1.05330 | 1.94 | 00-043-1022 |
| 94.3936 | 1316.89 | 0.2400 | 1.04989 | 2.28 | 00-004-0587 |
| 95.1181 | 917.22 | 0.1920 | 1.04380 | 1.58 | |
| 95.4464 | 501.05 | 0.1440 | 1.04367 | 0.87 | |
| 98.2478 | 191.79 | 0.2880 | 1.01874 | 0.33 | |
| 99.2320 | 601.64 | 0.1920 | 1.01126 | 1.04 | |
| 101.0090 | 2038.36 | 0.2160 | 0.99822 | 3.52 | |
| 101.3251 | 1462.45 | 0.1440 | 0.99843 | 2.53 | |

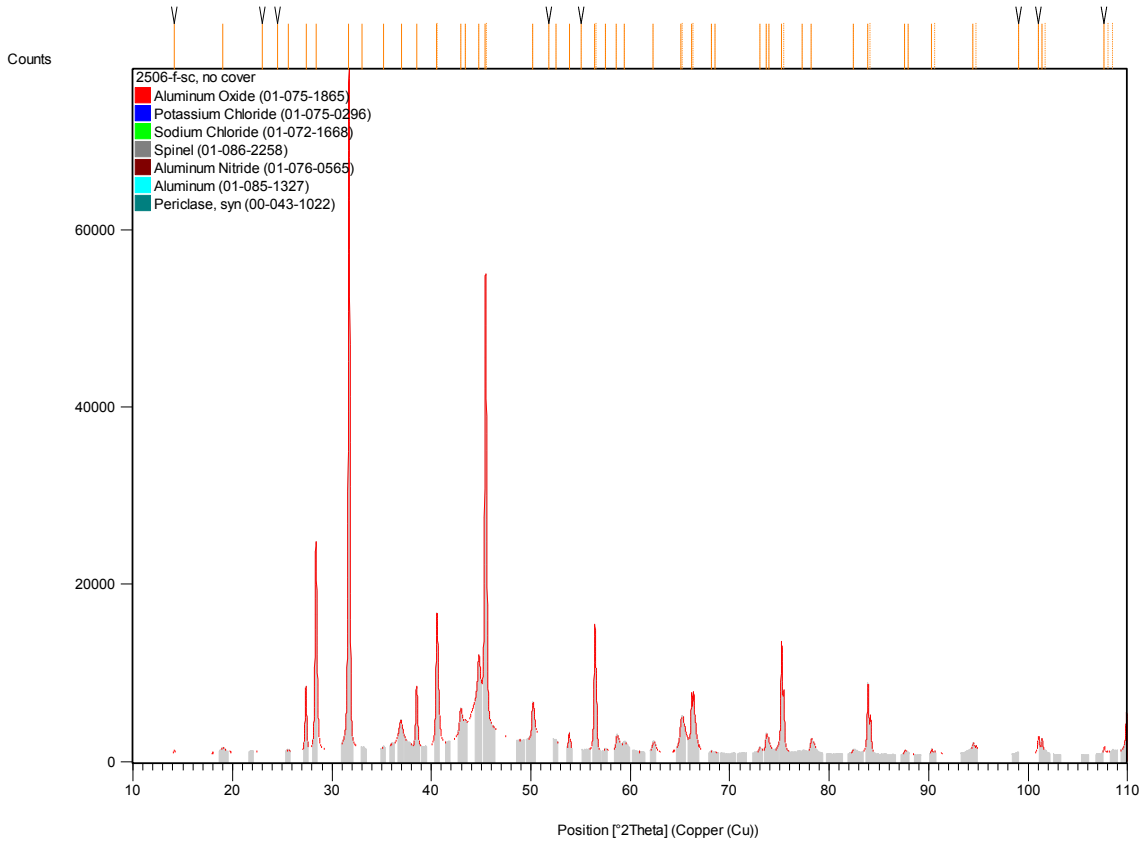
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 103.2523 | 45.47 | 0.5760 | 0.98255 | 0.08 | |
| 107.5948 | 482.64 | 0.1440 | 0.95460 | 0.83 | |
| 108.0210 | 246.65 | 0.1920 | 0.95438 | 0.43 | |

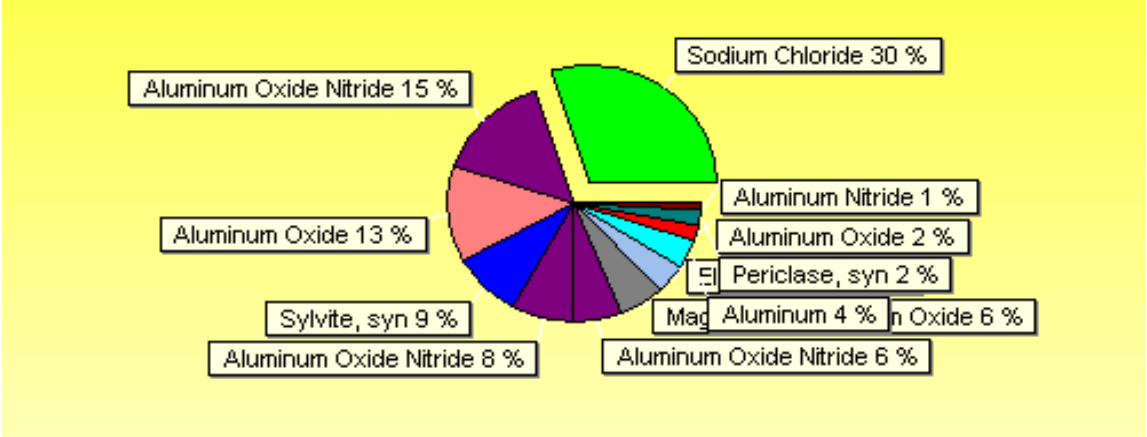
## 5.3: 2506-F-SC

### 5.3.1: Measurement Conditions of 2506-F

| | |
|---|---|
| Dataset Name | 2506-F-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2506-f-sc, no cover.udf |
| Sample Identification | 2506-F-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by todd in project Ft. Devens. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 5.3.2: Main Graphics, Analyze View of 2506-F


Counts
2506-f-sc, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
60000
40000
20000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Sodium Chloride 30 %
Aluminum Oxide Nitride 15 %
Aluminum Nitride 1 %
Aluminum Oxide 13 %
Aluminum Oxide 2 %
Periclase, syn 2 %
Sylvite, syn 9 %
Aluminum 4 %
Aluminum Oxide Nitride 8 %
Aluminum Oxide Nitride 6 %

**5.3.3: Pattern List of 2506-F**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 33 | Aluminum Oxide | Al2 O3 | 2 | 14 | 0 |
| 00-004-0587 | 57 | Sylvite, syn | K Cl | 9 | 10 | 0 |
| 01-077-2064 | 61 | Sodium Chloride | Na Cl | 30 | 9 | 0 |
| 01-074-1132 | 34 | Magnesium Aluminum Oxide | Mg Al2 O4 | 6 | 13 | 0 |
| 01-075-1620 | 16 | Aluminum Nitride | Al N | 1 | 6 | 0 |
| 01-080-2171 | 15 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 15 | 12 | 0 |
| 01-080-2172 | 17 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 8 | 11 | 0 |
| 01-080-2173 | 17 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 6 | 11 | 0 |
| 01-085-1327 | 49 | Aluminum | Al | 4 | 5 | 0 |
| 00-043-1022 | 40 | Periclase, syn | Mg O | 2 | 6 | 0 |
| 00-022-1235 | 28 | Elpasolite, syn | K2 Na Al F6 | 4 | 14 | 0 |
| 01-080-1385 | 17 | Aluminum Oxide | Al2.667 O4 | 13 | 12 | 0 |

**5.3.4: Peak List of 2506-F**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 14.1442 | 500.22 | 0.0492 | 6.26175 | 0.67 | |
| 19.0217 | 543.22 | 0.1968 | 4.66572 | 0.73 | 01-074-1132; 00-022-1235 |
| 22.9889 | 66.62 | 0.9446 | 3.86876 | 0.09 | |
| 24.4982 | 81.34 | 0.2362 | 3.63371 | 0.11 | |
| 25.6027 | 188.47 | 0.1574 | 3.47940 | 0.25 | 01-075-1862 |
| 27.3846 | 7238.35 | 0.0886 | 3.25691 | 9.71 | 01-077-2064 |
| 28.3651 | 22791.98 | 0.1771 | 3.14653 | 30.57 | 00-004-0587 |
| 31.6977 | 74557.24 | 0.1574 | 2.82291 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 32.9903 | 285.00 | 0.3149 | 2.71520 | 0.38 | 01-075-1620 |
| 35.1755 | 375.80 | 0.1378 | 2.55137 | 0.50 | 01-075-1862 |
| 36.9808 | 3047.98 | 0.2362 | 2.43086 | 4.09 | 01-074-1132; 00-043-1022 |
| 38.4878 | 6644.22 | 0.1771 | 2.33908 | 8.91 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.5512 | 14912.04 | 0.1440 | 2.22285 | 20.00 | 00-004-0587 |
| 40.6035 | 13626.94 | 0.0960 | 2.22563 | 18.28 | |
| 42.9210 | 3586.69 | 0.2400 | 2.10545 | 4.81 | 00-043-1022 |
| 43.3892 | 2386.69 | 0.1440 | 2.08381 | 3.20 | 01-075-1862 |
| 44.7702 | 9360.45 | 0.0960 | 2.02269 | 12.55 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.4214 | 51264.29 | 0.1800 | 1.99519 | 68.76 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 45.5552 | 31232.14 | 0.0840 | 1.99458 | 41.89 | |
| 50.1940 | 4680.78 | 0.0720 | 1.81609 | 6.28 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 51.7865 | 1177.28 | 0.3840 | 1.76393 | 1.58 | |
| 52.5136 | 808.87 | 0.2400 | 1.74120 | 1.08 | 01-075-1862 |
| 53.8456 | 1858.05 | 0.0840 | 1.70123 | 2.49 | 01-077-2064 |
| 55.0741 | 77.50 | 0.2880 | 1.66615 | 0.10 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 56.4383 | 14234.93 | 0.1560 | 1.62907 | 19.09 | 01-077-2064; 01-080-2171; 01-080-1385 |
| 56.6088 | 7065.04 | 0.1080 | 1.62861 | 9.48 | |
| 57.4512 | 289.16 | 0.3360 | 1.60273 | 0.39 | 01-075-1862 |
| 58.6108 | 1801.92 | 0.0960 | 1.57376 | 2.42 | 00-004-0587 |
| 59.4041 | 1135.33 | 0.4320 | 1.55462 | 1.52 | 01-074-1132; 01-075-1620 |
| 62.2881 | 1264.45 | 0.1200 | 1.48939 | 1.70 | 00-043-1022 |
| 65.0737 | 3499.82 | 0.2640 | 1.43220 | 4.69 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 65.3001 | 4092.17 | 0.1440 | 1.43133 | 5.49 | |
| 66.1467 | 6529.17 | 0.0840 | 1.41155 | 8.76 | 00-004-0587; 01-077-2064; 01-075-1620 |
| 66.3398 | 6981.57 | 0.0600 | 1.41141 | 9.36 | |
| 68.1870 | 319.64 | 0.1680 | 1.37420 | 0.43 | 01-075-1862; 00-022-1235 |
| 68.5406 | 118.39 | 0.7680 | 1.36797 | 0.16 | 01-074-1132 |
| 73.0032 | 751.95 | 0.1440 | 1.29496 | 1.01 | 01-077-2064 |
| 73.6719 | 2271.24 | 0.0840 | 1.28485 | 3.05 | 00-004-0587; 00-022-1235 |
| 73.9149 | 1664.24 | 0.1200 | 1.28122 | 2.23 | 01-075-1862; 00-004-0587; 01-074-1132; 00-022-1235 |
| 75.2080 | 12578.65 | 0.1560 | 1.26237 | 16.87 | 01-077-2064 |
| 75.4290 | 7181.86 | 0.1200 | 1.26235 | 9.63 | |
| 77.3035 | 358.48 | 0.3840 | 1.23330 | 0.48 | 01-075-1862; 01-074-1132 |
| 78.1993 | 1619.84 | 0.2160 | 1.22140 | 2.17 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 82.4005 | 536.91 | 0.2400 | 1.16944 | 0.72 | 01-074-1132; 01-085-1327; 00-022-1235 |

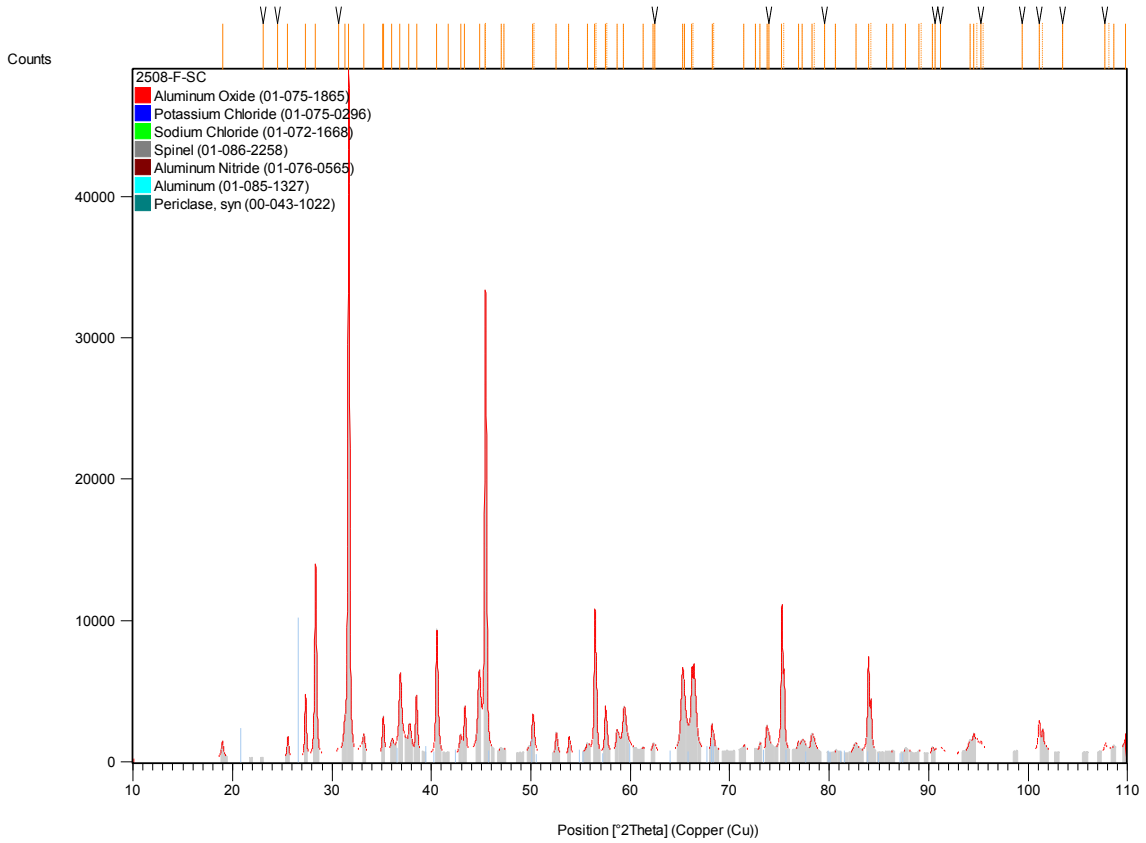
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 83.8950 | 8044.99 | 0.1680 | 1.15237 | 10.79 | 01-077-2064 |
| 84.1525 | 4384.14 | 0.1200 | 1.15235 | 5.88 | |
| 87.5990 | 474.16 | 0.1200 | 1.11293 | 0.64 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 87.9083 | 337.54 | 0.1440 | 1.10981 | 0.45 | 00-004-0587; 01-080-2172; 01-080-2173; 01-080-1385 |
| 90.3207 | 624.26 | 0.1440 | 1.08633 | 0.84 | 00-022-1235 |
| 90.6135 | 344.99 | 0.1440 | 1.08627 | 0.46 | |
| 94.4353 | 1155.34 | 0.1920 | 1.04954 | 1.55 | 00-004-0587 |
| 94.7861 | 814.48 | 0.1440 | 1.04918 | 1.09 | |
| 99.0772 | 304.56 | 0.7680 | 1.01243 | 0.41 | |
| 101.0395 | 2208.82 | 0.1560 | 0.99800 | 2.96 | |
| 101.3699 | 1742.00 | 0.1680 | 0.99564 | 2.34 | 00-004-0587 |
| 101.7534 | 519.96 | 0.1440 | 0.99539 | 0.70 | |
| 107.6610 | 684.04 | 0.0960 | 0.95420 | 0.92 | |
| 108.0382 | 231.46 | 0.1680 | 0.95428 | 0.31 | |
| 108.4854 | 223.21 | 0.2400 | 0.95159 | 0.30 | |

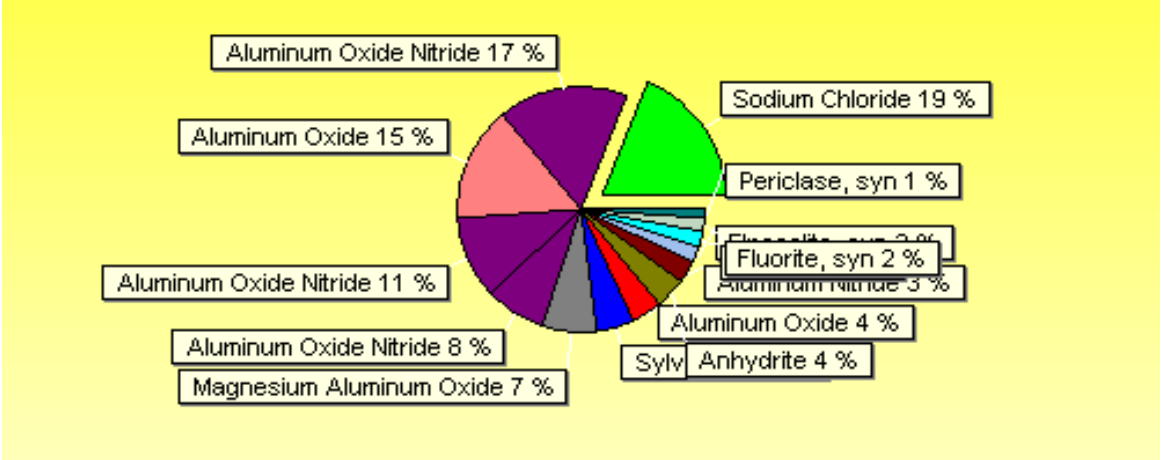
## 5.4: 2508-F-SC

### 5.4.1: Measurement Conditions of 2508-F

| | |
|---|---|
| Dataset Name | 2508-F-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2508-F-SC.udf |
| Sample Identification | 2508-F-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 5.4.2: Main Graphics, Analyze View of 2508-F


Counts
2508-F-SC
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
40000
30000
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Aluminum Oxide Nitride 17 %
Sodium Chloride 19 %
Aluminum Oxide 15 %
Periclase, syn 1 %
Fluorite, syn 2 %
Aluminum Oxide Nitride 11 %
Aluminum Oxide 4 %
Aluminum Oxide Nitride 8 %
Anhydrite 4 %
Magnesium Aluminum Oxide 7 %

**5.4.3: Pattern List of 2508-F**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 34 | Aluminum Oxide | Al2 O3 | 4 | 17 | 0 |
| 00-004-0587 | 41 | Sylvite, syn | K Cl | 5 | 10 | 0 |
| 01-077-2064 | 56 | Sodium Chloride | Na Cl | 19 | 9 | 0 |
| 01-074-1132 | 51 | Magnesium Aluminum Oxide | Mg Al2 O4 | 7 | 14 | 0 |
| 01-075-1620 | 31 | Aluminum Nitride | Al N | 3 | 9 | 0 |
| 01-080-2171 | 13 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 17 | 12 | 0 |
| 01-080-2172 | 12 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 11 | 12 | 0 |
| 01-080-2173 | 14 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 8 | 12 | 0 |
| 01-085-1327 | 33 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 29 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 26 | Fluorite, syn | Ca F2 | 2 | 6 | 0 |
| 00-022-1235 | 18 | Elpasolite, syn | K2 Na Al F6 | 2 | 14 | 0 |
| 01-086-2270 | 17 | Anhydrite | Ca ( S O4 ) | 4 | 34 | 0 |
| 01-080-1385 | 11 | Aluminum Oxide | Al2.667 O4 | 15 | 12 | 0 |

**5.4.4: Peak List of 2508-F**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9985 | 1202.02 | 0.1279 | 4.67136 | 2.54 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0884 | 33.59 | 0.2362 | 3.85230 | 0.07 | |
| 24.4847 | 113.71 | 0.1574 | 3.63569 | 0.24 | |
| 25.5509 | 1471.24 | 0.0590 | 3.48635 | 3.11 | 01-075-1862; 01-086-2270 |
| 27.3100 | 3991.90 | 0.1476 | 3.26564 | 8.44 | 01-077-2064 |
| 28.3345 | 13366.79 | 0.1574 | 3.14986 | 28.27 | 00-004-0587; 00-004-0864 |
| 30.6482 | 733.17 | 0.1574 | 2.91714 | 1.55 | |
| 31.2579 | 2585.51 | 0.0984 | 2.86162 | 5.47 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 31.6685 | 47285.34 | 0.1574 | 2.82545 | 100.00 | 01-077-2064; 01-080-2171 |
| 33.2106 | 1502.27 | 0.1968 | 2.69769 | 3.18 | 01-075-1620 |
| 35.1155 | 2654.03 | 0.0840 | 2.55347 | 5.61 | 01-075-1862 |
| 35.1850 | 2786.04 | 0.0689 | 2.55070 | 5.89 | 01-075-1862 |
| 36.0279 | 1248.52 | 0.1378 | 2.49294 | 2.64 | 01-075-1620 |
| 36.8197 | 5453.08 | 0.0886 | 2.44113 | 11.53 | 01-074-1132; 00-043-1022 |
| 37.7556 | 2270.19 | 0.0787 | 2.38274 | 4.80 | 01-075-1862 |
| 38.4920 | 4289.70 | 0.1673 | 2.33883 | 9.07 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.4909 | 7852.48 | 0.1476 | 2.22787 | 16.61 | 00-004-0587 |
| 41.7115 | 234.38 | 0.2362 | 2.16545 | 0.50 | 01-075-1862 |
| 42.9130 | 1459.60 | 0.1181 | 2.10757 | 3.09 | 00-043-1022 |
| 43.3323 | 3447.56 | 0.0886 | 2.08814 | 7.29 | 01-075-1862; 01-086-2270 |
| 44.8254 | 6033.44 | 0.0689 | 2.02200 | 12.76 | 01-074-1132; 01-085-1327 |
| 45.4157 | 32345.72 | 0.1560 | 1.99542 | 68.41 | 01-077-2064; 01-086-2270 |
| 45.5249 | 23375.21 | 0.1200 | 1.99584 | 49.43 | |
| 46.9812 | 489.76 | 0.1920 | 1.93252 | 1.04 | 00-004-0864 |
| 47.3226 | 431.10 | 0.1920 | 1.91937 | 0.91 | 01-086-2270 |
| 50.1909 | 2791.25 | 0.1200 | 1.81620 | 5.90 | 00-004-0587; 01-080-2173; 00-022-1235 |
| 50.3146 | 2210.94 | 0.0960 | 1.81652 | 4.68 | |
| 52.5343 | 1452.90 | 0.1200 | 1.74056 | 3.07 | 01-075-1862 |
| 53.8245 | 1122.15 | 0.0720 | 1.70185 | 2.37 | 01-077-2064 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 55.6627 | 701.66 | 0.1200 | 1.64992 | 1.48 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4314 | 9962.85 | 0.1440 | 1.62926 | 21.07 | 01-077-2064 |
| 56.5715 | 6619.78 | 0.1200 | 1.62959 | 14.00 | |
| 57.5076 | 3281.29 | 0.0960 | 1.60130 | 6.94 | 01-075-1862 |
| 57.6638 | 1902.93 | 0.0960 | 1.60130 | 4.02 | |
| 58.6427 | 1541.03 | 0.1440 | 1.57298 | 3.26 | 00-004-0587 |
| 59.3288 | 3144.83 | 0.0840 | 1.55641 | 6.65 | 01-074-1132; 01-075-1620 |
| 61.3008 | 401.03 | 0.1200 | 1.51099 | 0.85 | 01-075-1862 |
| 62.2715 | 582.96 | 0.1440 | 1.48974 | 1.23 | 00-043-1022; 01-086-2270 |
| 62.4791 | 553.18 | 0.1440 | 1.48529 | 1.17 | |
| 65.2432 | 5898.64 | 0.0720 | 1.42889 | 12.47 | 01-074-1132; 01-085-1327 |
| 65.4017 | 5029.96 | 0.1440 | 1.42581 | 10.64 | 01-074-1132; 01-086-2270 |
| 66.1936 | 6032.36 | 0.1200 | 1.41066 | 12.76 | 01-077-2064 |
| 66.3707 | 6208.49 | 0.0960 | 1.41082 | 13.13 | |
| 68.2043 | 1958.61 | 0.1680 | 1.37389 | 4.14 | 01-075-1862; 00-022-1235 |
| 68.3891 | 1221.52 | 0.1080 | 1.37403 | 2.58 | |
| 71.4445 | 434.09 | 0.2880 | 1.31933 | 0.92 | 01-075-1620; 01-086-2270 |
| 72.5389 | 203.58 | 0.2880 | 1.30210 | 0.43 | 01-075-1620 |
| 73.0499 | 640.74 | 0.1200 | 1.29424 | 1.36 | 01-077-2064 |
| 73.7148 | 1856.39 | 0.1080 | 1.28421 | 3.93 | 00-004-0587; 00-022-1235 |
| 73.9509 | 1206.83 | 0.1440 | 1.28069 | 2.55 | |
| 75.2366 | 10158.39 | 0.1560 | 1.26196 | 21.48 | 01-077-2064 |
| 75.4892 | 5401.76 | 0.1320 | 1.26149 | 11.42 | |
| 76.8825 | 775.80 | 0.1440 | 1.23900 | 1.64 | 01-075-1862 |
| 77.2933 | 893.07 | 0.2400 | 1.23343 | 1.89 | 01-075-1862; 01-074-1132 |
| 78.2753 | 1370.38 | 0.1920 | 1.22040 | 2.90 | 01-074-1132; 01-085-1327 |
| 78.4997 | 984.07 | 0.1440 | 1.22050 | 2.08 | |
| 79.5233 | 84.59 | 0.2880 | 1.20435 | 0.18 | |
| 80.6531 | 189.87 | 0.1440 | 1.19031 | 0.40 | 01-075-1862 |
| 82.6651 | 657.52 | 0.4800 | 1.16636 | 1.39 | 01-074-1132; 01-086-2270 |
| 83.9578 | 6799.55 | 0.1680 | 1.15166 | 14.38 | 01-077-2064 |
| 84.2225 | 3657.36 | 0.1080 | 1.15157 | 7.73 | |

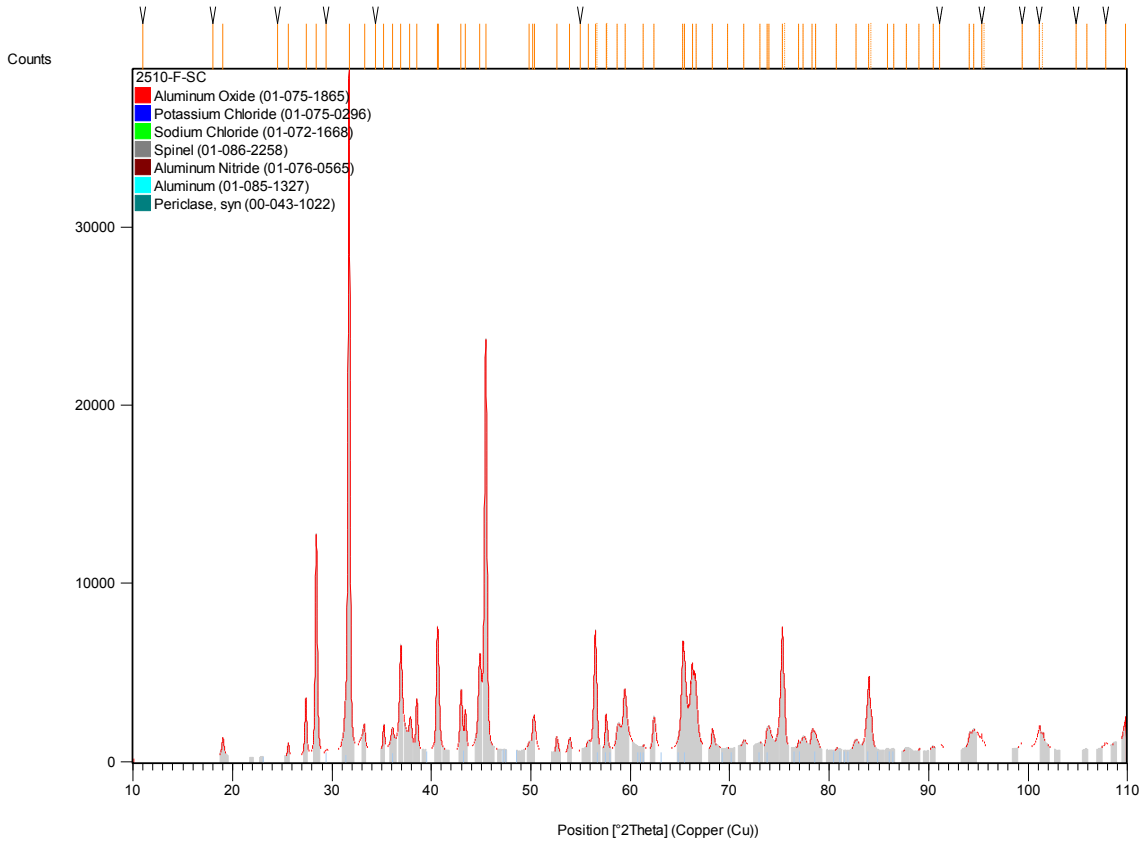
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 85.7945 | 119.60 | 0.3840 | 1.13165 | 0.25 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 86.4223 | 159.92 | 0.3840 | 1.12503 | 0.34 | 01-075-1862 |
| 87.6751 | 401.47 | 0.1440 | 1.11216 | 0.85 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 89.0177 | 158.41 | 0.1920 | 1.09883 | 0.34 | 01-075-1862 |
| 89.2933 | 113.60 | 0.1440 | 1.09887 | 0.24 | |
| 90.3667 | 474.07 | 0.1080 | 1.08590 | 1.00 | 00-022-1235 |
| 90.6586 | 354.95 | 0.1440 | 1.08316 | 0.75 | |
| 91.2014 | 441.67 | 0.2400 | 1.07812 | 0.93 | |
| 94.1543 | 931.81 | 0.2160 | 1.05193 | 1.97 | 00-043-1022; 00-004-0864 |
| 94.5411 | 1393.03 | 0.1440 | 1.04865 | 2.95 | 00-004-0587 |
| 94.8677 | 957.83 | 0.1440 | 1.04849 | 2.03 | |
| 95.2434 | 878.38 | 0.1440 | 1.04276 | 1.86 | |
| 95.5234 | 486.35 | 0.1680 | 1.04303 | 1.03 | |
| 99.3588 | 454.71 | 0.2400 | 1.01031 | 0.96 | |
| 101.1370 | 2302.10 | 0.1800 | 0.99730 | 4.87 | |
| 101.4579 | 1691.47 | 0.1200 | 0.99748 | 3.58 | |
| 103.4291 | 51.49 | 0.5760 | 0.98136 | 0.11 | |
| 107.7237 | 855.30 | 0.1680 | 0.95382 | 1.81 | |
| 108.1339 | 644.89 | 0.1440 | 0.95370 | 1.36 | |
| 108.6137 | 811.28 | 0.1920 | 0.94847 | 1.72 | 00-004-0587 |
| 109.8151 | 1666.63 | 0.0480 | 0.94143 | 3.52 | 00-043-1022 |

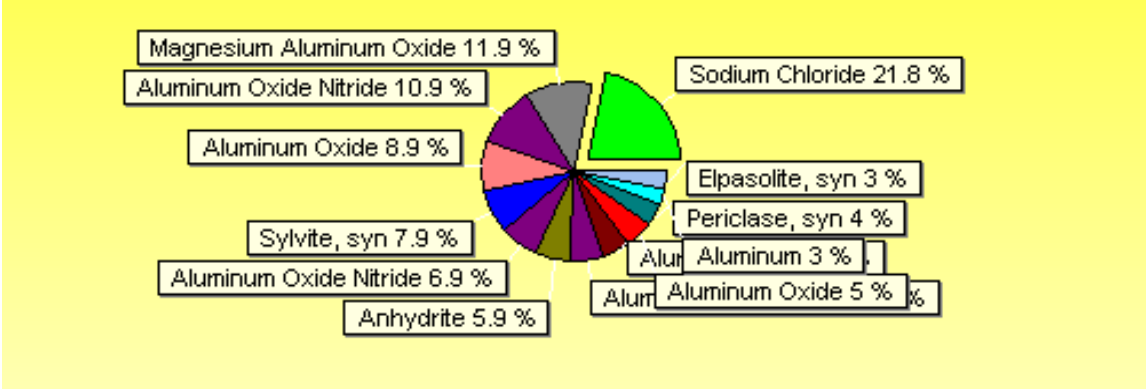
## 5.5: 2510-F-SC

### 5.5.1: Measurement Conditions of 2510-F

| | |
|---|---|
| Dataset Name | 2510-F-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2510-F-SC.udf |
| Sample Identification | 2510-F-SC NO COVER |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 5.5.2: Main Graphics, Analyze View of 2510-F


Counts
2510-F-SC
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
30000
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Magnesium Aluminum Oxide 11.9 %
Aluminum Oxide Nitride 10.9 %
Sodium Chloride 21.8 %
Aluminum Oxide 8.9 %
Elpasolite, syn 3 %
Periclase, syn 4 %
Sylvite, syn 7.9 %
Aluminum 3 %
Aluminum Oxide Nitride 6.9 %
Aluminum Oxide 5 %
Anhydrite 5.9 %

**5.5.3: Pattern List of 2510-F**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 49 | Aluminum Oxide | Al2 O3 | 5 | 19 | 0 |
| 00-004-0587 | 51 | Sylvite, syn | K Cl | 8 | 10 | 0 |
| 01-077-2064 | 49 | Sodium Chloride | Na Cl | 22 | 9 | 0 |
| 01-074-1132 | 49 | Magnesium Aluminum Oxide | Mg Al2 O4 | 12 | 15 | 0 |
| 01-075-1620 | 32 | Aluminum Nitride | Al N | 5 | 10 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 11 | 13 | 0 |
| 01-080-2172 | 19 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 7 | 13 | 0 |
| 01-080-2173 | 20 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 6 | 12 | 0 |
| 01-085-1327 | 38 | Aluminum | Al | 3 | 5 | 0 |
| 00-043-1022 | 37 | Periclase, syn | Mg O | 4 | 8 | 0 |
| 00-022-1235 | 24 | Elpasolite, syn | K2 Na Al F6 | 3 | 16 | 0 |
| 01-086-2270 | 14 | Anhydrite | Ca ( S O4 ) | 6 | 33 | 0 |
| 01-080-1385 | 19 | Aluminum Oxide | Al2.667 O4 | 9 | 13 | 0 |

**5.5.4: Peak List of 2510-F**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.9694 | 19.39 | 0.9446 | 8.06592 | 0.05 | |
| 18.0663 | 84.48 | 0.2362 | 4.91025 | 0.22 | |
| 19.0343 | 1147.14 | 0.0689 | 4.66266 | 2.98 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 24.5358 | 55.47 | 0.1574 | 3.62823 | 0.14 | |
| 25.5947 | 771.99 | 0.0787 | 3.48047 | 2.00 | 01-075-1862; 01-086-2270 |
| 27.3776 | 3264.60 | 0.1279 | 3.25774 | 8.47 | 01-077-2064 |
| 28.4092 | 12475.85 | 0.1673 | 3.14174 | 32.37 | 00-004-0587; 01-086-2270 |
| 29.4271 | 330.25 | 0.1574 | 3.03534 | 0.86 | |
| 31.7275 | 38538.18 | 0.2066 | 2.82032 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 33.2477 | 1670.97 | 0.1771 | 2.69476 | 4.34 | 01-075-1620 |
| 34.4150 | 300.20 | 0.2362 | 2.60598 | 0.78 | |
| 35.1561 | 1459.54 | 0.0960 | 2.55062 | 3.79 | 01-075-1862 |
| 35.2103 | 1671.17 | 0.0590 | 2.54892 | 4.34 | 01-075-1862 |
| 36.0501 | 1397.87 | 0.1771 | 2.49145 | 3.63 | 01-075-1620 |
| 36.9149 | 6066.86 | 0.1181 | 2.43505 | 15.74 | 01-074-1132; 00-043-1022 |
| 37.8289 | 1936.00 | 0.1968 | 2.37829 | 5.02 | 01-075-1862; 01-075-1620 |
| 38.5369 | 3042.66 | 0.1279 | 2.33621 | 7.90 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.5666 | 6639.73 | 0.0600 | 2.22205 | 17.23 | 00-004-0587 |
| 40.6652 | 6621.93 | 0.0886 | 2.21872 | 17.18 | 00-004-0587; 01-086-2270 |
| 42.9538 | 3372.68 | 0.0886 | 2.10566 | 8.75 | 00-043-1022 |
| 43.3945 | 2289.28 | 0.0984 | 2.08529 | 5.94 | 01-075-1862; 01-086-2270 |
| 44.8601 | 5367.20 | 0.2558 | 2.02051 | 13.93 | 01-074-1132; 01-085-1327 |
| 45.4545 | 23181.09 | 0.1673 | 1.99546 | 60.15 | 01-077-2064; 01-080-2171; 01-086-2270; 01-080-1385 |
| 49.8421 | 569.57 | 0.1574 | 1.82960 | 1.48 | 01-075-1620; 01-080-2171; |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| | | | | | 01-080-2172; 01-080-1385 |
| 50.1992 | 1668.89 | 0.1080 | 1.81591 | 4.33 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235 |
| 50.3456 | 2047.65 | 0.1574 | 1.81248 | 5.31 | 00-004-0587; 00-022-1235 |
| 52.5733 | 902.67 | 0.0984 | 1.74081 | 2.34 | 01-075-1862 |
| 53.8768 | 836.01 | 0.1771 | 1.70173 | 2.17 | 01-077-2064 |
| 55.0029 | 230.13 | 0.2362 | 1.66952 | 0.60 | |
| 55.7313 | 634.90 | 0.2362 | 1.64942 | 1.65 | 01-074-1132; 01-086-2270 |
| 56.4868 | 6888.85 | 0.1800 | 1.62779 | 17.88 | 01-077-2064; 01-080-2171 |
| 56.6463 | 3588.73 | 0.0960 | 1.62762 | 9.31 | |
| 57.5504 | 2147.35 | 0.1320 | 1.60021 | 5.57 | 01-075-1862 |
| 57.6994 | 1334.76 | 0.0720 | 1.60040 | 3.46 | |
| 58.6630 | 1331.13 | 0.1440 | 1.57248 | 3.45 | 00-004-0587 |
| 59.4424 | 3533.29 | 0.2400 | 1.55371 | 9.17 | 01-074-1132; 01-075-1620 |
| 61.3196 | 380.69 | 0.1440 | 1.51057 | 0.99 | 01-075-1862 |
| 62.3391 | 1923.62 | 0.2640 | 1.48829 | 4.99 | 00-043-1022; 01-086-2270 |
| 65.2624 | 5802.69 | 0.0960 | 1.42852 | 15.06 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 65.4649 | 5221.00 | 0.1440 | 1.42459 | 13.55 | 01-086-2270 |
| 66.2194 | 4981.36 | 0.1200 | 1.41017 | 12.93 | 00-004-0587; 01-077-2064; 01-075-1620 |
| 66.5772 | 3886.87 | 0.1680 | 1.40346 | 10.09 | 01-075-1862; 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 68.2387 | 1270.48 | 0.1680 | 1.37328 | 3.30 | 01-075-1862; 00-022-1235 |
| 69.7496 | 230.90 | 0.2880 | 1.34718 | 0.60 | 01-074-1132; 01-075-1620; 01-080-2171 |
| 71.4258 | 675.42 | 0.2880 | 1.31963 | 1.75 | 01-075-1620; 01-080-2173; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 73.0641 | 550.21 | 0.1920 | 1.29403 | 1.43 | 01-077-2064; 01-086-2270 |
| 73.7235 | 1285.16 | 0.1680 | 1.28408 | 3.33 | 00-004-0587; 00-022-1235 |
| 73.9586 | 1388.73 | 0.2400 | 1.28057 | 3.60 | 01-074-1132 |
| 75.2961 | 7031.92 | 0.1320 | 1.26111 | 18.25 | 01-077-2064 |
| 75.5193 | 3490.74 | 0.1320 | 1.26106 | 9.06 | |
| 76.9070 | 614.28 | 0.1440 | 1.23866 | 1.59 | 01-075-1862; 01-086-2270 |
| 77.3890 | 873.21 | 0.3840 | 1.23215 | 2.27 | 01-074-1132; 01-086-2270 |
| 78.2496 | 1194.23 | 0.1920 | 1.22074 | 3.10 | 01-074-1132; 01-085-1327 |
| 78.6713 | 945.72 | 0.2400 | 1.21525 | 2.45 | 01-080-2171; 00-043-1022; 01-086-2270 |
| 80.7201 | 200.32 | 0.1440 | 1.18949 | 0.52 | 01-075-1862 |
| 82.6947 | 683.98 | 0.1920 | 1.16602 | 1.77 | 01-074-1132; 01-086-2270 |
| 83.9939 | 4239.21 | 0.1440 | 1.15126 | 11.00 | 01-077-2064; 01-086-2270 |
| 84.2559 | 2130.40 | 0.1200 | 1.15120 | 5.53 | |
| 85.8663 | 150.39 | 0.2880 | 1.13089 | 0.39 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 86.4529 | 162.54 | 0.3840 | 1.12471 | 0.42 | 01-075-1862 |
| 87.7388 | 222.69 | 0.6720 | 1.11152 | 0.58 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 89.0481 | 135.33 | 0.1920 | 1.09853 | 0.35 | 01-075-1862 |
| 90.4274 | 299.70 | 0.1920 | 1.08533 | 0.78 | 00-022-1235 |
| 91.0669 | 307.97 | 0.3840 | 1.07936 | 0.80 | |
| 94.1100 | 983.20 | 0.2160 | 1.05231 | 2.55 | 00-043-1022 |
| 94.5417 | 1207.47 | 0.1920 | 1.04864 | 3.13 | 00-004-0587 |
| 95.3135 | 888.74 | 0.0960 | 1.04218 | 2.31 | |
| 95.5953 | 435.56 | 0.1440 | 1.04244 | 1.13 | |
| 99.3990 | 522.51 | 0.2400 | 1.01001 | 1.36 | |
| 101.1326 | 1411.02 | 0.1920 | 0.99733 | 3.66 | |
| 101.5207 | 965.83 | 0.1440 | 0.99704 | 2.51 | |
| 104.8529 | 93.00 | 0.5760 | 0.97190 | 0.24 | |
| 105.9477 | 170.79 | 0.5760 | 0.96485 | 0.44 | 00-043-1022 |
| 107.7967 | 657.51 | 0.1440 | 0.95337 | 1.71 | |
| 109.8246 | 2158.22 | 0.0720 | 0.94137 | 5.60 | 00-043-1022 |

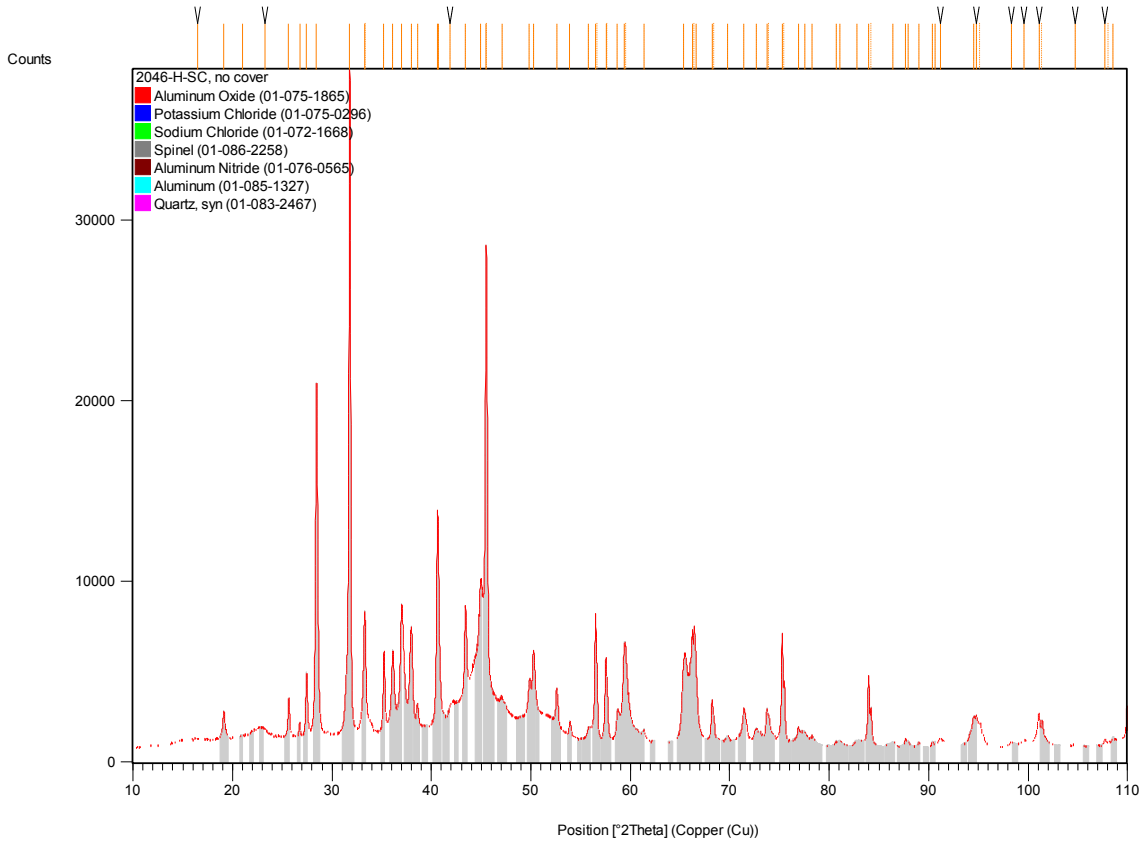
# FACILITY H

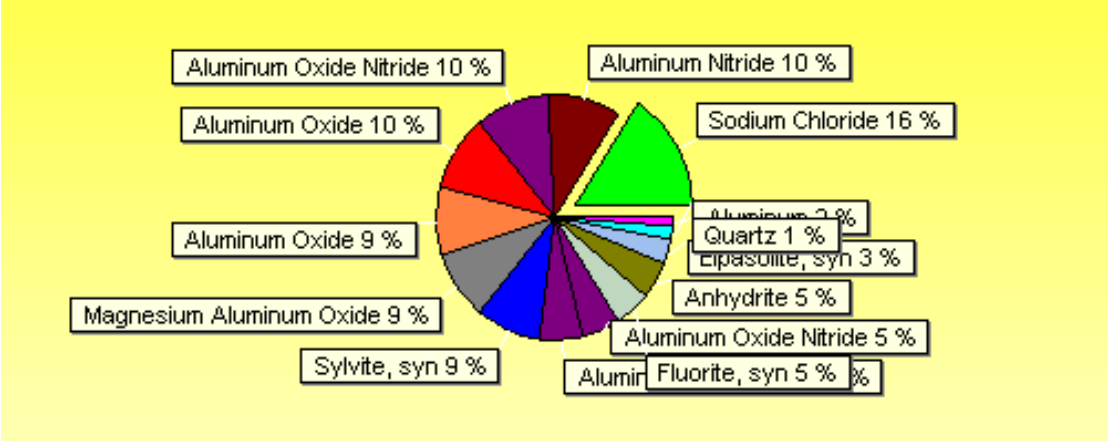
## 6.1: 2046-H-SC

### 6.1.1: Measurement Conditions of 2046-H

| | |
|---|---|
| Dataset Name | 2046-H-SC, no cover |
| File name | D:\SAP\2046-H-SC, no cover.sd |
| Sample Identification | 2046-H-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 9/27/2010 4:03:00 PM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 6.1.2: Main Graphics, Analyze View of 2046-H




**6.1.3: Pattern List of 2046-H**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 54 | Aluminum Oxide | Al2 O3 | 10 | 22 | 0 |
| 00-004-0587 | 49 | Sylvite, syn | K Cl | 9 | 10 | 0 |
| 01-077-2064 | 51 | Sodium Chloride | Na Cl | 16 | 9 | 0 |
| 01-074-1132 | 48 | Magnesium Aluminum Oxide | Mg Al2 O4 | 9 | 14 | 0 |
| 01-075-1620 | 55 | Aluminum Nitride | Al N | 10 | 11 | 0 |
| 01-080-2171 | 19 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 10 | 12 | 0 |
| 01-080-2172 | 20 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 11 | 0 |
| 01-080-2173 | 20 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 11 | 0 |
| 01-085-1327 | 26 | Aluminum | Al | 2 | 5 | 0 |
| 00-004-0864 | 17 | Fluorite, syn | Ca F2 | 5 | 5 | 0 |
| 00-022-1235 | 17 | Elpasolite, syn | K2 Na Al F6 | 3 | 13 | 0 |
| 01-086-2270 | 19 | Anhydrite | Ca ( S O4 ) | 5 | 38 | 0 |
| 01-080-1385 | 22 | Aluminum Oxide | Al2.667 O4 | 9 | 12 | 0 |
| 01-085-0930 | 15 | Quartz | Si O2 | 1 | 18 | 0 |

**6.1.4: Peak List of 2046-H**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 16.4748 | 81.83 | 0.4723 | 5.38081 | 0.22 | |
| 19.1387 | 1521.76 | 0.0689 | 4.63746 | 4.17 | 01-074-1132; 01-086-2270 |
| 20.9852 | 195.92 | 0.1968 | 4.23338 | 0.54 | 01-085-0930 |
| 23.2412 | 455.83 | 0.3149 | 3.82732 | 1.25 | |
| 25.6315 | 1961.53 | 0.0590 | 3.47556 | 5.37 | 01-075-1862 |
| 26.7539 | 711.68 | 0.0787 | 3.33225 | 1.95 | 01-085-0930 |
| 27.4351 | 3491.37 | 0.0787 | 3.25103 | 9.56 | 01-077-2064 |
| 28.4510 | 19480.03 | 0.1968 | 3.13722 | 53.34 | 00-004-0587; 00-004-0864; 01-086-2270 |
| 31.7722 | 36520.83 | 0.1673 | 2.81646 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 33.2970 | 6838.22 | 0.1440 | 2.68866 | 18.72 | 01-075-1620 |
| 33.3567 | 6072.16 | 0.0360 | 2.69065 | 16.63 | |
| 35.2212 | 4607.44 | 0.1080 | 2.54606 | 12.62 | 01-075-1862 |
| 36.0993 | 4736.92 | 0.1200 | 2.48611 | 12.97 | 01-075-1620 |
| 36.9913 | 7206.42 | 0.3360 | 2.42818 | 19.73 | 01-074-1132 |
| 38.0153 | 5766.71 | 0.1080 | 2.36510 | 15.79 | 01-075-1620 |
| 38.6174 | 1778.33 | 0.1680 | 2.32959 | 4.87 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 40.5850 | 12019.13 | 0.0960 | 2.22108 | 32.91 | 00-004-0587 |
| 40.6777 | 11576.22 | 0.1080 | 2.21623 | 31.70 | 00-004-0587; 01-086-2270 |
| 41.8989 | 1828.97 | 0.3840 | 2.15442 | 5.01 | |
| 43.4169 | 7283.36 | 0.0840 | 2.08254 | 19.94 | 01-075-1862; 01-086-2270 |
| 44.9242 | 8525.01 | 0.1680 | 2.01611 | 23.34 | 01-074-1132 |
| 45.4780 | 25957.25 | 0.1440 | 1.99284 | 71.08 | 01-077-2064; 01-080-2171; 01-080-2172; 01-086-2270; 01-080-1385 |
| 45.5989 | 20037.27 | 0.0960 | 1.99277 | 54.87 | |
| 47.0689 | 2271.43 | 0.2400 | 1.92912 | 6.22 | 00-004-0864 |
| 49.8215 | 3096.63 | 0.1920 | 1.82880 | 8.48 | 01-075-1620; 01-080-2171; 01-080-2172; 01-080-1385 |
| 50.2326 | 4807.98 | 0.0960 | 1.81479 | 13.17 | 00-004-0587; 01-080-2173; |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| | | | | | 00-022-1235; 01-085-0930 |
| 52.5861 | 2798.93 | 0.1920 | 1.73897 | 7.66 | 01-075-1862 |
| 53.8941 | 1034.15 | 0.1440 | 1.69981 | 2.83 | 01-077-2064 |
| 55.7338 | 631.48 | 0.2880 | 1.64798 | 1.73 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.5072 | 6988.29 | 0.1440 | 1.62725 | 19.14 | 01-077-2064; 01-080-2171; 01-080-1385 |
| 56.6772 | 3534.84 | 0.0960 | 1.62680 | 9.68 | |
| 57.5470 | 4579.54 | 0.1440 | 1.60029 | 12.54 | 01-075-1862 |
| 57.7088 | 2474.38 | 0.1440 | 1.60016 | 6.78 | |
| 58.6765 | 1588.22 | 0.1920 | 1.57215 | 4.35 | 00-004-0587 |
| 59.3827 | 5232.90 | 0.2400 | 1.55513 | 14.33 | 01-074-1132; 01-075-1620 |
| 59.5540 | 4982.68 | 0.1440 | 1.55492 | 13.64 | |
| 61.3612 | 699.29 | 0.1200 | 1.50964 | 1.91 | 01-075-1862 |
| 65.3366 | 4338.21 | 0.2640 | 1.42707 | 11.88 | 01-074-1132; 01-086-2270 |
| 66.2258 | 6101.62 | 0.1200 | 1.41005 | 16.71 | 00-004-0587; 01-077-2064; 01-075-1620 |
| 66.4389 | 6448.83 | 0.0840 | 1.40954 | 17.66 | |
| 66.5966 | 4682.26 | 0.0960 | 1.40310 | 12.82 | 01-075-1862; 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 68.2452 | 2331.23 | 0.1440 | 1.37317 | 6.38 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.4360 | 1221.39 | 0.1200 | 1.37321 | 3.34 | |
| 69.7391 | 424.94 | 0.2400 | 1.34736 | 1.16 | 01-074-1132; 01-075-1620; 01-080-2171 |
| 71.4206 | 1875.84 | 0.2400 | 1.31971 | 5.14 | 01-075-1620; 01-080-2173; 01-086-2270 |
| 72.6220 | 830.58 | 0.2640 | 1.30081 | 2.27 | 01-075-1620 |
| 73.7320 | 1973.92 | 0.1680 | 1.28395 | 5.40 | 00-004-0587; 00-022-1235 |
| 73.9490 | 1498.88 | 0.1440 | 1.28390 | 4.10 | |
| 75.2671 | 6100.75 | 0.1320 | 1.26153 | 16.70 | 01-077-2064 |
| 75.4989 | 3476.91 | 0.1320 | 1.26135 | 9.52 | |

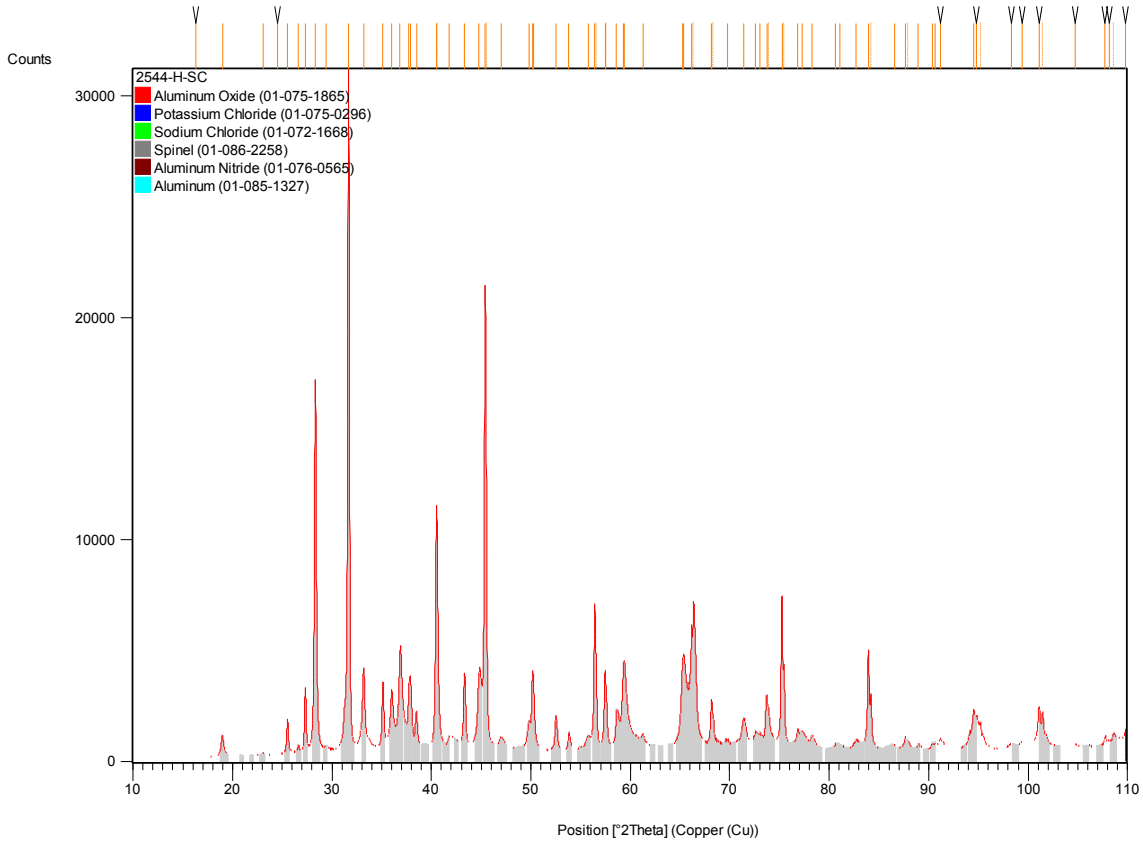
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 76.8793 | 994.21 | 0.1440 | 1.23904 | 2.72 | 01-075-1862; 01-086-2270 |
| 77.5282 | 735.60 | 0.4800 | 1.23028 | 2.01 | 01-074-1132; 01-086-2270; 01-085-0930 |
| 78.2986 | 512.45 | 0.1440 | 1.22009 | 1.40 | 01-074-1132; 01-085-1327 |
| 80.6599 | 273.97 | 0.1920 | 1.19022 | 0.75 | 01-075-1862 |
| 81.0708 | 288.31 | 0.3840 | 1.18523 | 0.79 | 01-075-1620; 01-085-0930 |
| 82.7867 | 319.64 | 0.5760 | 1.16496 | 0.88 | 01-074-1132; 01-086-2270 |
| 83.9516 | 3865.61 | 0.1680 | 1.15173 | 10.58 | 01-077-2064; 01-085-0930 |
| 84.2228 | 2090.23 | 0.1080 | 1.15157 | 5.72 | |
| 86.4027 | 212.95 | 0.3840 | 1.12524 | 0.58 | 01-075-1862 |
| 87.6745 | 410.04 | 0.1680 | 1.11216 | 1.12 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0930 |
| 87.9273 | 310.02 | 0.1920 | 1.10962 | 0.85 | 01-080-2173 |
| 88.9757 | 251.97 | 0.1920 | 1.09924 | 0.69 | 01-075-1862 |
| 90.3437 | 254.90 | 0.1200 | 1.08611 | 0.70 | 00-022-1235 |
| 90.6556 | 233.08 | 0.1440 | 1.08319 | 0.64 | 00-022-1235 |
| 91.1441 | 481.97 | 0.1680 | 1.07865 | 1.32 | |
| 94.5166 | 1624.78 | 0.2400 | 1.04885 | 4.45 | 00-004-0587 |
| 94.7986 | 1606.84 | 0.1920 | 1.04648 | 4.40 | |
| 95.1860 | 1272.35 | 0.1200 | 1.04583 | 3.48 | |
| 98.2940 | 289.58 | 0.1920 | 1.01839 | 0.79 | |
| 99.5697 | 309.55 | 0.7680 | 1.00874 | 0.85 | |
| 101.0890 | 1835.19 | 0.1680 | 0.99764 | 5.03 | |
| 101.4174 | 1416.46 | 0.1920 | 0.99777 | 3.88 | |
| 104.7427 | 117.20 | 0.4800 | 0.97262 | 0.32 | |
| 107.7151 | 265.93 | 0.1200 | 0.95387 | 0.73 | |
| 108.0901 | 89.56 | 0.1440 | 0.95396 | 0.25 | |
| 108.5642 | 193.48 | 0.1920 | 0.94876 | 0.53 | 00-004-0587 |

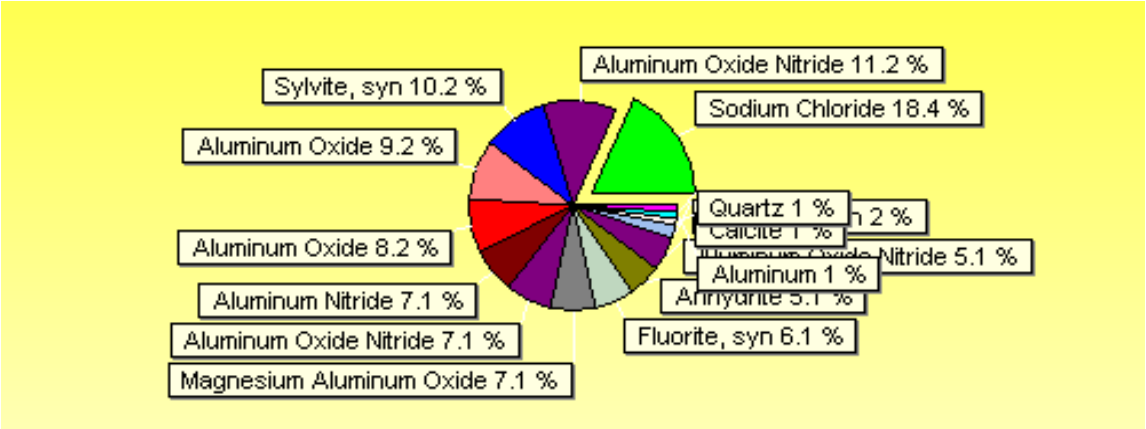
## 6.2: 2544-H-SC

### 6.2.1: Measurement Conditions of 2544-H

| | |
|---|---|
| Dataset Name | 2544-H-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2544-H-SC.udf |
| Sample Identification | 2544-H-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

**6.2.2: Main Graphics, Analyze View of 2544-H**




**6.2.3: Pattern List of 2544-H**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 51 | Aluminum Oxide | Al2 O3 | 8 | 20 | 0 |
| 00-004-0587 | 52 | Sylvite, syn | K Cl | 10 | 10 | 0 |
| 01-077-2064 | 52 | Sodium Chloride | Na Cl | 18 | 9 | 0 |
| 01-074-1132 | 49 | Magnesium Aluminum Oxide | Mg Al2 O4 | 7 | 14 | 0 |
| 01-075-1620 | 52 | Aluminum Nitride | Al N | 7 | 10 | 0 |
| 01-080-2171 | 15 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 11 | 13 | 0 |
| 01-080-2172 | 15 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 7 | 12 | 0 |
| 01-080-2173 | 18 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 12 | 0 |
| 01-085-1327 | 39 | Aluminum | Al | 1 | 5 | 0 |
| 00-004-0864 | 24 | Fluorite, syn | Ca F2 | 6 | 6 | 0 |
| 00-022-1235 | 21 | Elpasolite, syn | K2 Na Al F6 | 2 | 15 | 0 |
| 01-086-2270 | 18 | Anhydrite | Ca ( S O4 ) | 5 | 36 | 0 |
| 01-080-1385 | 13 | Aluminum Oxide | Al2.667 O4 | 9 | 12 | 0 |
| 01-086-2340 | 13 | Calcite | Ca ( C O3 ) | 1 | 19 | 0 |
| 01-085-0930 | 5 | Quartz | Si O2 | 1 | 18 | 0 |

**6.2.4: Peak List of 2544-H**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 16.2741 | 35.25 | 0.3149 | 5.44673 | 0.11 | |
| 18.9841 | 935.36 | 0.1771 | 4.67488 | 3.05 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0711 | 59.34 | 0.3149 | 3.85516 | 0.19 | 01-086-2270; 01-086-2340 |
| 24.4870 | 86.25 | 0.2755 | 3.63534 | 0.28 | |
| 25.5456 | 1534.15 | 0.1574 | 3.48706 | 5.00 | 01-075-1862; 01-086-2270 |
| 26.6018 | 433.06 | 0.1181 | 3.35096 | 1.41 | 01-085-0930 |
| 27.2985 | 2849.23 | 0.0984 | 3.26700 | 9.29 | 01-077-2064 |
| 28.3179 | 16571.75 | 0.1574 | 3.15167 | 54.05 | 00-004-0587; 00-004-0864 |
| 29.4181 | 362.53 | 0.2362 | 3.03625 | 1.18 | 01-086-2340 |
| 31.6538 | 30659.65 | 0.1771 | 2.82672 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.2057 | 3736.59 | 0.1968 | 2.69808 | 12.19 | 01-075-1620 |
| 35.0986 | 3099.12 | 0.1574 | 2.55678 | 10.11 | 01-075-1862 |
| 36.0093 | 2767.23 | 0.1771 | 2.49418 | 9.03 | 01-075-1620; 01-086-2340 |
| 36.8244 | 4481.50 | 0.0590 | 2.44082 | 14.62 | 01-074-1132 |
| 37.7235 | 2837.20 | 0.1440 | 2.38272 | 9.25 | 01-075-1862; 01-075-1620; 01-080-2173 |
| 37.8860 | 3385.89 | 0.1771 | 2.37484 | 11.04 | 01-075-1862; 01-075-1620 |
| 38.4867 | 1828.36 | 0.0689 | 2.33914 | 5.96 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.5062 | 11060.23 | 0.1680 | 2.22522 | 36.07 | 00-004-0587; 01-085-0930 |
| 40.5861 | 9346.78 | 0.0840 | 2.22654 | 30.49 | |
| 41.8025 | 676.17 | 0.4320 | 2.15916 | 2.21 | 01-075-1862 |
| 43.2996 | 3407.58 | 0.1680 | 2.08791 | 11.11 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 44.7567 | 3297.82 | 0.2640 | 2.02326 | 10.76 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3893 | 20587.23 | 0.1440 | 1.99652 | 67.15 | 01-077-2064; 01-086-2270 |
| 45.5283 | 12271.99 | 0.1080 | 1.99570 | 40.03 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 47.0005 | 604.22 | 0.3360 | 1.93177 | 1.97 | 00-004-0864; 01-086-2270; 01-086-2340 |
| 49.8302 | 1312.32 | 0.1920 | 1.82849 | 4.28 | 01-075-1620; 01-080-2171; 01-080-2172; 01-080-1385 |
| 50.1688 | 3509.51 | 0.1200 | 1.81694 | 11.45 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 50.2736 | 2972.16 | 0.1200 | 1.81340 | 9.69 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 52.5164 | 1513.30 | 0.1200 | 1.74112 | 4.94 | 01-075-1862 |
| 53.8128 | 734.55 | 0.1440 | 1.70219 | 2.40 | 01-077-2064 |
| 55.7461 | 592.34 | 0.2880 | 1.64765 | 1.93 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4247 | 6520.11 | 0.1320 | 1.62943 | 21.27 | 01-077-2064 |
| 56.5861 | 3613.99 | 0.0600 | 1.62921 | 11.79 | |
| 57.4605 | 3516.87 | 0.1560 | 1.60250 | 11.47 | 01-075-1862; 01-086-2340; 01-085-0930 |
| 57.6248 | 2028.35 | 0.0720 | 1.60229 | 6.62 | |
| 58.6044 | 1642.63 | 0.1320 | 1.57392 | 5.36 | 00-004-0587 |
| 59.3253 | 3796.09 | 0.1440 | 1.55650 | 12.38 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 59.4221 | 3826.77 | 0.1200 | 1.55419 | 12.48 | 01-074-1132; 01-075-1620 |
| 61.2761 | 541.72 | 0.1680 | 1.51154 | 1.77 | 01-075-1862; 01-086-2270 |
| 65.2479 | 3768.47 | 0.1920 | 1.42880 | 12.29 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 65.3710 | 4086.52 | 0.1680 | 1.42641 | 13.33 | 01-074-1132; 01-086-2270 |
| 66.1732 | 5373.75 | 0.1200 | 1.41105 | 17.53 | 00-004-0587; 01-077-2064; 01-075-1620 |
| 66.3584 | 6398.17 | 0.0840 | 1.41106 | 20.87 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 68.1678 | 2067.32 | 0.1920 | 1.37454 | 6.74 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.3664 | 1260.98 | 0.1200 | 1.37443 | 4.11 | |
| 69.7966 | 295.00 | 0.4320 | 1.34639 | 0.96 | 01-074-1132; 01-075-1620; 01-080-2171; 01-080-2172; 01-080-1385; 01-086-2340 |
| 71.3780 | 1334.45 | 0.0960 | 1.32040 | 4.35 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270 |
| 72.5977 | 676.50 | 0.2400 | 1.30119 | 2.21 | 01-075-1620; 01-086-2340 |
| 73.0395 | 686.95 | 0.1440 | 1.29440 | 2.24 | 01-077-2064; 01-086-2270 |
| 73.7144 | 2358.20 | 0.1320 | 1.28421 | 7.69 | 00-004-0587; 00-022-1235; 01-086-2340 |
| 73.9082 | 1740.39 | 0.1200 | 1.28451 | 5.68 | |
| 75.2488 | 6843.19 | 0.1320 | 1.26179 | 22.32 | 01-077-2064 |
| 75.4685 | 3599.17 | 0.1320 | 1.26179 | 11.74 | |
| 76.8357 | 844.44 | 0.1440 | 1.23964 | 2.75 | 01-075-1862 |
| 77.2803 | 716.10 | 0.4800 | 1.23361 | 2.34 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.2348 | 569.57 | 0.2880 | 1.22093 | 1.86 | 01-074-1132; 01-085-1327 |
| 80.6465 | 258.13 | 0.1920 | 1.19039 | 0.84 | 01-075-1862; 01-086-2340 |
| 81.0525 | 204.95 | 0.2880 | 1.18545 | 0.67 | 01-075-1620; 01-086-2340; 01-085-0930 |
| 82.6744 | 369.59 | 0.4800 | 1.16625 | 1.21 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.9320 | 4428.34 | 0.1200 | 1.15195 | 14.44 | 01-077-2064; 01-086-2340; 01-085-0930 |
| 84.2028 | 2405.51 | 0.1320 | 1.15179 | 7.85 | |
| 86.5318 | 185.84 | 0.4800 | 1.12389 | 0.61 | 01-075-1862 |

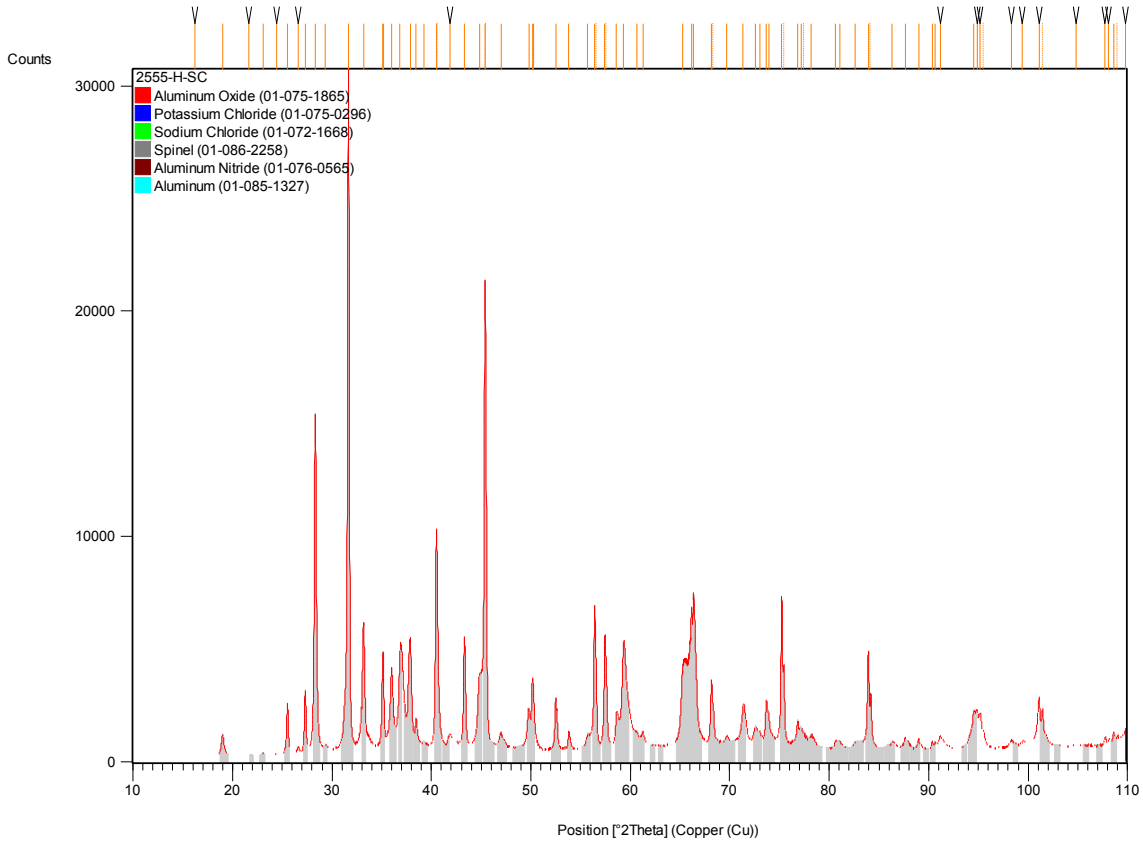
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 87.6495 | 544.31 | 0.1440 | 1.11242 | 1.78 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 87.9418 | 376.91 | 0.1440 | 1.11223 | 1.23 | |
| 88.9377 | 213.82 | 0.1920 | 1.09961 | 0.70 | 01-075-1862; 01-086-2270 |
| 90.3547 | 319.34 | 0.1680 | 1.08601 | 1.04 | 00-022-1235 |
| 90.6426 | 283.88 | 0.1440 | 1.08331 | 0.93 | 00-022-1235 |
| 91.1428 | 459.16 | 0.1920 | 1.07866 | 1.50 | |
| 94.5310 | 1657.65 | 0.1080 | 1.04873 | 5.41 | 00-004-0587 |
| 94.8289 | 1447.37 | 0.1440 | 1.04622 | 4.72 | |
| 95.2183 | 1086.36 | 0.1200 | 1.04556 | 3.54 | |
| 98.3242 | 207.94 | 0.2880 | 1.01816 | 0.68 | |
| 99.3718 | 264.06 | 0.7680 | 1.01022 | 0.86 | |
| 101.1071 | 1828.22 | 0.1680 | 0.99751 | 5.96 | |
| 101.4597 | 1534.91 | 0.1680 | 0.99747 | 5.01 | |
| 104.7300 | 72.02 | 0.3840 | 0.97270 | 0.23 | |
| 107.7531 | 615.89 | 0.1200 | 0.95364 | 2.01 | |
| 108.1347 | 549.35 | 0.1440 | 0.95133 | 1.79 | |
| 108.5981 | 882.95 | 0.2160 | 0.95091 | 2.88 | |
| 109.7971 | 1252.81 | 0.0480 | 0.94153 | 4.09 | |

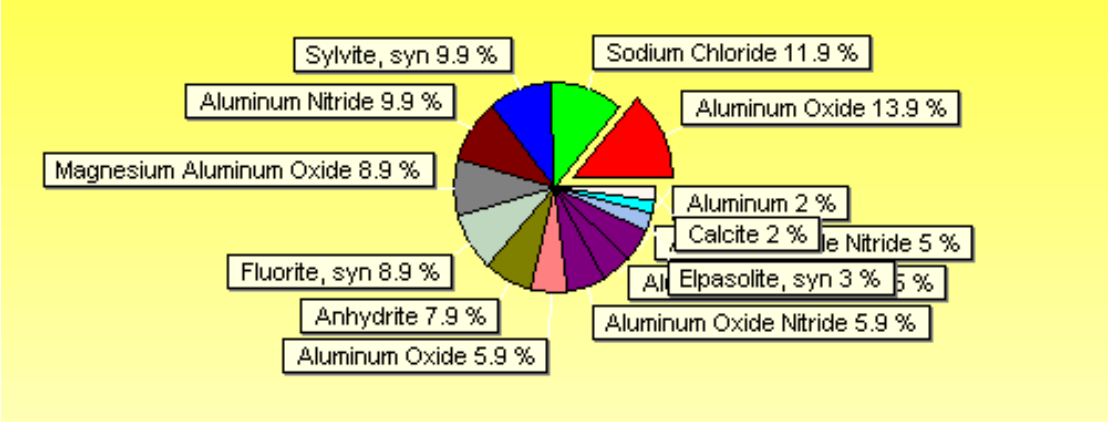
## 6.3: 2555-H-SC

### 6.3.1: Measurement Conditions of 2555-H

| | |
|---|---|
| Dataset Name | 2555-H-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2555-H-SC.udf |
| Sample Identification | 2555-H-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 6.3.2: Main Graphics, Analyze View of 2555-H




**6.3.3: Pattern List of 2555-H**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 46 | Aluminum Oxide | Al2 O3 | 14 | 21 | 0 |
| 00-004-0587 | 36 | Sylvite, syn | K Cl | 10 | 10 | 0 |
| 01-077-2064 | 33 | Sodium Chloride | Na Cl | 12 | 9 | 0 |
| 01-074-1132 | 36 | Magnesium Aluminum Oxide | Mg Al2 O4 | 9 | 15 | 0 |
| 01-075-1620 | 36 | Aluminum Nitride | Al N | 10 | 12 | 0 |
| 01-080-2171 | 16 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 6 | 13 | 0 |
| 01-080-2172 | 20 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 5 | 12 | 0 |
| 01-080-2173 | 21 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 11 | 0 |
| 01-085-1327 | 26 | Aluminum | Al | 2 | 5 | 0 |
| 00-004-0864 | 21 | Fluorite, syn | Ca F2 | 9 | 6 | 0 |
| 00-022-1235 | 25 | Elpasolite, syn | K2 Na Al F6 | 3 | 16 | 0 |
| 01-086-2270 | 26 | Anhydrite | Ca ( S O4 ) | 8 | 40 | 0 |
| 01-080-1385 | 20 | Aluminum Oxide | Al2.667 O4 | 6 | 13 | 0 |
| 01-086-2340 | 14 | Calcite | Ca ( C O3 ) | 2 | 22 | 0 |

**6.3.4: Peak List of 2555-H**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 16.2129 | 61.99 | 0.1968 | 5.46714 | 0.20 | |
| 19.0277 | 941.61 | 0.2165 | 4.66426 | 3.09 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 21.6765 | 58.60 | 0.2362 | 4.09992 | 0.19 | |
| 23.0390 | 89.40 | 0.2362 | 3.86044 | 0.29 | 01-086-2270; 01-086-2340 |
| 24.4605 | 101.25 | 0.2362 | 3.63923 | 0.33 | |
| 25.5167 | 2296.59 | 0.0689 | 3.49094 | 7.54 | 01-075-1862; 01-086-2270 |
| 26.5799 | 357.49 | 0.1181 | 3.35366 | 1.17 | |
| 27.3061 | 2833.00 | 0.1673 | 3.26611 | 9.30 | 01-077-2064 |
| 28.2976 | 14619.11 | 0.1673 | 3.15388 | 48.00 | 00-004-0587; 00-004-0864 |
| 29.3564 | 409.12 | 0.1574 | 3.04249 | 1.34 | 01-086-2340 |
| 31.6512 | 30458.97 | 0.1181 | 2.82694 | 100.00 | 01-077-2064; 01-080-2171; 01-080-1385 |
| 33.1715 | 5792.04 | 0.0689 | 2.70078 | 19.02 | 01-075-1620 |
| 35.0783 | 4144.79 | 0.0720 | 2.55610 | 13.61 | 01-075-1862 |
| 35.1413 | 4428.18 | 0.0492 | 2.55378 | 14.54 | 01-075-1862 |
| 35.9937 | 3779.48 | 0.0590 | 2.49523 | 12.41 | 01-075-1620; 01-086-2340 |
| 36.8083 | 4052.91 | 0.2755 | 2.44185 | 13.31 | 01-074-1132 |
| 37.8844 | 4870.29 | 0.3149 | 2.37493 | 15.99 | 01-075-1862; 01-075-1620 |
| 38.4428 | 1447.80 | 0.1574 | 2.34171 | 4.75 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 39.2597 | 429.00 | 0.2362 | 2.29485 | 1.41 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-086-2340 |
| 40.4851 | 9721.87 | 0.1560 | 2.22633 | 31.92 | 00-004-0587 |
| 40.5657 | 8662.00 | 0.0840 | 2.22761 | 28.44 | |
| 41.8271 | 762.11 | 0.3360 | 2.15795 | 2.50 | |
| 43.2942 | 5002.49 | 0.1080 | 2.08816 | 16.42 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 44.8099 | 3333.31 | 0.2400 | 2.02098 | 10.94 | 01-074-1132; 01-085-1327 |
| 45.3820 | 20556.10 | 0.1560 | 1.99683 | 67.49 | 01-077-2064; 01-086-2270 |
| 45.5031 | 13356.11 | 0.0720 | 1.99675 | 43.85 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 46.9814 | 768.82 | 0.2880 | 1.93251 | 2.52 | 00-004-0864; 01-086-2270; 01-086-2340 |
| 49.7808 | 1813.66 | 0.2160 | 1.83019 | 5.95 | 01-075-1620; 01-080-2171 |
| 50.1461 | 3119.33 | 0.0840 | 1.81772 | 10.24 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 50.2936 | 2297.19 | 0.1200 | 1.81273 | 7.54 | 00-004-0587; 00-022-1235 |
| 52.4810 | 2160.72 | 0.1080 | 1.74221 | 7.09 | 01-075-1862 |
| 53.8035 | 719.21 | 0.1200 | 1.70246 | 2.36 | 01-077-2064 |
| 55.7190 | 625.00 | 0.2400 | 1.64838 | 2.05 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4267 | 6327.52 | 0.1320 | 1.62938 | 20.77 | 01-077-2064 |
| 56.5760 | 3485.62 | 0.0840 | 1.62947 | 11.44 | |
| 57.4317 | 4835.67 | 0.1680 | 1.60323 | 15.88 | 01-075-1862; 01-086-2340 |
| 57.6130 | 2908.39 | 0.1200 | 1.60259 | 9.55 | |
| 58.6134 | 1558.38 | 0.1200 | 1.57369 | 5.12 | 00-004-0587 |
| 59.3154 | 4618.51 | 0.2400 | 1.55673 | 15.16 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 60.6214 | 632.54 | 0.2880 | 1.52628 | 2.08 | 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385; 01-086-2340 |
| 65.2332 | 3268.60 | 0.1200 | 1.42909 | 10.73 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 66.1651 | 6082.56 | 0.1200 | 1.41120 | 19.97 | 01-077-2064; 01-075-1620 |
| 66.3563 | 6642.55 | 0.1920 | 1.40760 | 21.81 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 68.1372 | 2825.04 | 0.0960 | 1.37508 | 9.27 | 01-075-1862; 00-022-1235 |
| 68.3663 | 1561.01 | 0.1200 | 1.37444 | 5.12 | |
| 69.6863 | 486.70 | 0.2400 | 1.34825 | 1.60 | 01-074-1132; 01-075-1620; 01-086-2340 |
| 71.3465 | 1854.66 | 0.1920 | 1.32090 | 6.09 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270 |
| 72.5788 | 925.39 | 0.3360 | 1.30148 | 3.04 | 01-075-1620; 01-086-2340 |
| 73.0270 | 699.22 | 0.1440 | 1.29459 | 2.30 | 01-077-2064; 01-086-2270 |
| 73.6748 | 2072.68 | 0.0720 | 1.28480 | 6.80 | 00-004-0587; 00-022-1235; 01-086-2340 |
| 73.9183 | 1476.24 | 0.1200 | 1.28117 | 4.85 | 00-004-0587; 01-074-1132; 00-022-1235 |
| 75.2278 | 6697.10 | 0.1680 | 1.26209 | 21.99 | 01-077-2064 |
| 75.4534 | 3617.75 | 0.1320 | 1.26200 | 11.88 | |
| 76.8271 | 1232.77 | 0.0840 | 1.23975 | 4.05 | 01-075-1862 |
| 77.1388 | 900.24 | 0.1920 | 1.23552 | 2.96 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 77.4524 | 702.34 | 0.1440 | 1.23436 | 2.31 | |
| 78.2197 | 576.62 | 0.1920 | 1.22113 | 1.89 | 01-074-1132; 01-085-1327 |
| 80.6222 | 353.81 | 0.1680 | 1.19069 | 1.16 | 01-075-1862; 01-086-2340 |
| 81.0211 | 323.90 | 0.2880 | 1.18583 | 1.06 | 01-075-1620; 01-086-2340 |
| 82.6276 | 285.34 | 0.3840 | 1.16680 | 0.94 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.9472 | 4168.92 | 0.1680 | 1.15178 | 13.69 | 01-077-2064; 01-086-2340 |
| 84.1626 | 2433.86 | 0.1200 | 1.15224 | 7.99 | |
| 86.2864 | 287.61 | 0.1920 | 1.12646 | 0.94 | 01-075-1862 |
| 87.6408 | 511.53 | 0.1200 | 1.11251 | 1.68 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |

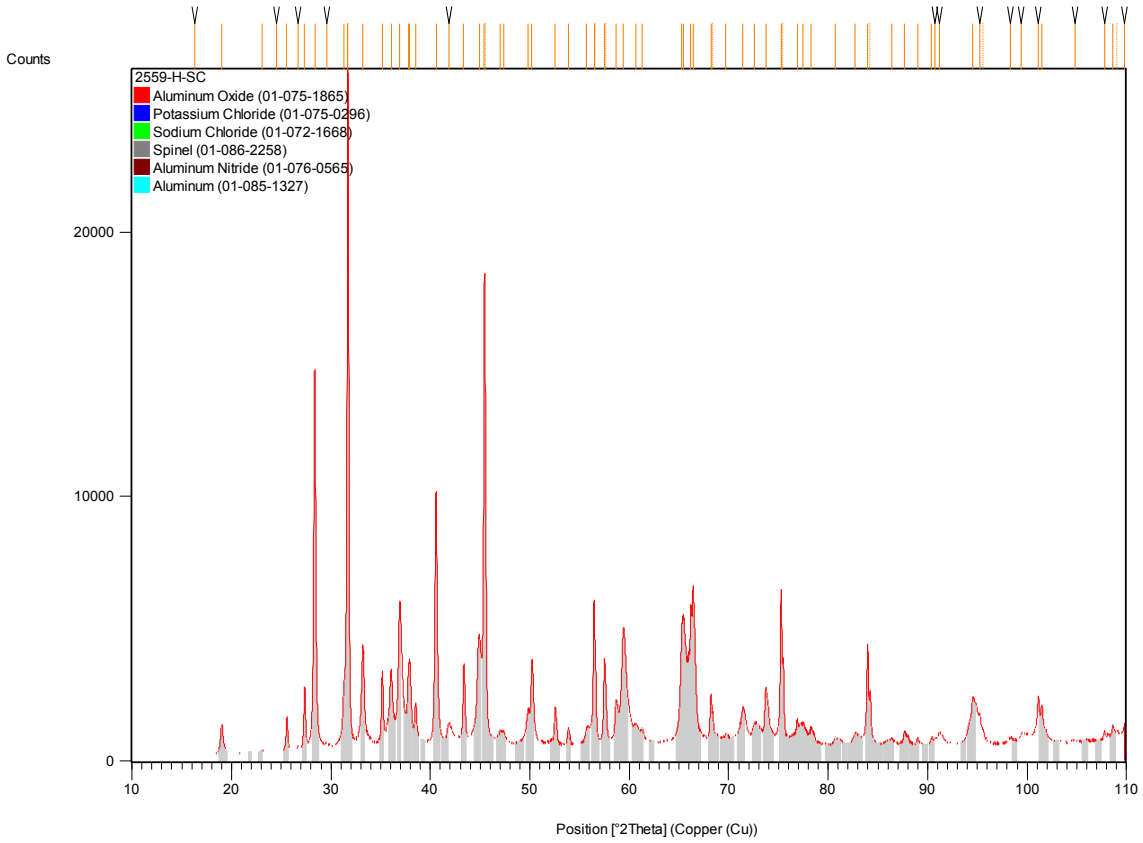
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 88.9924 | 375.10 | 0.1920 | 1.09907 | 1.23 | 01-075-1862 |
| 90.3295 | 279.33 | 0.1440 | 1.08625 | 0.92 | 00-022-1235 |
| 90.6507 | 276.42 | 0.1440 | 1.08323 | 0.91 | 00-022-1235 |
| 91.1371 | 550.77 | 0.1440 | 1.07872 | 1.81 | |
| 94.5337 | 1664.85 | 0.1440 | 1.04871 | 5.47 | 00-004-0587 |
| 94.8409 | 1712.98 | 0.1680 | 1.04612 | 5.62 | |
| 95.1980 | 1530.79 | 0.2640 | 1.04314 | 5.03 | |
| 95.5193 | 757.63 | 0.1440 | 1.04306 | 2.49 | |
| 98.3182 | 309.23 | 0.3360 | 1.01820 | 1.02 | |
| 99.3751 | 227.41 | 0.3840 | 1.01019 | 0.75 | |
| 101.1008 | 2175.52 | 0.0720 | 0.99756 | 7.14 | |
| 101.4735 | 1528.53 | 0.1920 | 0.99737 | 5.02 | |
| 104.7802 | 210.73 | 0.5760 | 0.97237 | 0.69 | |
| 107.7294 | 707.64 | 0.1200 | 0.95378 | 2.32 | |
| 108.0997 | 626.63 | 0.1440 | 0.95154 | 2.06 | |
| 108.6081 | 932.57 | 0.1200 | 0.94850 | 3.06 | 00-004-0587 |
| 109.0126 | 844.37 | 0.1920 | 0.94846 | 2.77 | |
| 109.7994 | 1284.43 | 0.0360 | 0.94152 | 4.22 | |

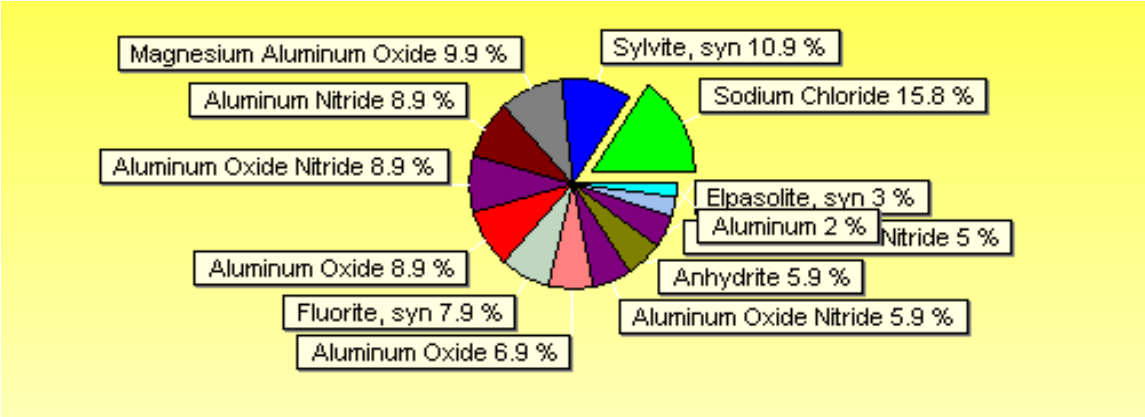
## 6.4: 2559-H-SC

### 6.4.1: Measurement Conditions of 2559-H

| | |
|---|---|
| Dataset Name | 2559-H-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2559-H-SC.udf |
| Sample Identification | 2559-H-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 6.4.2: Main Graphics, Analyze View of 2559-H


Counts
2559-H-SC
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Magnesium Aluminum Oxide 9.9 %
Sylvite, syn 10.9 %
Aluminum Nitride 8.9 %
Sodium Chloride 15.8 %
Aluminum Oxide Nitride 8.9 %
Elpasolite, syn 3 %
Aluminum 2 %
Nitride 5 %
Aluminum Oxide 8.9 %
Anhydrite 5.9 %
Fluorite, syn 7.9 %
Aluminum Oxide Nitride 5.9 %
Aluminum Oxide 6.9 %

**6.4.3: Pattern List of 2559-H**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 52 | Aluminum Oxide | Al2 O3 | 9 | 21 | 0 |
| 00-004-0587 | 53 | Sylvite, syn | K Cl | 11 | 10 | 0 |
| 01-077-2064 | 47 | Sodium Chloride | Na Cl | 16 | 9 | 0 |
| 01-074-1132 | 53 | Magnesium Aluminum Oxide | Mg Al2 O4 | 10 | 15 | 0 |
| 01-075-1620 | 52 | Aluminum Nitride | Al N | 9 | 12 | 0 |
| 01-080-2171 | 15 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 9 | 12 | 0 |
| 01-080-2172 | 21 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 22 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 11 | 0 |
| 01-085-1327 | 34 | Aluminum | Al | 2 | 5 | 0 |
| 00-004-0864 | 24 | Fluorite, syn | Ca F2 | 8 | 7 | 0 |
| 00-022-1235 | 24 | Elpasolite, syn | K2 Na Al F6 | 3 | 15 | 0 |
| 01-086-2270 | 23 | Anhydrite | Ca ( S O4 ) | 6 | 40 | 0 |
| 01-080-1385 | 18 | Aluminum Oxide | Al2.667 O4 | 7 | 12 | 0 |

**6.4.4: Peak List of 2559-H**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 16.2868 | 33.91 | 0.2362 | 5.44250 | 0.13 | |
| 19.0058 | 1126.17 | 0.0787 | 4.66958 | 4.39 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0794 | 65.88 | 0.2362 | 3.85378 | 0.26 | 01-086-2270 |
| 24.5074 | 84.57 | 0.2362 | 3.63237 | 0.33 | |
| 25.5530 | 1245.87 | 0.0787 | 3.48605 | 4.86 | 01-075-1862; 01-086-2270 |
| 26.6502 | 210.70 | 0.1181 | 3.34498 | 0.82 | |
| 27.3489 | 2407.18 | 0.0984 | 3.26109 | 9.39 | 01-077-2064 |
| 28.3798 | 14358.28 | 0.1476 | 3.14493 | 55.99 | 00-004-0587; 00-004-0864 |
| 29.5809 | 287.56 | 0.2362 | 3.01991 | 1.12 | |
| 31.2914 | 2330.61 | 0.1181 | 2.85863 | 9.09 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 31.6929 | 25646.05 | 0.1200 | 2.82098 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 31.7474 | 21958.13 | 0.0360 | 2.82326 | 85.62 | |
| 33.2198 | 3955.02 | 0.1320 | 2.69473 | 15.42 | 01-075-1620 |
| 35.1564 | 2945.16 | 0.0960 | 2.55060 | 11.48 | 01-075-1862 |
| 36.0485 | 2972.55 | 0.2640 | 2.48950 | 11.59 | 01-075-1620 |
| 36.9127 | 5525.10 | 0.1080 | 2.43317 | 21.54 | 01-074-1132 |
| 37.7892 | 3070.86 | 0.1680 | 2.37872 | 11.97 | 01-075-1862; 01-075-1620 |
| 37.9298 | 3262.90 | 0.1440 | 2.37023 | 12.72 | 01-075-1620 |
| 38.5343 | 1740.88 | 0.0960 | 2.33443 | 6.79 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.5670 | 9641.07 | 0.1920 | 2.22202 | 37.59 | 00-004-0587 |
| 41.8325 | 908.64 | 0.2880 | 2.15768 | 3.54 | |
| 43.3520 | 3133.37 | 0.1440 | 2.08551 | 12.22 | 01-075-1862; 01-086-2270 |
| 44.8969 | 4257.67 | 0.2880 | 2.01727 | 16.60 | 01-074-1132; 01-085-1327 |
| 45.4331 | 17639.97 | 0.1320 | 1.99470 | 68.78 | 01-077-2064; 01-080-2171; 01-086-2270 |
| 45.5788 | 9306.39 | 0.1080 | 1.99360 | 36.29 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 47.0024 | 605.28 | 0.1920 | 1.93170 | 2.36 | 00-004-0864; 01-086-2270 |
| 47.3326 | 582.18 | 0.1920 | 1.91899 | 2.27 | 01-086-2270 |
| 49.8164 | 1327.97 | 0.2400 | 1.82897 | 5.18 | 01-075-1620; 01-080-2171; 01-080-1385 |
| 50.1951 | 3094.02 | 0.2160 | 1.81606 | 12.06 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235 |
| 52.5588 | 1375.85 | 0.0840 | 1.73981 | 5.36 | 01-075-1862 |
| 53.8567 | 637.98 | 0.1680 | 1.70091 | 2.49 | 01-077-2064 |
| 55.7192 | 624.97 | 0.2880 | 1.64838 | 2.44 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4562 | 5398.76 | 0.1200 | 1.62860 | 21.05 | 01-077-2064 |
| 56.6193 | 3087.71 | 0.0960 | 1.62833 | 12.04 | |
| 57.5101 | 3226.01 | 0.1440 | 1.60123 | 12.58 | 01-075-1862 |
| 57.6588 | 1985.77 | 0.0960 | 1.60143 | 7.74 | |
| 58.6603 | 1582.56 | 0.1080 | 1.57255 | 6.17 | 00-004-0587 |
| 59.3644 | 4135.33 | 0.2880 | 1.55557 | 16.12 | 01-074-1132; 01-075-1620 |
| 60.6178 | 741.92 | 0.3360 | 1.52637 | 2.89 | 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 61.3124 | 535.00 | 0.1920 | 1.51073 | 2.09 | 01-075-1862 |
| 65.2901 | 4394.81 | 0.1200 | 1.42798 | 17.14 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 65.4584 | 4753.23 | 0.1680 | 1.42471 | 18.53 | 01-086-2270 |
| 66.2007 | 5080.68 | 0.0960 | 1.41053 | 19.81 | 00-004-0587; 01-077-2064; 01-075-1620 |
| 66.4188 | 5788.29 | 0.1200 | 1.40642 | 22.57 | 01-075-1862; 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 68.2273 | 1804.82 | 0.0720 | 1.37348 | 7.04 | 01-075-1862; 00-022-1235 |
| 68.4081 | 1115.79 | 0.0720 | 1.37370 | 4.35 | |

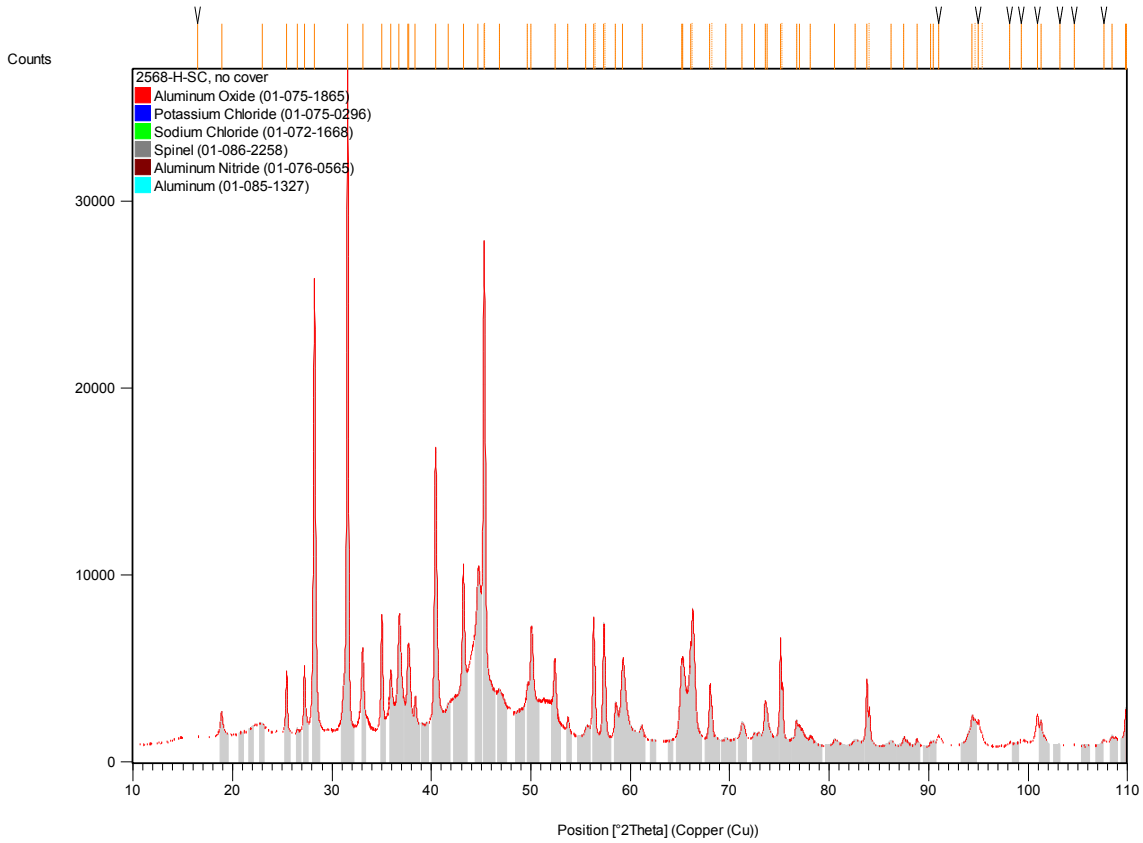
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 69.7277 | 338.80 | 0.1920 | 1.34755 | 1.32 | 01-074-1132; 01-075-1620; 01-080-2171 |
| 71.4000 | 1356.23 | 0.1920 | 1.32004 | 5.29 | 01-075-1620; 01-080-2173; 01-086-2270 |
| 72.5746 | 761.56 | 0.4320 | 1.30154 | 2.97 | 01-075-1620 |
| 73.7344 | 2130.76 | 0.1920 | 1.28391 | 8.31 | 00-004-0587; 00-022-1235 |
| 75.2769 | 5811.09 | 0.1440 | 1.26139 | 22.66 | 01-077-2064 |
| 75.4928 | 3212.94 | 0.1080 | 1.26144 | 12.53 | |
| 76.8908 | 909.25 | 0.0840 | 1.23888 | 3.55 | 01-075-1862; 01-086-2270 |
| 77.4671 | 783.36 | 0.1920 | 1.23110 | 3.05 | 01-074-1132; 01-086-2270 |
| 78.2743 | 601.92 | 0.2880 | 1.22041 | 2.35 | 01-074-1132; 01-085-1327 |
| 80.6936 | 267.57 | 0.1920 | 1.18981 | 1.04 | 01-075-1862 |
| 82.7096 | 416.66 | 0.2880 | 1.16585 | 1.62 | 01-074-1132; 01-086-2270 |
| 83.9588 | 3760.38 | 0.1200 | 1.15165 | 14.66 | 01-077-2064 |
| 84.2094 | 2034.45 | 0.1320 | 1.15172 | 7.93 | |
| 86.4091 | 216.13 | 0.3840 | 1.12517 | 0.84 | 01-075-1862 |
| 87.6448 | 461.06 | 0.1920 | 1.11247 | 1.80 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 88.9982 | 215.51 | 0.1440 | 1.09902 | 0.84 | 01-075-1862 |
| 90.4029 | 250.72 | 0.1440 | 1.08556 | 0.98 | 00-022-1235 |
| 90.6879 | 248.54 | 0.1440 | 1.08289 | 0.97 | |
| 91.1810 | 454.42 | 0.1920 | 1.07831 | 1.77 | |
| 94.5511 | 1751.69 | 0.1680 | 1.04856 | 6.83 | 00-004-0587 |
| 95.2649 | 1104.85 | 0.1200 | 1.04258 | 4.31 | |
| 95.5638 | 541.33 | 0.1440 | 1.04270 | 2.11 | |
| 98.3264 | 232.82 | 0.2880 | 1.01814 | 0.91 | |
| 99.4302 | 339.23 | 0.5760 | 1.00978 | 1.32 | |
| 101.1213 | 1690.63 | 0.1920 | 0.99741 | 6.59 | |
| 101.4956 | 1372.11 | 0.2160 | 0.99474 | 5.35 | 00-004-0587 |
| 104.7758 | 148.96 | 0.6720 | 0.97240 | 0.58 | |
| 107.7752 | 711.57 | 0.1680 | 0.95350 | 2.77 | |
| 108.6391 | 986.76 | 0.1920 | 0.94831 | 3.85 | 00-004-0587 |
| 109.0581 | 852.74 | 0.1920 | 0.94819 | 3.33 | |
| 109.7991 | 1204.45 | 0.0360 | 0.94152 | 4.70 | |

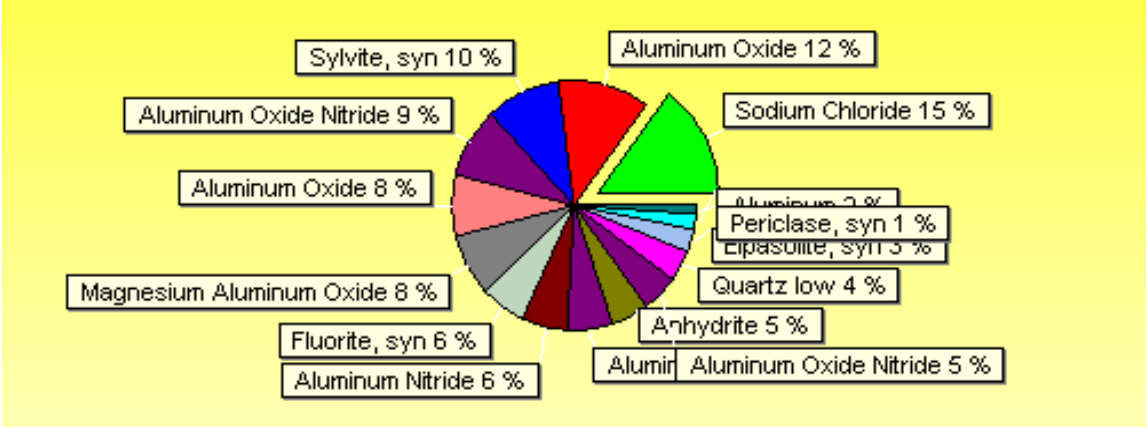
## 6.5: 2568-H-SC

### 6.5.1: Measurement Conditions of 2568-H

| | |
|---|---|
| Dataset Name | 2568-H-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2568-H-SC, no cover.udf |
| Sample Identification | 2568-H-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 6.5.2: Main Graphics, Analyze View of 2568-H


Counts
2568-H-SC, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
30000
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Sylvite, syn 10 %
Aluminum Oxide 12 %
Aluminum Oxide Nitride 9 %
Sodium Chloride 15 %
Aluminum Oxide 8 %
Periclase, syn 1 %
Magnesium Aluminum Oxide 8 %
Quartz low 4 %
Anhydrite 5 %
Fluorite, syn 6 %
Aluminum Oxide Nitride 5 %
Aluminum Nitride 6 %

**6.5.3: Pattern List of 2568-H**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 57 | Aluminum Oxide | Al2 O3 | 12 | 22 | 0 |
| 00-004-0587 | 55 | Sylvite, syn | K Cl | 10 | 10 | 0 |
| 01-077-2064 | 56 | Sodium Chloride | Na Cl | 15 | 9 | 0 |
| 01-074-1132 | 52 | Magnesium Aluminum Oxide | Mg Al2 O4 | 8 | 14 | 0 |
| 01-075-1620 | 50 | Aluminum Nitride | Al N | 6 | 11 | 0 |
| 01-080-2171 | 15 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 9 | 12 | 0 |
| 01-080-2172 | 19 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 18 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 11 | 0 |
| 01-085-1327 | 31 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 8 | Periclase, syn | Mg O | 1 | 5 | 1 |
| 00-004-0864 | 21 | Fluorite, syn | Ca F2 | 6 | 6 | 0 |
| 00-022-1235 | 21 | Elpasolite, syn | K2 Na Al F6 | 3 | 13 | 0 |
| 01-086-2270 | 21 | Anhydrite | Ca ( S O4 ) | 5 | 38 | 0 |
| 01-080-1385 | 18 | Aluminum Oxide | Al2.667 O4 | 8 | 12 | 0 |
| 01-085-0335 | 13 | Quartz low | Si O2 | 4 | 18 | 0 |

**6.5.4: Peak List of 2568-H**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 16.4673 | 44.39 | 0.9446 | 5.38325 | 0.13 | |
| 18.9356 | 1221.41 | 0.0886 | 4.68674 | 3.45 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0051 | 376.57 | 0.4723 | 3.86607 | 1.06 | 01-086-2270 |
| 25.4596 | 3203.47 | 0.1082 | 3.49864 | 9.04 | 01-075-1862; 01-086-2270 |
| 26.4988 | 194.26 | 0.1181 | 3.36375 | 0.55 | 01-085-0335 |
| 27.2284 | 3538.93 | 0.0689 | 3.27524 | 9.99 | 01-077-2064 |
| 28.2244 | 23968.65 | 0.1771 | 3.16189 | 67.65 | 00-004-0587; 00-004-0864 |
| 31.5634 | 35429.42 | 0.1673 | 2.83461 | 100.00 | 01-077-2064 |
| 33.0828 | 4365.47 | 0.1771 | 2.70782 | 12.32 | 01-075-1620 |
| 35.0036 | 6210.84 | 0.1082 | 2.56350 | 17.53 | 01-075-1862 |
| 35.9153 | 3252.96 | 0.1968 | 2.50050 | 9.18 | 01-075-1620 |
| 36.7562 | 6203.02 | 0.0886 | 2.44519 | 17.51 | 01-074-1132; 00-043-1022; 01-085-0335 |
| 37.6375 | 4620.16 | 0.1080 | 2.38796 | 13.04 | 01-075-1862; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 37.7551 | 4509.00 | 0.1574 | 2.38277 | 12.73 | 01-075-1862; 01-075-1620; 01-080-2173 |
| 38.3521 | 1901.62 | 0.0787 | 2.34704 | 5.37 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.3957 | 15139.92 | 0.1320 | 2.23105 | 42.73 | 00-004-0587; 01-085-0335 |
| 40.4563 | 14423.37 | 0.0840 | 2.23339 | 40.71 | |
| 41.6724 | 1590.41 | 0.6720 | 2.16560 | 4.49 | 01-075-1862 |
| 43.1978 | 8970.76 | 0.1560 | 2.09260 | 25.32 | 01-075-1862; 01-086-2270 |
| 44.6970 | 8853.07 | 0.2640 | 2.02583 | 24.99 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.2820 | 25914.17 | 0.1440 | 2.00101 | 73.14 | 01-077-2064; 01-086-2270 |
| 45.4192 | 16931.78 | 0.0840 | 2.00024 | 47.79 | |
| 46.8242 | 2508.25 | 0.2400 | 1.93863 | 7.08 | 00-004-0864; 01-086-2270 |
| 49.6631 | 2784.18 | 0.1440 | 1.83425 | 7.86 | 01-075-1620 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 50.0288 | 5635.59 | 0.0960 | 1.82170 | 15.91 | 00-004-0587; 01-075-1620; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0335 |
| 52.4517 | 3720.19 | 0.0960 | 1.74311 | 10.50 | 01-075-1862; 01-086-2270 |
| 53.7021 | 1085.50 | 0.1200 | 1.70544 | 3.06 | 01-077-2064 |
| 55.5382 | 600.03 | 0.3840 | 1.65332 | 1.69 | 01-074-1132; 00-022-1235; 01-086-2270; 01-085-0335 |
| 56.3024 | 6537.91 | 0.1560 | 1.63268 | 18.45 | 01-077-2064 |
| 56.4781 | 3374.41 | 0.1080 | 1.63206 | 9.52 | |
| 57.3382 | 6189.44 | 0.1440 | 1.60563 | 17.47 | 01-075-1862; 01-085-0335 |
| 57.5095 | 3304.03 | 0.0960 | 1.60523 | 9.33 | |
| 58.5007 | 1854.44 | 0.1680 | 1.57646 | 5.23 | 00-004-0587 |
| 59.1943 | 4063.08 | 0.2880 | 1.55963 | 11.47 | 01-074-1132; 01-075-1620; 00-022-1235; 01-086-2270 |
| 61.1660 | 820.69 | 0.1680 | 1.51399 | 2.32 | 01-075-1862; 01-086-2270 |
| 65.1468 | 4257.08 | 0.2160 | 1.43077 | 12.02 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 65.2642 | 4470.73 | 0.1680 | 1.42848 | 12.62 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 66.0460 | 5251.10 | 0.0960 | 1.41346 | 14.82 | 01-077-2064; 01-075-1620 |
| 66.2314 | 7029.01 | 0.0840 | 1.41345 | 19.84 | |
| 68.0013 | 3029.19 | 0.0960 | 1.37750 | 8.55 | 01-075-1862; 01-085-0335 |
| 68.2341 | 1766.40 | 0.0960 | 1.37677 | 4.99 | |
| 69.6129 | 252.20 | 0.3840 | 1.34949 | 0.71 | 01-074-1132; 01-075-1620 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 71.2617 | 1134.65 | 0.2400 | 1.32226 | 3.20 | 01-075-1620; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 72.4711 | 553.95 | 0.3360 | 1.30315 | 1.56 | 01-075-1620 |
| 73.5321 | 2217.13 | 0.1080 | 1.28694 | 6.26 | 00-004-0587; 00-022-1235; 01-085-0335 |
| 73.7357 | 1905.86 | 0.1440 | 1.28389 | 5.38 | 00-004-0587; 00-022-1235 |
| 75.1005 | 5651.03 | 0.1200 | 1.26391 | 15.95 | 01-077-2064 |
| 75.3105 | 3326.66 | 0.0840 | 1.26404 | 9.39 | |
| 76.7007 | 1282.31 | 0.1440 | 1.24148 | 3.62 | 01-075-1862 |
| 76.9778 | 1046.26 | 0.1920 | 1.23770 | 2.95 | 01-075-1862; 01-086-2270 |
| 78.1130 | 488.41 | 0.2400 | 1.22253 | 1.38 | 01-085-1327; 00-022-1235 |
| 80.5525 | 354.18 | 0.1440 | 1.19154 | 1.00 | 01-075-1862 |
| 82.5717 | 321.53 | 0.5760 | 1.16744 | 0.91 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.7895 | 3555.69 | 0.1440 | 1.15355 | 10.04 | 01-077-2064; 01-085-0335 |
| 84.0487 | 2090.44 | 0.1200 | 1.15351 | 5.90 | |
| 86.2496 | 254.95 | 0.4800 | 1.12684 | 0.72 | 01-075-1862 |
| 87.4887 | 484.63 | 0.1920 | 1.11405 | 1.37 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0335 |
| 88.8070 | 367.88 | 0.1920 | 1.10089 | 1.04 | 01-075-1862; 01-086-2270 |
| 90.1990 | 273.78 | 0.1200 | 1.08748 | 0.77 | 00-022-1235 |
| 90.4952 | 267.49 | 0.1440 | 1.08469 | 0.75 | 00-022-1235 |
| 90.9673 | 517.53 | 0.2880 | 1.08029 | 1.46 | |
| 94.3655 | 1579.54 | 0.2400 | 1.05013 | 4.46 | 00-004-0587; 00-004-0864 |
| 94.6718 | 1401.45 | 0.1440 | 1.05015 | 3.96 | |
| 95.0169 | 1357.42 | 0.1200 | 1.04465 | 3.83 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 95.3872 | 608.19 | 0.1440 | 1.04416 | 1.72 | |
| 98.1651 | 223.76 | 0.3360 | 1.01938 | 0.63 | |
| 99.2892 | 308.68 | 0.4800 | 1.01083 | 0.87 | |
| 100.9263 | 1778.52 | 0.1920 | 0.99881 | 5.02 | |
| 101.2999 | 1414.12 | 0.2160 | 0.99614 | 3.99 | 00-004-0587 |
| 103.2325 | 67.41 | 0.5760 | 0.98269 | 0.19 | |
| 104.6721 | 67.80 | 0.7680 | 0.97308 | 0.19 | |
| 107.5767 | 393.98 | 0.1200 | 0.95471 | 1.11 | |
| 108.4034 | 571.96 | 0.2400 | 0.94972 | 1.61 | 00-004-0587 |
| 109.8079 | 1997.74 | 0.1080 | 0.94147 | 5.64 | 00-043-1022 |
| 109.8921 | 1554.86 | 0.0600 | 0.94098 | 4.39 | 00-043-1022 |

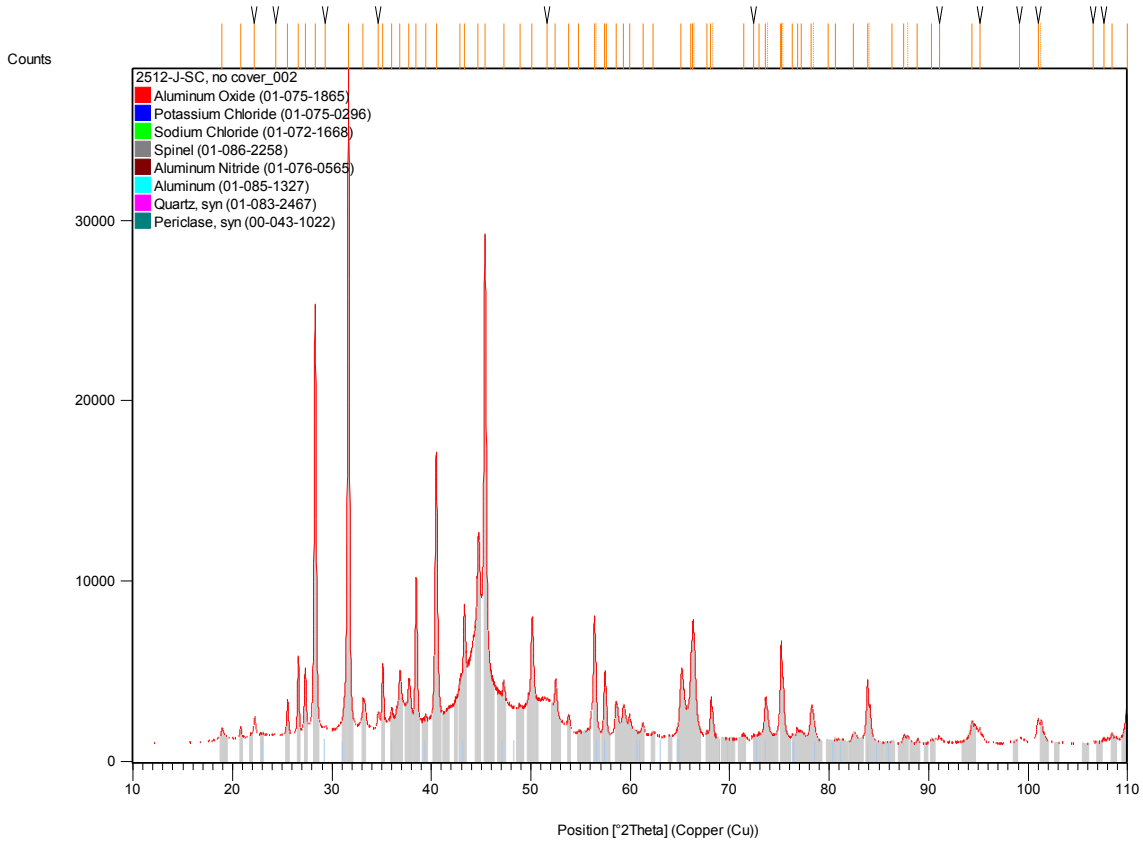
# FACILITY J

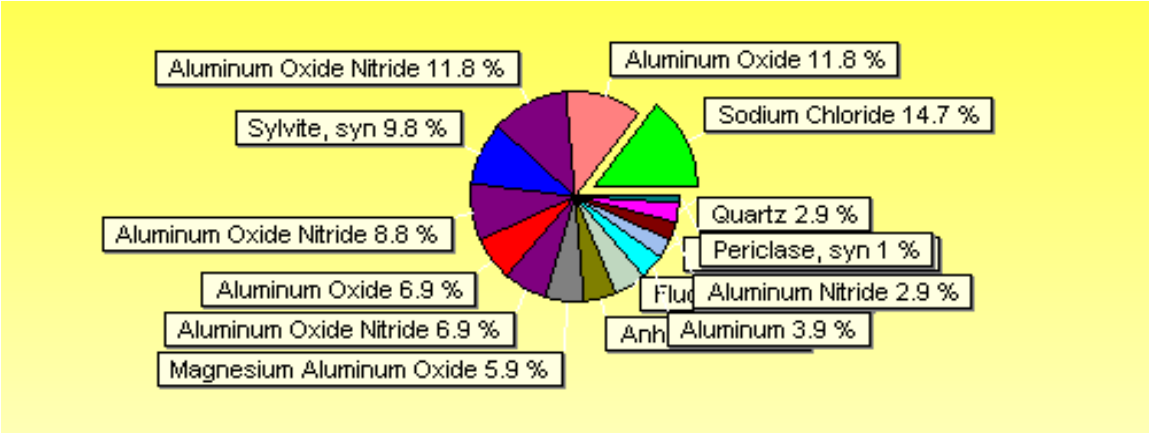
## 7.1: 2512-J-SC

### 7.1.1: Measurement Conditions of 2512-J

| | |
|---|---|
| Dataset Name | 2512-J-SC, no cover_002 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2512-J-SC, no cover_002.udf |
| Sample Identification | 2512-J-SC |
| Comment | Exported by X'Pert SW Generated by todd in project Ft. Devens. |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 7.1.2: Main Graphics, Analyze View of 2512-J



**7.1.3: Pattern List of 2512-J**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 52 | Aluminum Oxide | Al2 O3 | 7 | 19 | 0 |
| 00-004-0587 | 50 | Sylvite, syn | K Cl | 10 | 10 | 0 |
| 01-077-2064 | 55 | Sodium Chloride | Na Cl | 15 | 9 | 0 |
| 01-074-1132 | 37 | Magnesium Aluminum Oxide | Mg Al2 O4 | 6 | 12 | 0 |
| 01-075-1620 | 36 | Aluminum Nitride | Al N | 3 | 9 | 0 |
| 01-080-2171 | 14 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 12 | 13 | 0 |
| 01-080-2172 | 15 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 9 | 11 | 0 |
| 01-080-2173 | 16 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 7 | 11 | 0 |
| 01-085-1327 | 47 | Aluminum | Al | 4 | 5 | 0 |
| 00-043-1022 | 23 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 15 | Fluorite, syn | Ca F2 | 5 | 6 | 0 |
| 00-022-1235 | 19 | Elpasolite, syn | K2 Na Al F6 | 3 | 13 | 0 |
| 01-086-2270 | 15 | Anhydrite | Ca ( S O4 ) | 5 | 37 | 0 |
| 01-080-1385 | 16 | Aluminum Oxide | Al2.667 O4 | 12 | 13 | 0 |
| 01-085-0930 | 31 | Quartz | Si O2 | 3 | 22 | 0 |

**7.1.4: Peak List of 2512-J**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9425 | 733.50 | 0.1378 | 4.68506 | 1.98 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.8213 | 811.17 | 0.1181 | 4.26635 | 2.19 | 01-085-0930 |
| 22.2139 | 1338.68 | 0.0787 | 4.00194 | 3.62 | |
| 24.3813 | 477.10 | 0.2362 | 3.65087 | 1.29 | |
| 25.5309 | 2210.65 | 0.0787 | 3.48903 | 5.97 | 01-075-1862; 01-086-2270 |
| 26.5923 | 4548.01 | 0.1181 | 3.35213 | 12.28 | 01-085-0930 |
| 27.3301 | 3970.43 | 0.1181 | 3.26329 | 10.72 | 01-077-2064 |
| 28.3034 | 24120.18 | 0.1476 | 3.15325 | 65.15 | 00-004-0587; 00-004-0864 |
| 29.3339 | 819.89 | 0.2362 | 3.04478 | 2.21 | |
| 31.6478 | 37021.99 | 0.1476 | 2.82724 | 100.00 | 01-077-2064 |
| 33.1353 | 2269.23 | 0.0984 | 2.70364 | 6.13 | 01-075-1620 |
| 34.6286 | 1627.65 | 0.1574 | 2.59040 | 4.40 | |
| 35.0924 | 4218.03 | 0.0689 | 2.55722 | 11.39 | 01-075-1862 |
| 36.0066 | 1788.66 | 0.1771 | 2.49436 | 4.83 | 01-075-1620 |
| 36.8275 | 3809.62 | 0.1574 | 2.44062 | 10.29 | 01-074-1132; 00-043-1022 |
| 37.7277 | 3374.33 | 0.1378 | 2.38444 | 9.11 | 01-075-1862 |
| 38.4594 | 8915.70 | 0.1279 | 2.34074 | 24.08 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 39.4240 | 1376.23 | 0.1574 | 2.28566 | 3.72 | 01-080-2173; 01-085-0930 |
| 40.4741 | 15997.41 | 0.1378 | 2.22875 | 43.21 | 00-004-0587 |
| 42.8928 | 3401.98 | 0.1574 | 2.10852 | 9.19 | 00-043-1022 |
| 43.3001 | 7273.68 | 0.0886 | 2.08962 | 19.65 | 01-075-1862; 01-086-2270 |
| 44.7024 | 11195.21 | 0.1378 | 2.02727 | 30.24 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3670 | 27708.14 | 0.1378 | 1.99911 | 74.84 | 01-077-2064; 01-086-2270 |
| 47.2702 | 3303.69 | 0.1181 | 1.92297 | 8.92 | 01-086-2270 |
| 48.8972 | 1920.46 | 0.2362 | 1.86272 | 5.19 | 00-022-1235 |
| 50.1007 | 6703.75 | 0.0689 | 1.82076 | 18.11 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 51.6643 | 2276.05 | 0.5510 | 1.76927 | 6.15 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 52.4761 | 3329.06 | 0.1378 | 1.74380 | 8.99 | 01-075-1862 |
| 53.7826 | 1417.74 | 0.1181 | 1.70448 | 3.83 | 01-077-2064 |
| 54.7964 | 518.85 | 0.2362 | 1.67532 | 1.40 | 01-085-0930 |
| 56.3891 | 6818.51 | 0.1560 | 1.63038 | 18.42 | 01-077-2064 |
| 56.5476 | 4197.83 | 0.0960 | 1.63022 | 11.34 | |
| 57.4017 | 3703.19 | 0.0480 | 1.60400 | 10.00 | 01-075-1862; 01-085-0930 |
| 57.5798 | 2438.05 | 0.0960 | 1.59946 | 6.59 | 01-075-1862 |
| 58.5600 | 2084.93 | 0.0960 | 1.57500 | 5.63 | 00-004-0587 |
| 59.2876 | 1887.44 | 0.2160 | 1.55740 | 5.10 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 59.9195 | 1393.98 | 0.1200 | 1.54248 | 3.77 | 01-085-0930 |
| 61.2472 | 911.96 | 0.1200 | 1.51218 | 2.46 | 01-075-1862; 01-086-2270 |
| 62.2415 | 416.56 | 0.1920 | 1.49039 | 1.13 | 00-043-1022; 01-086-2270 |
| 65.0860 | 3455.60 | 0.2640 | 1.43196 | 9.33 | 01-074-1132; 01-085-1327 |
| 66.1172 | 4830.12 | 0.0720 | 1.41211 | 13.05 | 01-077-2064; 01-075-1620 |
| 66.2918 | 6550.74 | 0.1440 | 1.40881 | 17.69 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171 |
| 66.4728 | 4979.74 | 0.1200 | 1.40890 | 13.45 | |
| 67.6591 | 414.18 | 0.1440 | 1.38363 | 1.12 | 01-085-0930 |
| 68.0802 | 2082.19 | 0.1200 | 1.37609 | 5.62 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.2982 | 1422.63 | 0.0960 | 1.37564 | 3.84 | |
| 71.3757 | 325.47 | 0.3360 | 1.32043 | 0.88 | 01-075-1620; 01-080-2173; 01-086-2270 |
| 72.3942 | 238.96 | 0.3840 | 1.30434 | 0.65 | |
| 72.9608 | 400.13 | 0.1440 | 1.29561 | 1.08 | 01-077-2064; 01-086-2270 |
| 73.5923 | 2289.08 | 0.1680 | 1.28604 | 6.18 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 73.8147 | 1534.18 | 0.1440 | 1.28590 | 4.14 | |
| 75.1408 | 5224.82 | 0.0600 | 1.26333 | 14.11 | 01-077-2064 |
| 75.1936 | 5563.04 | 0.0480 | 1.26258 | 15.03 | 01-077-2064 |
| 75.3792 | 3425.78 | 0.0840 | 1.26306 | 9.25 | |
| 76.2842 | 333.33 | 0.1440 | 1.24722 | 0.90 | 01-075-1620 |
| 76.7761 | 704.68 | 0.1200 | 1.24045 | 1.90 | 01-075-1862 |

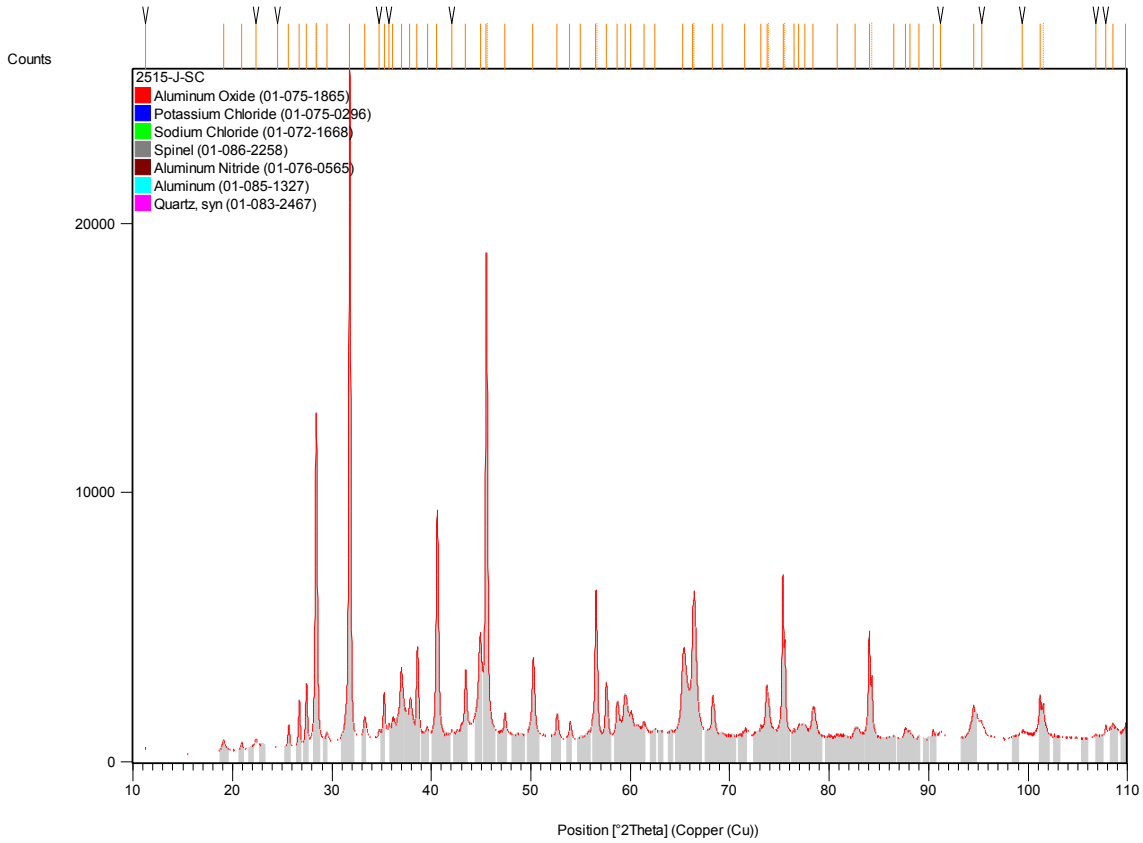
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 77.2033 | 512.86 | 0.3840 | 1.23465 | 1.39 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.1629 | 1758.52 | 0.2400 | 1.22187 | 4.75 | 01-085-1327 |
| 78.4547 | 1339.85 | 0.1680 | 1.22108 | 3.62 | |
| 79.8518 | 86.09 | 0.2880 | 1.20022 | 0.23 | 01-080-2171; 01-086-2270; 01-080-1385; 01-085-0930 |
| 80.5893 | 203.23 | 0.1440 | 1.19109 | 0.55 | 01-075-1862 |
| 82.4251 | 553.78 | 0.1920 | 1.16915 | 1.50 | 01-085-1327 |
| 83.8912 | 3340.41 | 0.1680 | 1.15241 | 9.02 | 01-077-2064; 01-085-0930 |
| 84.0820 | 2122.09 | 0.0840 | 1.15314 | 5.73 | |
| 86.3208 | 122.73 | 0.3840 | 1.12610 | 0.33 | 01-075-1862 |
| 87.5155 | 463.14 | 0.2400 | 1.11378 | 1.25 | 00-004-0587; 01-080-2171; 01-080-2172; 00-004-0864; 01-080-1385; 01-085-0930 |
| 87.9410 | 379.80 | 0.1920 | 1.11224 | 1.03 | |
| 88.8665 | 228.24 | 0.1440 | 1.10030 | 0.62 | 01-075-1862; 01-086-2270 |
| 90.2618 | 256.10 | 0.1440 | 1.08689 | 0.69 | 00-022-1235 |
| 91.0466 | 351.22 | 0.1920 | 1.07955 | 0.95 | |
| 94.3651 | 1167.46 | 0.2160 | 1.05014 | 3.15 | 00-004-0587; 00-004-0864 |
| 95.1416 | 782.36 | 0.1680 | 1.04361 | 2.11 | |
| 99.0923 | 275.79 | 0.5760 | 1.01231 | 0.74 | |
| 101.0075 | 1368.78 | 0.0960 | 0.99823 | 3.70 | |
| 101.3011 | 1290.49 | 0.1680 | 0.99860 | 3.49 | |
| 106.5669 | 109.66 | 0.5760 | 0.96095 | 0.30 | |
| 107.6149 | 343.72 | 0.1920 | 0.95448 | 0.93 | |
| 108.4030 | 581.79 | 0.1680 | 0.94972 | 1.57 | 00-004-0587 |
| 109.9386 | 1570.04 | 0.0840 | 0.94071 | 4.24 | 00-043-1022 |

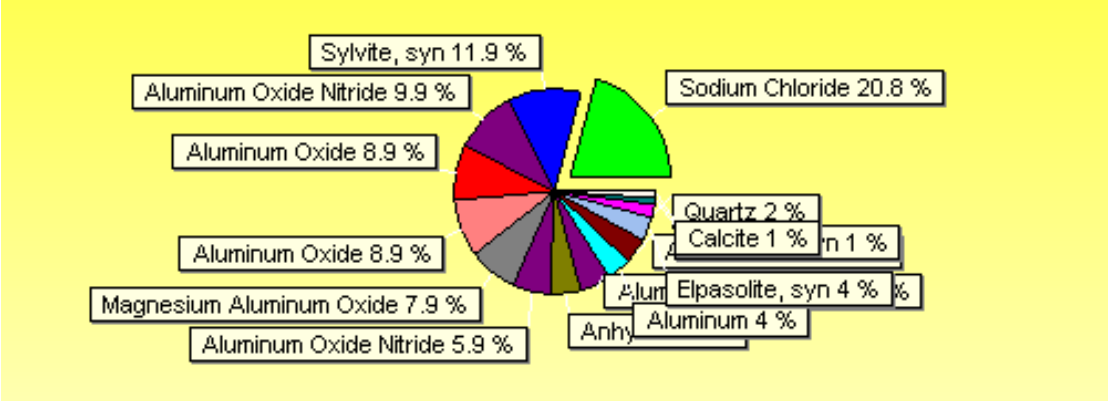
## 7.2: 2515-J-SC

### 7.2:1: Measurement Conditions of 2515-J

| | |
|---|---|
| Dataset Name | 2515-J-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2515-J-SC.udf |
| Sample Identification | 2515-J-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 7.2.2: Main Graphics, Analyze View of 2515-J


Counts
2515-J-SC
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Quartz, syn (01-083-2467)
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Sylvite, syn 11.9 %
Aluminum Oxide Nitride 9.9 %
Sodium Chloride 20.8 %
Aluminum Oxide 8.9 %
Quartz 2 %
Calcite 1 %
Aluminum Oxide 8.9 %
Elpasolite, syn 4 %
Magnesium Aluminum Oxide 7.9 %
Aluminum 4 %
Aluminum Oxide Nitride 5.9 %

**7.2.3: Pattern List of 2515-J**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 55 | Aluminum Oxide | Al2 O3 | 9 | 20 | 0 |
| 00-004-0587 | 43 | Sylvite, syn | K Cl | 12 | 10 | 0 |
| 01-077-2064 | 48 | Sodium Chloride | Na Cl | 21 | 9 | 0 |
| 01-074-1132 | 44 | Magnesium Aluminum Oxide | Mg Al2 O4 | 8 | 12 | 0 |
| 01-075-1620 | 35 | Aluminum Nitride | Al N | 4 | 8 | 0 |
| 01-080-2171 | 11 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 10 | 11 | 0 |
| 01-080-2172 | 14 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 11 | 0 |
| 01-080-2173 | 17 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 12 | 0 |
| 01-085-1327 | 40 | Aluminum | Al | 4 | 5 | 0 |
| 00-043-1022 | 7 | Periclase, syn | Mg O | 1 | 6 | 0 |
| 00-022-1235 | 24 | Elpasolite, syn | K2 Na Al F6 | 4 | 14 | 0 |
| 01-086-2270 | 20 | Anhydrite | Ca ( S O4 ) | 5 | 33 | 0 |
| 01-080-1385 | 14 | Aluminum Oxide | Al2.667 O4 | 9 | 11 | 0 |
| 01-086-2340 | 18 | Calcite | Ca ( C O3 ) | 1 | 17 | 0 |
| 01-085-0930 | 23 | Quartz | Si O2 | 2 | 20 | 0 |

**7.2.4: Peak List of 2515-J**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 11.2362 | 152.77 | 0.7872 | 7.87493 | 0.61 | |
| 19.0814 | 472.96 | 0.2362 | 4.65126 | 1.90 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.9154 | 366.23 | 0.1378 | 4.24737 | 1.47 | 01-085-0930 |
| 22.3293 | 537.92 | 0.0886 | 3.98151 | 2.16 | |
| 24.4889 | 203.42 | 0.3149 | 3.63507 | 0.82 | |
| 25.6375 | 993.07 | 0.0787 | 3.47476 | 3.99 | 01-075-1862; 01-086-2270 |
| 26.6921 | 1883.33 | 0.0787 | 3.33982 | 7.56 | 01-085-0930 |
| 27.4594 | 2468.88 | 0.1279 | 3.24822 | 9.91 | 01-077-2064 |
| 28.4280 | 12463.55 | 0.1560 | 3.13710 | 50.05 | 00-004-0587; 01-086-2270 |
| 28.4912 | 9561.35 | 0.0480 | 3.13808 | 38.40 | |
| 29.4775 | 702.72 | 0.1680 | 3.02776 | 2.82 | 01-086-2340 |
| 31.7656 | 24900.67 | 0.2280 | 2.81469 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 33.2680 | 1214.76 | 0.1680 | 2.69094 | 4.88 | 01-075-1620 |
| 34.7448 | 768.27 | 0.1920 | 2.57986 | 3.09 | |
| 35.2639 | 2108.40 | 0.1320 | 2.54307 | 8.47 | 01-075-1862 |
| 35.7137 | 938.09 | 0.1920 | 2.51207 | 3.77 | |
| 36.1128 | 1182.65 | 0.2400 | 2.48521 | 4.75 | 01-075-1620; 01-086-2270; 01-086-2340 |
| 36.9769 | 3030.78 | 0.1080 | 2.42909 | 12.17 | 01-074-1132; 00-043-1022 |
| 37.8437 | 1916.15 | 0.1680 | 2.37543 | 7.70 | 01-075-1862; 01-075-1620 |
| 38.5469 | 3420.15 | 0.1560 | 2.33370 | 13.74 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 39.5676 | 815.15 | 0.1920 | 2.27582 | 3.27 | 01-086-2340; 01-085-0930 |
| 40.5550 | 8595.17 | 0.1920 | 2.22265 | 34.52 | 00-004-0587 |
| 42.0552 | 626.22 | 0.3840 | 2.14677 | 2.51 | |
| 43.4416 | 2902.02 | 0.1320 | 2.08142 | 11.65 | 01-075-1862; 01-086-2270 |
| 44.9197 | 4240.69 | 0.2640 | 2.01630 | 17.03 | 01-074-1132; 01-085-1327 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 45.5022 | 17807.90 | 0.1560 | 1.99183 | 71.52 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 45.6223 | 12998.78 | 0.0840 | 1.99181 | 52.20 | |
| 47.4019 | 1299.42 | 0.1680 | 1.91634 | 5.22 | 01-086-2270 |
| 50.1908 | 3083.67 | 0.0840 | 1.81620 | 12.38 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.6021 | 1206.79 | 0.1920 | 1.73848 | 4.85 | 01-075-1862; 01-086-2270 |
| 53.9244 | 908.69 | 0.1200 | 1.69893 | 3.65 | 01-077-2064 |
| 54.9751 | 354.25 | 0.2880 | 1.66892 | 1.42 | 01-085-0930 |
| 56.5191 | 5691.75 | 0.1440 | 1.62693 | 22.86 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385; 01-086-2340 |
| 56.6884 | 3628.94 | 0.0960 | 1.62651 | 14.57 | |
| 57.5447 | 2254.77 | 0.0960 | 1.60035 | 9.06 | 01-075-1862; 01-086-2340 |
| 58.6596 | 1588.71 | 0.1920 | 1.57257 | 6.38 | 00-004-0587 |
| 59.4596 | 1910.69 | 0.2160 | 1.55330 | 7.67 | 01-075-1862; 01-074-1132; 01-075-1620 |
| 60.0654 | 1219.87 | 0.2400 | 1.53908 | 4.90 | 01-085-0930 |
| 61.3697 | 829.34 | 0.3360 | 1.50946 | 3.33 | 01-075-1862 |
| 62.4473 | 554.52 | 0.2880 | 1.48597 | 2.23 | 00-043-1022; 01-086-2270 |
| 65.2945 | 3315.82 | 0.1920 | 1.42789 | 13.32 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 66.2636 | 4880.26 | 0.1080 | 1.40934 | 19.60 | 01-075-1862; 00-004-0587; 01-077-2064; 01-075-1620; 01-080-2171; 01-080-1385 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 66.4360 | 5642.53 | 0.0720 | 1.40960 | 22.66 | |
| 68.2613 | 1780.96 | 0.0840 | 1.37288 | 7.15 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 69.2154 | 385.51 | 0.2880 | 1.35627 | 1.55 | 00-022-1235; 01-086-2340 |
| 71.5169 | 477.42 | 0.6720 | 1.31817 | 1.92 | 01-075-1620; 01-086-2270 |
| 73.1179 | 614.70 | 0.1440 | 1.29321 | 2.47 | 01-077-2064 |
| 73.7198 | 2075.25 | 0.1680 | 1.28413 | 8.33 | 00-004-0587; 00-022-1235; 01-086-2340 |
| 73.9256 | 1468.36 | 0.1440 | 1.28425 | 5.90 | |
| 75.3495 | 6167.73 | 0.1560 | 1.26035 | 24.77 | 01-077-2064; 01-080-2171 |
| 75.5686 | 3532.98 | 0.1200 | 1.26036 | 14.19 | |
| 76.4353 | 486.28 | 0.1440 | 1.24513 | 1.95 | 01-075-1620; 01-086-2340 |
| 76.9280 | 654.93 | 0.1440 | 1.23838 | 2.63 | 01-075-1862; 01-086-2270 |
| 77.5337 | 587.00 | 0.4800 | 1.23021 | 2.36 | 01-074-1132; 01-086-2270; 01-085-0930 |
| 78.3612 | 1278.55 | 0.1440 | 1.21928 | 5.13 | 01-074-1132; 01-085-1327; 00-043-1022; 01-086-2270; 01-086-2340 |
| 80.8035 | 228.82 | 0.2880 | 1.18847 | 0.92 | 01-075-1862; 01-075-1620 |
| 82.5605 | 417.49 | 0.4320 | 1.16758 | 1.68 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 84.0238 | 4061.48 | 0.1320 | 1.15093 | 16.31 | 01-075-1862; 01-077-2064; 01-086-2270; 01-086-2340; 01-085-0930 |
| 84.2884 | 2339.44 | 0.1080 | 1.15084 | 9.40 | |
| 86.4853 | 118.61 | 0.3840 | 1.12438 | 0.48 | 01-075-1862 |
| 87.6209 | 398.02 | 0.1920 | 1.11271 | 1.60 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0930 |

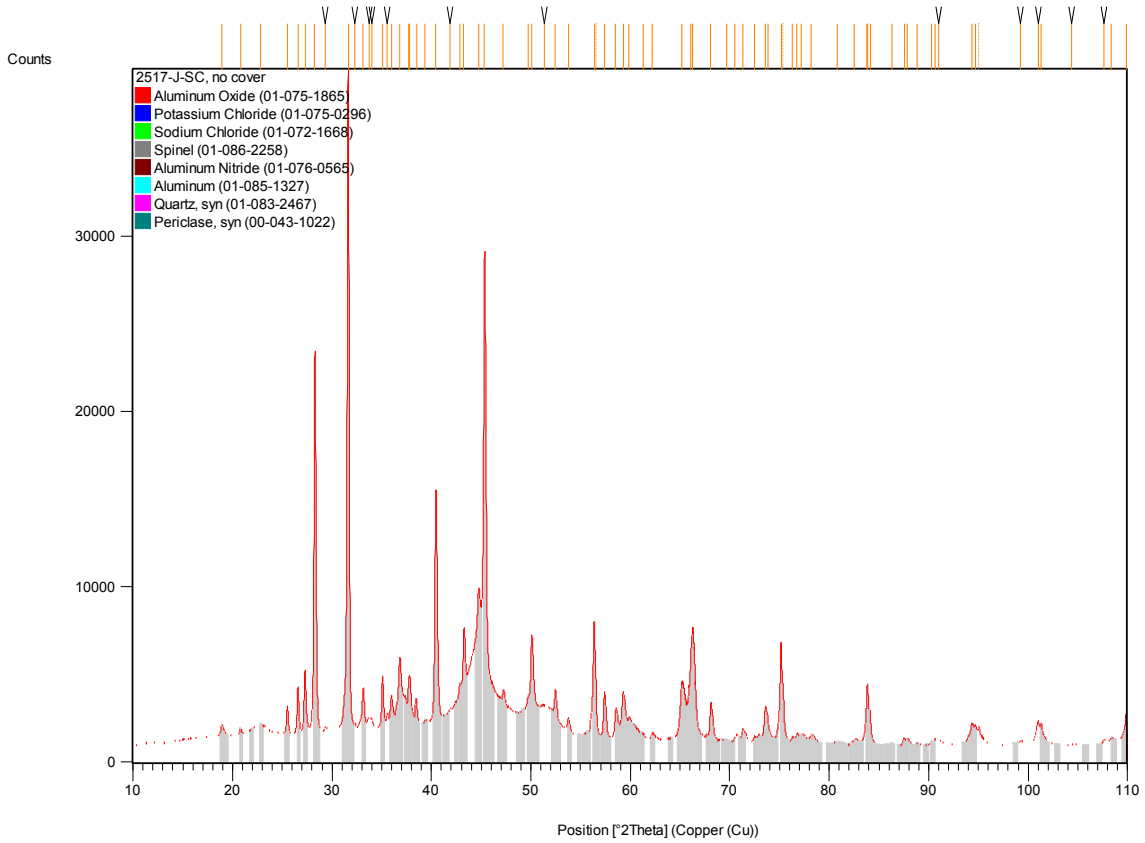
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 88.0863 | 319.14 | 0.1920 | 1.10803 | 1.28 | 01-086-2270 |
| 89.0270 | 139.71 | 0.2400 | 1.09874 | 0.56 | 01-075-1862 |
| 90.4466 | 345.01 | 0.1440 | 1.08515 | 1.39 | 00-022-1235 |
| 91.2174 | 249.14 | 0.2400 | 1.07798 | 1.00 | |
| 94.5138 | 1163.37 | 0.1680 | 1.04888 | 4.67 | 00-004-0587 |
| 95.2953 | 641.66 | 0.1440 | 1.04233 | 2.58 | |
| 99.4425 | 250.40 | 0.5760 | 1.00969 | 1.01 | |
| 101.1670 | 1475.46 | 0.1440 | 0.99709 | 5.93 | 00-004-0587 |
| 101.5251 | 1221.61 | 0.1920 | 0.99701 | 4.91 | |
| 106.8023 | 264.85 | 0.5760 | 0.95948 | 1.06 | |
| 107.8112 | 692.97 | 0.2160 | 0.95328 | 2.78 | |
| 108.5503 | 854.81 | 0.2400 | 0.94884 | 3.43 | 00-004-0587 |
| 109.8275 | 1150.60 | 0.0360 | 0.94135 | 4.62 | 00-043-1022 |

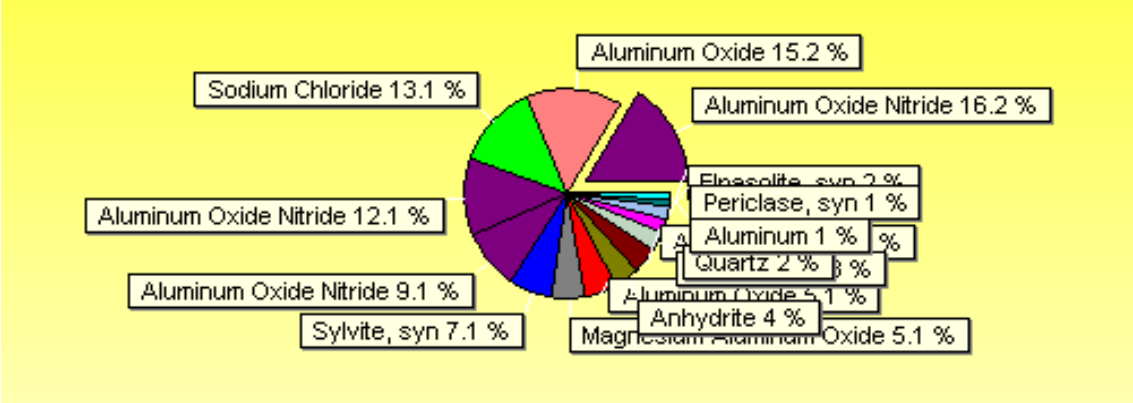
## 7.3: 2517-J-SC

### 7.3.1: Measurement Conditions of 2517-J

| | |
|---|---|
| Dataset Name | 2517-J-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2517-J-SC, no cover.udf |
| Sample Identification | 2517-J-SC, NO COVER |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 7.3.2: Main Graphics, Analyze View of 2517-J


Counts
2517-J-SC, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Quartz, syn (01-083-2467)
Periclase, syn (00-043-1022)
30000
20000
10000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Aluminum Oxide 15.2 %
Sodium Chloride 13.1 %
Aluminum Oxide Nitride 16.2 %
Periclase, syn 1 %
Aluminum Oxide Nitride 12.1 %
Aluminum 1 %
Quartz 2 %
Aluminum Oxide Nitride 9.1 %
Anhydrite 4 %
Sylvite, syn 7.1 %
Oxide 5.1 %

**7.3.3: Pattern List of 2517-J**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 39 | Aluminum Oxide | Al2 O3 | 5 | 19 | 0 |
| 00-004-0587 | 47 | Sylvite, syn | K Cl | 7 | 10 | 0 |
| 01-077-2064 | 54 | Sodium Chloride | Na Cl | 13 | 9 | 0 |
| 01-074-1132 | 39 | Magnesium Aluminum Oxide | Mg Al2 O4 | 5 | 13 | 0 |
| 01-075-1620 | 44 | Aluminum Nitride | Al N | 4 | 11 | 0 |
| 01-080-2171 | 16 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 16 | 13 | 0 |
| 01-080-2172 | 17 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 12 | 12 | 0 |
| 01-080-2173 | 18 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 9 | 12 | 0 |
| 01-085-1327 | 23 | Aluminum | Al | 1 | 5 | 0 |
| 00-043-1022 | 27 | Periclase, syn | Mg O | 1 | 6 | 0 |
| 00-004-0864 | 12 | Fluorite, syn | Ca F2 | 3 | 4 | 0 |
| 00-022-1235 | 15 | Elpasolite, syn | K2 Na Al F6 | 2 | 13 | 0 |
| 01-086-2270 | 12 | Anhydrite | Ca ( S O4 ) | 4 | 38 | 0 |
| 01-080-1385 | 17 | Aluminum Oxide | Al2.667 O4 | 15 | 12 | 0 |
| 01-085-0930 | 27 | Quartz | Si O2 | 2 | 21 | 0 |

**7.3.4: Peak List of 2517-J**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9184 | 799.87 | 0.1181 | 4.69096 | 2.08 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.7936 | 575.99 | 0.1574 | 4.27196 | 1.50 | 01-085-0930 |
| 22.7664 | 826.15 | 0.4723 | 3.90605 | 2.15 | 01-086-2270 |
| 25.5271 | 1828.19 | 0.0787 | 3.48954 | 4.76 | 01-075-1862; 01-086-2270 |
| 26.5663 | 2980.30 | 0.1082 | 3.35536 | 7.76 | 01-085-0930 |
| 27.2928 | 3963.47 | 0.0984 | 3.26766 | 10.32 | 01-077-2064 |
| 28.2597 | 21338.74 | 0.1574 | 3.15803 | 55.57 | 00-004-0587; 00-004-0864 |
| 29.3011 | 737.34 | 0.1378 | 3.04811 | 1.92 | |
| 31.6249 | 38403.13 | 0.1870 | 2.82924 | 100.00 | 01-077-2064 |
| 32.3243 | 975.42 | 0.1181 | 2.76960 | 2.54 | |
| 33.1117 | 2840.36 | 0.0590 | 2.70552 | 7.40 | 01-075-1620 |
| 33.7248 | 1275.91 | 0.2362 | 2.65772 | 3.32 | |
| 34.0521 | 1087.06 | 0.1574 | 2.63293 | 2.83 | |
| 35.0865 | 3731.25 | 0.0787 | 2.55763 | 9.72 | 01-075-1862 |
| 35.5643 | 1571.47 | 0.1181 | 2.52436 | 4.09 | |
| 35.9866 | 2510.86 | 0.1378 | 2.49570 | 6.54 | 01-075-1620 |
| 36.8281 | 4674.30 | 0.2165 | 2.44059 | 12.17 | 01-074-1132; 00-043-1022 |
| 37.7099 | 3379.84 | 0.1200 | 2.38355 | 8.80 | 01-075-1862; 01-080-2173 |
| 37.8408 | 3558.44 | 0.1574 | 2.37757 | 9.27 | 01-075-1862; 01-075-1620 |
| 38.4941 | 2335.85 | 0.1968 | 2.33871 | 6.08 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 39.3737 | 1173.46 | 0.1574 | 2.28847 | 3.06 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0930 |
| 40.4120 | 13312.09 | 0.1870 | 2.23203 | 34.66 | 00-004-0587; 01-085-0930 |
| 41.8673 | 1815.96 | 0.2362 | 2.15776 | 4.73 | |
| 42.8452 | 3234.29 | 0.1574 | 2.11075 | 8.42 | 00-043-1022 |
| 43.2394 | 6225.85 | 0.0590 | 2.09241 | 16.21 | 01-075-1862; 01-086-2270 |
| 44.7328 | 8580.70 | 0.1181 | 2.02596 | 22.34 | 01-074-1132; 01-085-1327; 00-022-1235 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 45.3419 | 27654.29 | 0.0886 | 2.00016 | 72.01 | 01-077-2064; 01-086-2270 |
| 47.2083 | 2933.97 | 0.0984 | 1.92534 | 7.64 | 00-004-0864; 01-086-2270 |
| 49.7122 | 2603.08 | 0.1181 | 1.83408 | 6.78 | 01-075-1620 |
| 50.0580 | 5724.11 | 0.0886 | 1.82221 | 14.91 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 51.3941 | 2049.87 | 0.4723 | 1.77794 | 5.34 | |
| 52.4489 | 2962.26 | 0.1378 | 1.74464 | 7.71 | 01-075-1862; 01-086-2270 |
| 53.7564 | 1363.96 | 0.1181 | 1.70525 | 3.55 | 01-077-2064 |
| 56.3639 | 6909.97 | 0.1680 | 1.63105 | 17.99 | 01-077-2064 |
| 56.5468 | 3501.15 | 0.0960 | 1.63024 | 9.12 | |
| 57.4036 | 2878.14 | 0.1200 | 1.60395 | 7.49 | 01-075-1862; 01-085-0930 |
| 58.5260 | 1828.41 | 0.1920 | 1.57584 | 4.76 | 00-004-0587 |
| 59.2823 | 2880.40 | 0.2160 | 1.55752 | 7.50 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 59.8733 | 1467.03 | 0.2400 | 1.54356 | 3.82 | 01-085-0930 |
| 61.2501 | 537.44 | 0.2880 | 1.51212 | 1.40 | 01-075-1862; 01-086-2270 |
| 62.2015 | 536.68 | 0.3360 | 1.49125 | 1.40 | 00-043-1022; 01-086-2270 |
| 65.1483 | 3270.57 | 0.2160 | 1.43074 | 8.52 | 01-074-1132; 01-085-1327 |
| 66.0946 | 5309.74 | 0.1200 | 1.41253 | 13.83 | 01-077-2064; 01-075-1620 |
| 66.2745 | 6533.08 | 0.2160 | 1.40913 | 17.01 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171 |
| 68.0825 | 2286.81 | 0.1680 | 1.37605 | 5.95 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 69.6647 | 213.23 | 0.2880 | 1.34862 | 0.56 | 01-074-1132; 01-075-1620 |
| 70.5276 | 384.81 | 0.4800 | 1.33422 | 1.00 | 01-075-1862 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 71.2977 | 809.88 | 0.1680 | 1.32168 | 2.11 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 72.4845 | 373.23 | 0.1440 | 1.30294 | 0.97 | 01-075-1620 |
| 73.6062 | 2137.21 | 0.0960 | 1.28583 | 5.57 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 73.8180 | 1522.59 | 0.1680 | 1.28267 | 3.96 | 00-004-0587; 00-022-1235 |
| 75.1600 | 5830.71 | 0.1320 | 1.26306 | 15.18 | 01-077-2064 |
| 75.3769 | 3207.02 | 0.1200 | 1.26309 | 8.35 | |
| 76.2942 | 440.15 | 0.1440 | 1.24708 | 1.15 | 01-075-1620 |
| 76.7544 | 628.36 | 0.1440 | 1.24075 | 1.64 | 01-075-1862 |
| 77.1914 | 551.53 | 0.4800 | 1.23481 | 1.44 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.1696 | 547.72 | 0.3360 | 1.22178 | 1.43 | 01-085-1327; 00-022-1235 |
| 80.7655 | 139.20 | 0.9600 | 1.18893 | 0.36 | 01-075-1862 |
| 82.4755 | 283.31 | 0.2880 | 1.16856 | 0.74 | 01-074-1132; 01-085-1327 |
| 83.8120 | 3258.39 | 0.0840 | 1.15330 | 8.48 | 01-077-2064; 01-085-0930 |
| 83.8903 | 3244.46 | 0.0600 | 1.15242 | 8.45 | 01-077-2064; 01-085-0930 |
| 84.0974 | 1938.17 | 0.1440 | 1.15011 | 5.05 | 01-075-1862; 01-077-2064; 01-080-2171; 01-086-2270; 01-085-0930 |
| 86.2753 | 93.41 | 0.5760 | 1.12657 | 0.24 | 01-075-1862 |
| 87.5589 | 392.11 | 0.1680 | 1.11333 | 1.02 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 87.8621 | 350.31 | 0.2880 | 1.11027 | 0.91 | 00-004-0587; 01-080-2172; 01-080-2173 |
| 88.8417 | 119.61 | 0.5760 | 1.10055 | 0.31 | 01-075-1862; 01-086-2270 |
| 90.2449 | 219.83 | 0.1440 | 1.08705 | 0.57 | 00-022-1235 |
| 90.6372 | 288.02 | 0.1920 | 1.08336 | 0.75 | 00-022-1235 |

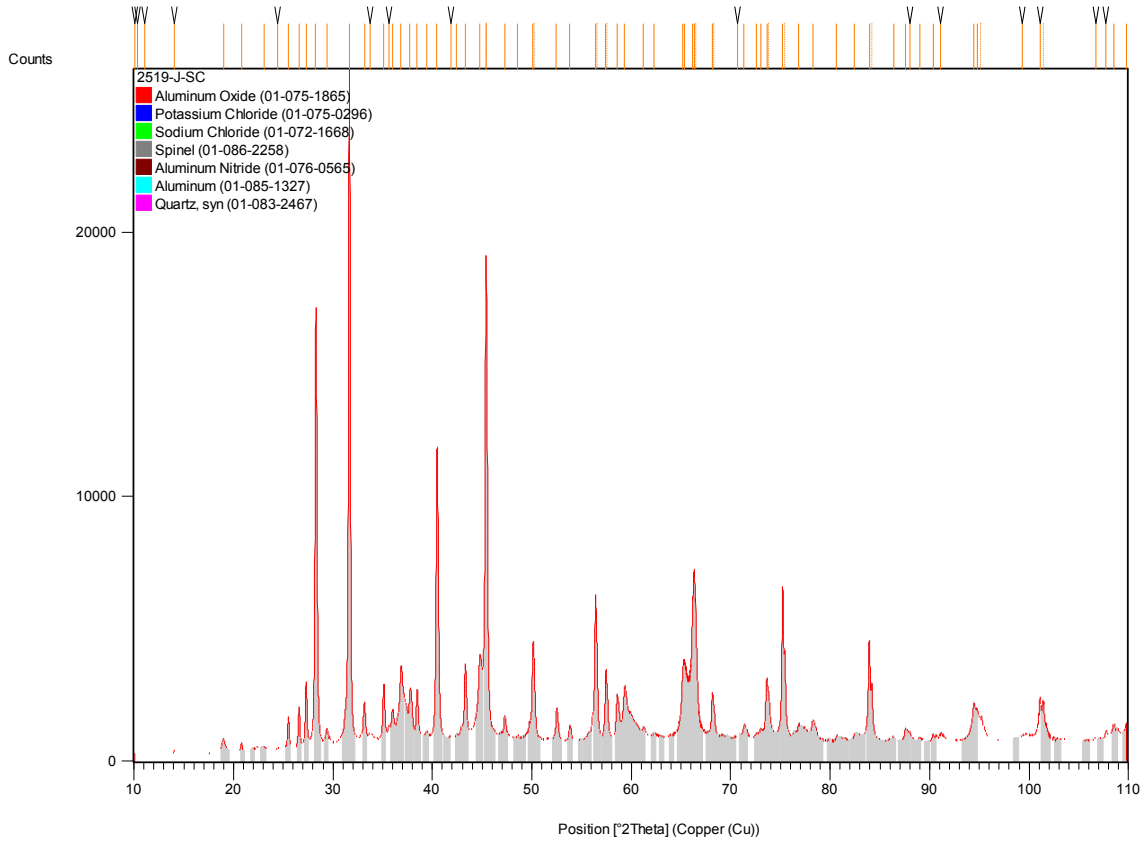
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 91.0246 | 317.52 | 0.2400 | 1.07976 | 0.83 | |
| 94.3065 | 1102.09 | 0.1920 | 1.05063 | 2.87 | 00-004-0587; 00-004-0864 |
| 94.7096 | 1011.67 | 0.1920 | 1.04722 | 2.63 | 00-004-0587 |
| 95.0684 | 914.64 | 0.1680 | 1.04681 | 2.38 | |
| 99.2305 | 227.06 | 0.4800 | 1.01127 | 0.59 | |
| 100.9912 | 1384.47 | 0.1920 | 0.99835 | 3.61 | |
| 101.3026 | 1233.72 | 0.2160 | 0.99612 | 3.21 | 00-004-0587 |
| 104.3879 | 40.82 | 0.9600 | 0.97495 | 0.11 | |
| 107.6243 | 326.94 | 0.2400 | 0.95442 | 0.85 | |
| 108.3281 | 442.56 | 0.2400 | 0.95017 | 1.15 | 00-004-0587 |
| 109.9130 | 1694.51 | 0.0720 | 0.94086 | 4.41 | 00-043-1022 |

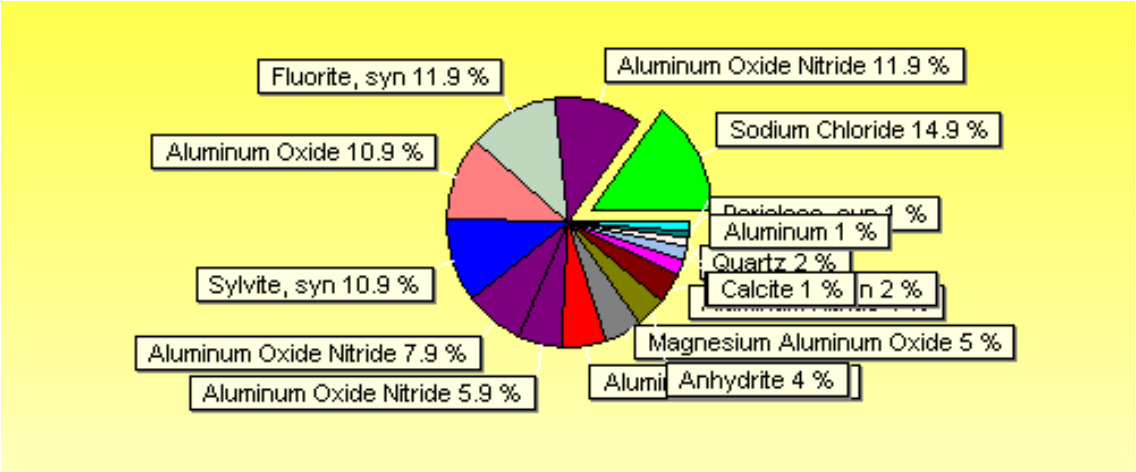
## 7.4: 2519-J-SC

### 7.4.1: Measurement Conditions of 2519-J

| | |
|---|---|
| Dataset Name | 2519-J-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2519-J-SC.udf |
| Sample Identification | 2519-J-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 7.4.2: Main Graphics, Analyze View of 2519-J




**7.4.3: Pattern List of 2519-J**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 46 | Aluminum Oxide | Al2 O3 | 6 | 19 | 0 |
| 00-004-0587 | 51 | Sylvite, syn | K Cl | 11 | 10 | 0 |
| 01-077-2064 | 56 | Sodium Chloride | Na Cl | 15 | 9 | 0 |
| 01-074-1132 | 46 | Magnesium Aluminum Oxide | Mg Al2 O4 | 5 | 12 | 0 |
| 01-075-1620 | 36 | Aluminum Nitride | Al N | 4 | 9 | 0 |
| 01-080-2171 | 13 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 12 | 11 | 0 |
| 01-080-2172 | 14 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 8 | 11 | 0 |
| 01-080-2173 | 16 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 6 | 12 | 0 |
| 01-085-1327 | 38 | Aluminum | Al | 1 | 5 | 0 |
| 00-043-1022 | 7 | Periclase, syn | Mg O | 1 | 6 | 0 |
| 00-004-0864 | 13 | Fluorite, syn | Ca F2 | 12 | 4 | 1 |
| 00-022-1235 | 18 | Elpasolite, syn | K2 Na Al F6 | 2 | 13 | 0 |
| 01-086-2270 | 18 | Anhydrite | Ca ( S O4 ) | 4 | 35 | 0 |
| 01-080-1385 | 14 | Aluminum Oxide | Al2.667 O4 | 11 | 11 | 0 |
| 01-086-2340 | 17 | Calcite | Ca ( C O3 ) | 1 | 18 | 0 |
| 01-085-0930 | 30 | Quartz | Si O2 | 2 | 20 | 0 |

**7.4.4: Peak List of 2519-J**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.0589 | 74.52 | 0.0295 | 8.79389 | 0.29 | |
| 10.3355 | 131.55 | 0.6298 | 8.55912 | 0.51 | |
| 11.0537 | 145.67 | 0.4723 | 8.00458 | 0.56 | |
| 14.0181 | 138.97 | 0.0787 | 6.31781 | 0.54 | |
| 18.9755 | 540.80 | 0.1574 | 4.67697 | 2.09 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.7948 | 392.52 | 0.0787 | 4.27173 | 1.52 | 01-085-0930 |
| 23.1205 | 183.58 | 0.3936 | 3.84703 | 0.71 | 01-086-2340 |
| 24.4491 | 164.99 | 0.2362 | 3.64090 | 0.64 | |
| 25.5029 | 1274.00 | 0.0787 | 3.49280 | 4.93 | 01-075-1862; 01-086-2270 |
| 26.5860 | 1658.84 | 0.1181 | 3.35291 | 6.42 | 01-085-0930 |
| 27.2905 | 2547.92 | 0.1181 | 3.26794 | 9.86 | 01-077-2064 |
| 28.2729 | 16122.50 | 0.1968 | 3.15657 | 62.39 | 00-004-0587; 00-004-0864 |
| 29.3783 | 835.55 | 0.1771 | 3.04027 | 3.23 | 01-086-2340 |
| 31.6538 | 25840.60 | 0.1771 | 2.82672 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.1804 | 1842.89 | 0.0689 | 2.70007 | 7.13 | 01-075-1620 |
| 33.7617 | 665.47 | 0.2362 | 2.65491 | 2.58 | |
| 35.0862 | 2478.00 | 0.0886 | 2.55766 | 9.59 | 01-075-1862 |
| 35.5961 | 890.16 | 0.1181 | 2.52218 | 3.44 | |
| 36.0358 | 1543.28 | 0.1574 | 2.49241 | 5.97 | 01-075-1620; 01-086-2340 |
| 36.8206 | 3046.15 | 0.2165 | 2.44107 | 11.79 | 01-074-1132; 00-043-1022 |
| 37.7333 | 2221.08 | 0.1771 | 2.38410 | 8.60 | 01-075-1862; 01-075-1620; 01-080-2173 |
| 38.4496 | 2267.76 | 0.0886 | 2.34131 | 8.78 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 39.4381 | 713.30 | 0.1574 | 2.28488 | 2.76 | 01-080-2172; 01-080-2173; 01-086-2340; 01-085-0930 |
| 40.4406 | 10893.05 | 0.1378 | 2.23052 | 42.15 | 00-004-0587; 01-085-0930 |
| 41.8457 | 507.82 | 0.2362 | 2.15882 | 1.97 | |
| 42.4193 | 588.06 | 0.1181 | 2.13094 | 2.28 | 01-085-0930 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 43.2989 | 3201.20 | 0.1476 | 2.08968 | 12.39 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 44.7914 | 3554.39 | 0.2362 | 2.02345 | 13.76 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3740 | 17926.42 | 0.1560 | 1.99716 | 69.37 | 01-077-2064; 01-086-2270 |
| 45.5202 | 11370.81 | 0.1200 | 1.99603 | 44.00 | |
| 47.2660 | 1246.66 | 0.1320 | 1.92153 | 4.82 | 01-086-2270; 01-086-2340 |
| 48.5280 | 462.21 | 0.2880 | 1.87447 | 1.79 | 01-086-2270; 01-086-2340 |
| 50.1059 | 3932.55 | 0.1200 | 1.81908 | 15.22 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 50.2401 | 3221.71 | 0.0960 | 1.81904 | 12.47 | |
| 52.4744 | 1415.53 | 0.1320 | 1.74241 | 5.48 | 01-075-1862 |
| 53.8050 | 861.25 | 0.1320 | 1.70242 | 3.33 | 01-077-2064 |
| 56.3999 | 5682.39 | 0.1200 | 1.63009 | 21.99 | 01-077-2064 |
| 56.5717 | 3491.35 | 0.1200 | 1.62958 | 13.51 | |
| 57.4337 | 2838.22 | 0.0960 | 1.60318 | 10.98 | 01-075-1862; 01-086-2340; 01-085-0930 |
| 57.6194 | 1809.91 | 0.0960 | 1.60243 | 7.00 | |
| 58.5791 | 1954.50 | 0.1680 | 1.57453 | 7.56 | 00-004-0587 |
| 59.3218 | 2207.87 | 0.1920 | 1.55658 | 8.54 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 61.2377 | 726.51 | 0.2880 | 1.51239 | 2.81 | 01-075-1862; 01-086-2270 |
| 62.3222 | 476.57 | 0.3840 | 1.48866 | 1.84 | 00-043-1022; 01-086-2270 |
| 65.2034 | 2957.03 | 0.2160 | 1.42967 | 11.44 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 65.3264 | 3196.61 | 0.1680 | 1.42727 | 12.37 | 01-074-1132; 01-086-2270 |
| 66.1630 | 5434.16 | 0.0960 | 1.41124 | 21.03 | 01-077-2064; 01-075-1620 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 66.3308 | 6568.07 | 0.1200 | 1.40808 | 25.42 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 66.4859 | 5042.50 | 0.0960 | 1.40866 | 19.51 | |
| 68.1512 | 1933.61 | 0.1680 | 1.37483 | 7.48 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.3450 | 1314.26 | 0.1200 | 1.37481 | 5.09 | |
| 71.3431 | 740.76 | 0.1680 | 1.32095 | 2.87 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270 |
| 72.5487 | 405.80 | 0.2880 | 1.30195 | 1.57 | 01-075-1620; 01-086-2340 |
| 73.0472 | 616.47 | 0.1200 | 1.29429 | 2.39 | 01-077-2064; 01-086-2270 |
| 73.6339 | 2517.82 | 0.1560 | 1.28542 | 9.74 | 00-004-0587; 00-022-1235; 01-086-2340; 01-085-0930 |
| 73.8609 | 1728.37 | 0.1440 | 1.28521 | 6.69 | |
| 75.2254 | 5915.75 | 0.1440 | 1.26212 | 22.89 | 01-077-2064 |
| 75.4408 | 3469.29 | 0.0960 | 1.26218 | 13.43 | |
| 76.8376 | 741.59 | 0.1680 | 1.23961 | 2.87 | 01-075-1862 |
| 78.2565 | 875.16 | 0.3360 | 1.22065 | 3.39 | 01-074-1132; 01-085-1327 |
| 80.6446 | 265.91 | 0.1440 | 1.19041 | 1.03 | 01-075-1862; 01-086-2340 |
| 82.4473 | 320.77 | 0.2880 | 1.16889 | 1.24 | 01-074-1132; 01-085-1327 |
| 83.9140 | 3814.05 | 0.1320 | 1.15215 | 14.76 | 01-077-2064; 01-086-2340; 01-085-0930 |
| 84.1926 | 2192.54 | 0.1440 | 1.15191 | 8.48 | |
| 86.3569 | 200.27 | 0.1920 | 1.12572 | 0.78 | 01-075-1862 |
| 87.5595 | 527.34 | 0.1680 | 1.11333 | 2.04 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| | | | | | 01-080-1385; 01-085-0930 |
| 88.0079 | 399.67 | 0.2400 | 1.10881 | 1.55 | |
| 88.9746 | 181.31 | 0.1920 | 1.09925 | 0.70 | 01-075-1862 |
| 90.3288 | 256.14 | 0.1680 | 1.08625 | 0.99 | 00-022-1235 |
| 91.1338 | 323.33 | 0.1920 | 1.07875 | 1.25 | |
| 94.4244 | 1360.72 | 0.1920 | 1.04963 | 5.27 | 00-004-0587; 00-004-0864 |
| 94.7518 | 1155.00 | 0.1920 | 1.04687 | 4.47 | 00-004-0587 |
| 95.1873 | 918.07 | 0.1680 | 1.04582 | 3.55 | |
| 99.3439 | 208.49 | 0.3840 | 1.01042 | 0.81 | |
| 101.0843 | 1554.98 | 0.1080 | 0.99768 | 6.02 | |
| 101.4370 | 1388.63 | 0.1680 | 0.99763 | 5.37 | |
| 106.6915 | 301.22 | 0.1440 | 0.96017 | 1.17 | |
| 107.7243 | 706.79 | 0.2640 | 0.95381 | 2.74 | |
| 108.5223 | 1026.38 | 0.2400 | 0.94901 | 3.97 | 00-004-0587 |
| 109.7716 | 1199.51 | 0.1320 | 0.94168 | 4.64 | 00-043-1022 |

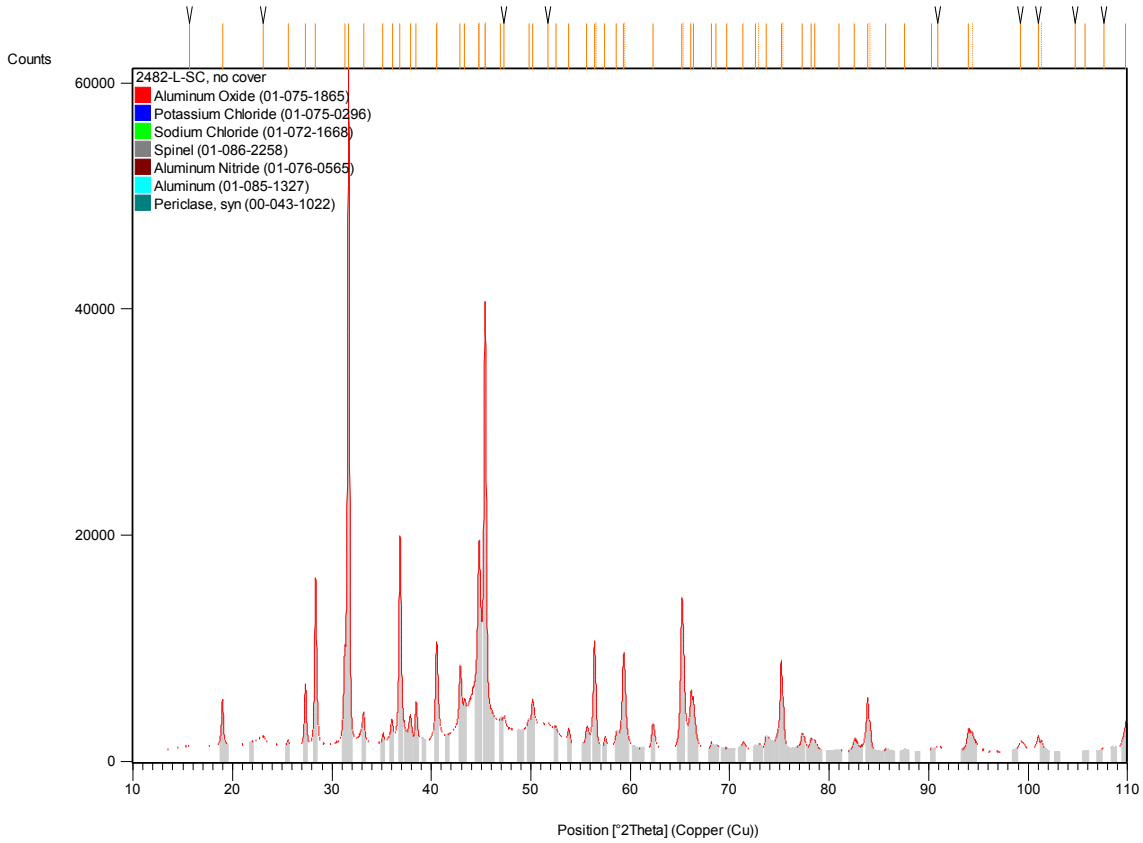
# FACILITY L

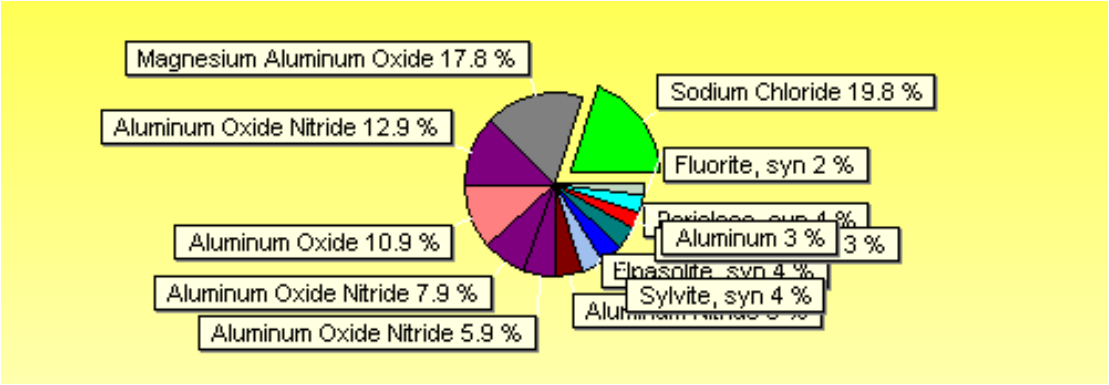
## 8.1: 2482-L-SC

### 8.1.1: Measurement Conditions of 2482-L

| | |
|---|---|
| Dataset Name | 2482-L-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2482-L-SC, no cover.sd |
| Sample Identification | 2482-L-Sc, no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 9/20/2010 10:56:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

**8.1.2: Main Graphics, Analyze View of 2482-L**



**8.1.3: Pattern List of 2482-L**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 34 | Aluminum Oxide | Al2 O3 | 3 | 15 | 0 |
| 00-004-0587 | 52 | Sylvite, syn | K Cl | 4 | 10 | 0 |
| 01-077-2064 | 61 | Sodium Chloride | Na Cl | 20 | 9 | 0 |
| 01-074-1132 | 61 | Magnesium Aluminum Oxide | Mg Al2 O4 | 18 | 15 | 0 |
| 01-075-1620 | 45 | Aluminum Nitride | Al N | 5 | 11 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 13 | 11 | 0 |
| 01-080-2172 | 15 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 8 | 12 | 0 |
| 01-080-2173 | 15 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 6 | 12 | 0 |
| 01-085-1327 | 31 | Aluminum | Al | 3 | 5 | 0 |
| 00-043-1022 | 42 | Periclase, syn | Mg O | 4 | 8 | 0 |
| 00-004-0864 | 30 | Fluorite, syn | Ca F2 | 2 | 6 | 0 |
| 00-022-1235 | 26 | Elpasolite, syn | K2 Na Al F6 | 4 | 16 | 0 |
| 01-080-1385 | 16 | Aluminum Oxide | Al2.667 O4 | 11 | 12 | 0 |

**8.1.4: Peak List of 2482-L**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 15.6736 | 155.17 | 0.2362 | 5.65403 | 0.26 | |
| 18.9830 | 4080.36 | 0.1082 | 4.67514 | 6.81 | 01-074-1132; 00-022-1235 |
| 23.1067 | 637.18 | 0.1574 | 3.84929 | 1.06 | |
| 25.5651 | 433.66 | 0.1574 | 3.48443 | 0.72 | 01-075-1862 |
| 27.3381 | 5445.36 | 0.1279 | 3.26235 | 9.09 | 01-077-2064 |
| 28.3316 | 14807.44 | 0.1378 | 3.15017 | 24.72 | 00-004-0587; 00-004-0864 |
| 31.2570 | 8015.88 | 0.0787 | 2.86169 | 13.38 | 01-074-1132; 00-022-1235 |
| 31.6772 | 59892.18 | 0.1870 | 2.82468 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.1793 | 3023.05 | 0.0984 | 2.70016 | 5.05 | 01-075-1620 |
| 35.1020 | 1126.58 | 0.1574 | 2.55654 | 1.88 | 01-075-1862 |
| 36.0588 | 2233.93 | 0.1771 | 2.49087 | 3.73 | 01-075-1620 |
| 36.8138 | 17746.47 | 0.1574 | 2.44150 | 29.63 | 01-074-1132; 00-043-1022 |
| 37.8800 | 2783.95 | 0.1968 | 2.37520 | 4.65 | 01-075-1862; 01-075-1620 |
| 38.4495 | 3893.46 | 0.0787 | 2.34132 | 6.50 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.4744 | 8586.98 | 0.1440 | 2.22689 | 14.34 | 00-004-0587 |
| 40.5663 | 8843.97 | 0.0840 | 2.22758 | 14.77 | |
| 42.8845 | 7138.10 | 0.0960 | 2.10716 | 11.92 | 00-043-1022 |
| 43.3042 | 4201.50 | 0.0960 | 2.08770 | 7.02 | 01-075-1862 |
| 44.7704 | 18157.76 | 0.1440 | 2.02268 | 30.32 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 44.8247 | 18264.11 | 0.0360 | 2.02537 | 30.49 | |
| 45.3908 | 39429.00 | 0.1800 | 1.99646 | 65.83 | 01-077-2064 |
| 45.5041 | 26127.38 | 0.0720 | 1.99670 | 43.62 | |
| 46.9674 | 2629.51 | 0.1920 | 1.93305 | 4.39 | 00-004-0864 |
| 47.3013 | 2885.08 | 0.1920 | 1.92018 | 4.82 | |
| 49.7867 | 2514.96 | 0.1920 | 1.82999 | 4.20 | 01-075-1620; 01-080-2171 |
| 50.1628 | 4238.78 | 0.1920 | 1.81715 | 7.08 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 51.7282 | 2168.48 | 0.4800 | 1.76578 | 3.62 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 52.4871 | 1909.65 | 0.2400 | 1.74202 | 3.19 | 01-075-1862 |
| 53.7696 | 1657.34 | 0.1440 | 1.70345 | 2.77 | 01-077-2064 |
| 55.5881 | 1780.70 | 0.2400 | 1.65196 | 2.97 | 01-074-1132; 00-004-0864 |
| 56.3880 | 9573.32 | 0.1440 | 1.63040 | 15.98 | 01-077-2064 |
| 56.5619 | 5150.39 | 0.0960 | 1.62984 | 8.60 | |
| 57.4468 | 1026.94 | 0.1920 | 1.60285 | 1.71 | 01-075-1862 |
| 58.5654 | 1459.87 | 0.1920 | 1.57487 | 2.44 | 00-004-0587 |
| 59.2919 | 8260.74 | 0.1080 | 1.55730 | 13.79 | 01-074-1132; 01-075-1620 |
| 59.4898 | 5856.39 | 0.0720 | 1.55644 | 9.78 | |
| 62.2363 | 2214.11 | 0.2640 | 1.49050 | 3.70 | 00-043-1022 |
| 65.1970 | 13452.39 | 0.1800 | 1.42979 | 22.46 | 01-074-1132; 01-085-1327 |
| 65.3791 | 8913.20 | 0.1440 | 1.42979 | 14.88 | |
| 66.1023 | 5125.14 | 0.0960 | 1.41239 | 8.56 | 01-077-2064; 01-075-1620 |
| 66.3335 | 4553.34 | 0.1440 | 1.40802 | 7.60 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 68.1430 | 661.29 | 0.1680 | 1.37498 | 1.10 | 01-075-1862; 00-022-1235 |
| 68.5901 | 491.88 | 0.2880 | 1.36710 | 0.82 | 01-074-1132; 00-004-0864 |
| 69.7148 | 156.04 | 0.2880 | 1.34777 | 0.26 | 01-074-1132; 01-075-1620; 01-080-2171 |
| 71.3356 | 701.26 | 0.1920 | 1.32108 | 1.17 | 01-075-1620; 01-080-2172; 01-080-2173 |
| 72.5980 | 460.46 | 0.3360 | 1.30118 | 0.77 | 01-075-1620 |
| 72.9769 | 613.53 | 0.1440 | 1.29858 | 1.02 | |
| 73.6244 | 1267.78 | 0.1680 | 1.28556 | 2.12 | 00-004-0587; 00-022-1235 |
| 75.1836 | 8017.12 | 0.1320 | 1.26272 | 13.39 | 01-077-2064 |
| 75.3930 | 4370.18 | 0.1320 | 1.26286 | 7.30 | |
| 77.2562 | 1553.01 | 0.1920 | 1.23393 | 2.59 | 01-075-1862; 01-074-1132 |
| 78.1756 | 1113.56 | 0.1920 | 1.22171 | 1.86 | 01-085-1327; 00-022-1235 |
| 78.5565 | 912.08 | 0.1920 | 1.21673 | 1.52 | 01-074-1132; 00-043-1022 |
| 80.9314 | 74.50 | 0.6720 | 1.18691 | 0.12 | 01-075-1620 |

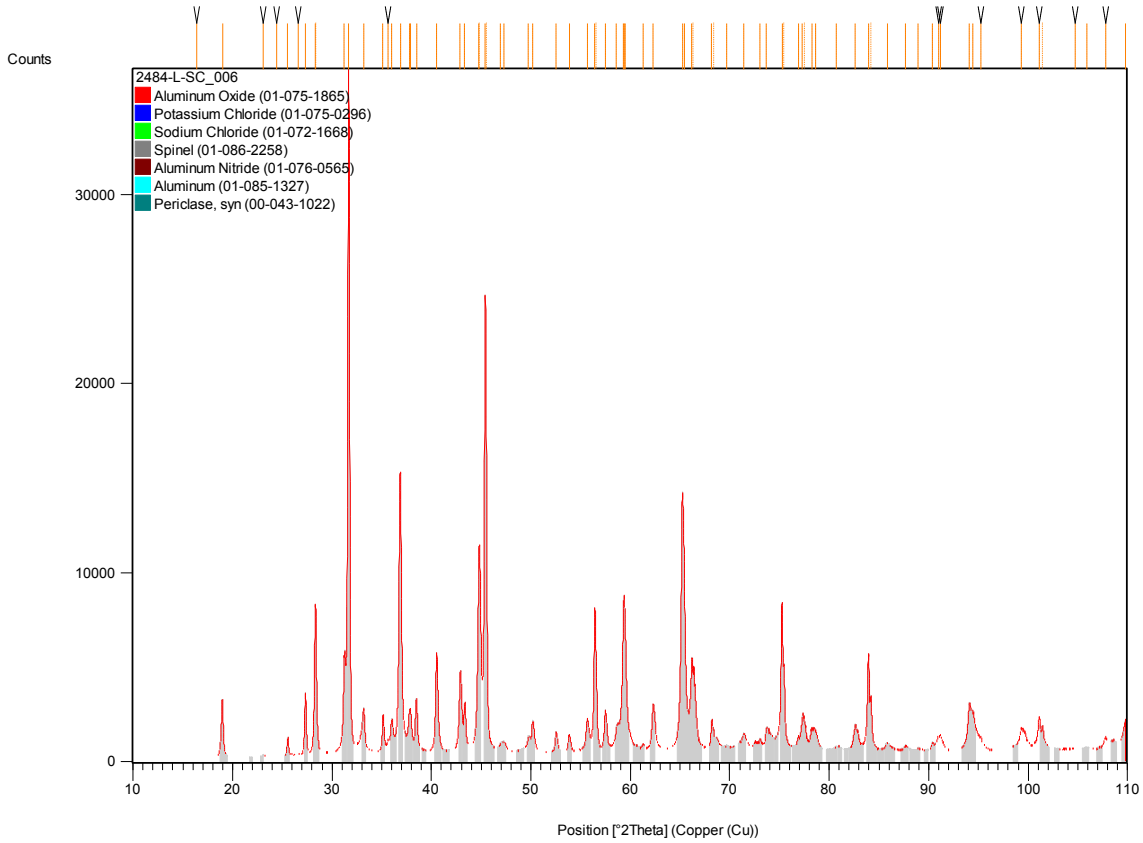
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 82.5515 | 1158.94 | 0.1440 | 1.16768 | 1.94 | 01-074-1132; 01-085-1327 |
| 83.8645 | 4744.69 | 0.1920 | 1.15271 | 7.92 | 01-077-2064 |
| 84.1308 | 2453.10 | 0.1440 | 1.15259 | 4.10 | |
| 85.6938 | 294.34 | 0.1440 | 1.13272 | 0.49 | 01-074-1132; 01-075-1620 |
| 87.5711 | 244.70 | 0.1920 | 1.11321 | 0.41 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385 |
| 90.2642 | 368.27 | 0.1920 | 1.08686 | 0.61 | 00-022-1235 |
| 90.8797 | 592.62 | 0.2400 | 1.08110 | 0.99 | |
| 93.9884 | 1906.23 | 0.2880 | 1.05335 | 3.18 | 00-043-1022; 00-004-0864 |
| 94.3951 | 1527.01 | 0.2400 | 1.05249 | 2.55 | |
| 99.2439 | 920.27 | 0.2880 | 1.01117 | 1.54 | |
| 101.0082 | 1440.77 | 0.0840 | 0.99822 | 2.41 | |
| 101.3682 | 976.67 | 0.1920 | 0.99812 | 1.63 | |
| 104.7107 | 60.50 | 0.5760 | 0.97283 | 0.10 | |
| 105.7555 | 86.68 | 0.5760 | 0.96607 | 0.14 | 00-043-1022; 00-004-0864 |
| 107.6329 | 597.58 | 0.2400 | 0.95437 | 1.00 | |
| 109.8329 | 2743.73 | 0.0960 | 0.94132 | 4.58 | 00-043-1022 |

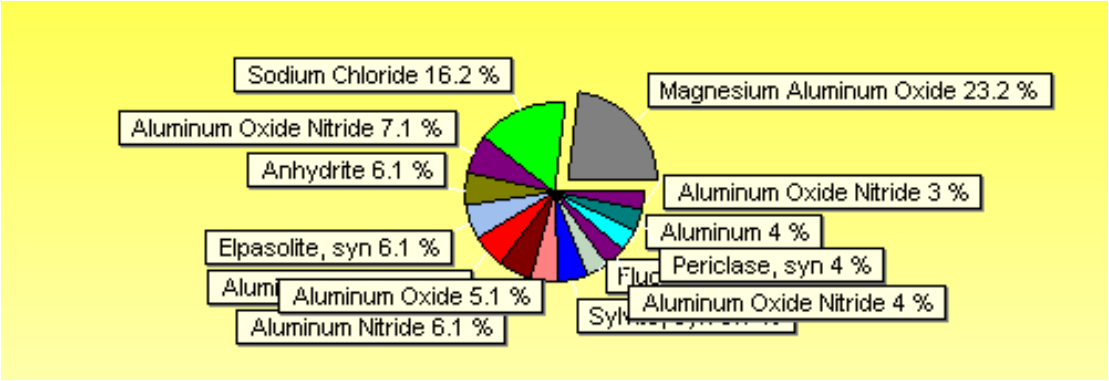
## 8.2: 2484-L-SC

### 8.2.1: Measurement Conditions of 2484-L

| | |
|---|---|
| Dataset Name | 2484-L-SC_006 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2484-L-SC_006.sd |
| Sample Identification | 2484-L-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 8/5/2010 6:54:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

**8.2.2: Main Graphics, Analyze View of 2484-L**



**8.2.3:Pattern List of 2484-L**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 50 | Aluminum Oxide | Al2 O3 | 6 | 20 | 0 |
| 00-004-0587 | 39 | Sylvite, syn | K Cl | 5 | 10 | 0 |
| 01-077-2064 | 41 | Sodium Chloride | Na Cl | 16 | 9 | 0 |
| 01-074-1132 | 50 | Magnesium Aluminum Oxide | Mg Al2 O4 | 23 | 14 | 0 |
| 01-075-1620 | 34 | Aluminum Nitride | Al N | 6 | 10 | 0 |
| 01-080-2171 | 13 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 7 | 12 | 0 |
| 01-080-2172 | 14 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 4 | 12 | 0 |
| 01-080-2173 | 15 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 3 | 12 | 0 |
| 01-085-1327 | 31 | Aluminum | Al | 4 | 5 | 0 |
| 00-043-1022 | 34 | Periclase, syn | Mg O | 4 | 8 | 0 |
| 00-004-0864 | 24 | Fluorite, syn | Ca F2 | 4 | 6 | 0 |
| 00-022-1235 | 32 | Elpasolite, syn | K2 Na Al F6 | 6 | 17 | 0 |
| 01-086-2270 | 22 | Anhydrite | Ca ( S O4 ) | 6 | 37 | 0 |
| 01-080-1385 | 14 | Aluminum Oxide | Al2.667 O4 | 5 | 12 | 0 |

**8.2.4: Peak List of 2484-L**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 16.3933 | 29.20 | 0.2362 | 5.40737 | 0.08 | |
| 19.0042 | 2935.13 | 0.1279 | 4.66998 | 8.07 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0943 | 127.55 | 0.1574 | 3.85133 | 0.35 | |
| 24.4679 | 89.84 | 0.1181 | 3.63814 | 0.25 | |
| 25.5611 | 1011.78 | 0.1181 | 3.48498 | 2.78 | 01-075-1862; 01-086-2270 |
| 26.6202 | 160.21 | 0.1181 | 3.34868 | 0.44 | |
| 27.3502 | 3393.97 | 0.0492 | 3.26094 | 9.33 | 01-077-2064 |
| 28.3146 | 7823.07 | 0.1080 | 3.14942 | 21.52 | 00-004-0587; 00-004-0864 |
| 28.3741 | 7618.29 | 0.0600 | 3.15076 | 20.95 | |
| 31.2412 | 5345.08 | 0.0840 | 2.86074 | 14.70 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 31.6693 | 36360.15 | 0.1680 | 2.82304 | 100.00 | 01-077-2064; 01-080-2171; 01-080-1385 |
| 33.2008 | 2479.83 | 0.2160 | 2.69623 | 6.82 | 01-075-1620 |
| 35.1381 | 2169.41 | 0.1080 | 2.55188 | 5.97 | 01-075-1862 |
| 35.6153 | 840.57 | 0.1440 | 2.51878 | 2.31 | |
| 36.0187 | 1940.96 | 0.2160 | 2.49149 | 5.34 | 01-075-1620 |
| 36.8682 | 14981.02 | 0.1920 | 2.43601 | 41.20 | 01-074-1132; 00-043-1022 |
| 37.7662 | 2289.08 | 0.1440 | 2.38012 | 6.30 | 01-075-1862; 01-075-1620 |
| 37.9115 | 2324.36 | 0.1440 | 2.37133 | 6.39 | 01-075-1620 |
| 38.5093 | 3007.98 | 0.1680 | 2.33589 | 8.27 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.5357 | 5402.53 | 0.1440 | 2.22367 | 14.86 | 00-004-0587 |
| 42.8889 | 4401.33 | 0.0840 | 2.10695 | 12.10 | 00-043-1022 |
| 43.3570 | 2805.64 | 0.0960 | 2.08528 | 7.72 | 01-075-1862; 01-086-2270 |
| 44.7964 | 10963.51 | 0.1080 | 2.02156 | 30.15 | 01-074-1132; 01-085-1327 |
| 44.8679 | 10621.90 | 0.0720 | 2.02352 | 29.21 | |
| 45.4104 | 24150.21 | 0.1440 | 1.99565 | 66.42 | 01-077-2064; 01-086-2270 |
| 45.5258 | 16258.59 | 0.0840 | 1.99580 | 44.72 | |
| 46.9632 | 626.10 | 0.2400 | 1.93322 | 1.72 | 00-004-0864; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 47.3246 | 658.25 | 0.2880 | 1.91929 | 1.81 | 01-086-2270 |
| 49.7393 | 913.28 | 0.2400 | 1.83163 | 2.51 | 01-075-1620 |
| 50.1521 | 1726.87 | 0.2400 | 1.81751 | 4.75 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235 |
| 52.5316 | 1163.79 | 0.1680 | 1.74065 | 3.20 | 01-075-1862 |
| 53.8366 | 1023.58 | 0.1680 | 1.70149 | 2.82 | 01-077-2064 |
| 55.6551 | 1752.47 | 0.2880 | 1.65013 | 4.82 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.4219 | 7592.67 | 0.1680 | 1.62951 | 20.88 | 01-077-2064 |
| 56.5827 | 4783.21 | 0.0960 | 1.62930 | 13.16 | |
| 57.4762 | 2278.94 | 0.1560 | 1.60210 | 6.27 | 01-075-1862 |
| 58.5972 | 1362.66 | 0.1440 | 1.57409 | 3.75 | 00-004-0587 |
| 59.3031 | 7575.05 | 0.0600 | 1.55703 | 20.83 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 59.3900 | 8261.37 | 0.0840 | 1.55496 | 22.72 | 01-074-1132; 01-075-1620 |
| 59.4959 | 6726.73 | 0.1200 | 1.55244 | 18.50 | 01-074-1132; 01-075-1620 |
| 61.3019 | 441.51 | 0.2880 | 1.51096 | 1.21 | 01-075-1862 |
| 62.2782 | 2608.95 | 0.0720 | 1.48960 | 7.18 | 00-043-1022; 01-086-2270 |
| 65.2207 | 13469.00 | 0.1320 | 1.42933 | 37.04 | 01-074-1132; 01-085-1327 |
| 65.2698 | 13738.25 | 0.0720 | 1.42837 | 37.78 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 65.4156 | 10243.71 | 0.1440 | 1.42554 | 28.17 | 01-074-1132; 01-086-2270 |
| 66.1631 | 4882.33 | 0.0840 | 1.41124 | 13.43 | 01-077-2064; 01-075-1620 |
| 66.3699 | 4517.80 | 0.0960 | 1.41084 | 12.43 | |
| 68.1804 | 1671.57 | 0.1440 | 1.37431 | 4.60 | 01-075-1862; 00-022-1235 |
| 68.3987 | 1215.92 | 0.0720 | 1.37386 | 3.34 | |
| 69.6973 | 393.73 | 0.2880 | 1.34807 | 1.08 | 01-074-1132; 01-075-1620 |
| 71.3995 | 1024.75 | 0.1440 | 1.32005 | 2.82 | 01-075-1620; 01-080-2173; 01-086-2270 |
| 73.0366 | 673.44 | 0.1680 | 1.29445 | 1.85 | 01-077-2064; 01-086-2270 |
| 73.6996 | 1289.19 | 0.1680 | 1.28443 | 3.55 | 00-004-0587; 00-022-1235 |

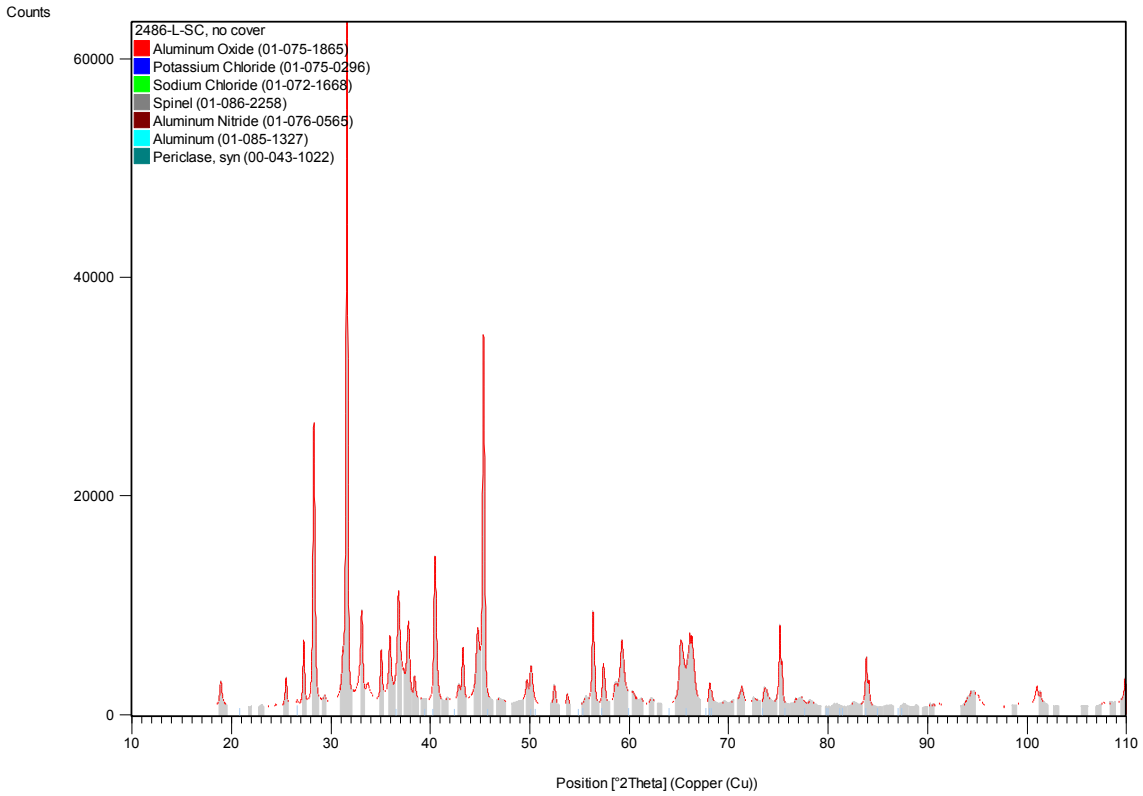
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 75.2686 | 7731.11 | 0.1320 | 1.26150 | 21.26 | 01-077-2064 |
| 75.4553 | 4601.85 | 0.1080 | 1.26197 | 12.66 | |
| 76.8932 | 803.24 | 0.1440 | 1.23885 | 2.21 | 01-075-1862; 01-086-2270 |
| 77.3110 | 1896.42 | 0.1440 | 1.23320 | 5.22 | 01-075-1862; 01-074-1132 |
| 77.5781 | 1403.17 | 0.1440 | 1.23267 | 3.86 | |
| 78.2247 | 1164.60 | 0.1920 | 1.22106 | 3.20 | 01-074-1132; 01-085-1327 |
| 78.5960 | 1147.49 | 0.2880 | 1.21622 | 3.16 | 01-074-1132; 00-043-1022; 01-086-2270 |
| 80.7128 | 248.08 | 0.4800 | 1.18958 | 0.68 | 01-075-1862 |
| 82.6144 | 1392.74 | 0.2160 | 1.16695 | 3.83 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.9217 | 4922.23 | 0.1080 | 1.15207 | 13.54 | 01-077-2064 |
| 84.2258 | 2690.92 | 0.1080 | 1.15154 | 7.40 | |
| 85.8286 | 427.08 | 0.2640 | 1.13129 | 1.17 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 87.6733 | 252.41 | 0.2400 | 1.11218 | 0.69 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 88.9651 | 155.84 | 0.1440 | 1.09934 | 0.43 | 01-075-1862 |
| 90.3854 | 411.68 | 0.1440 | 1.08572 | 1.13 | 00-022-1235 |
| 94.0936 | 2441.48 | 0.2160 | 1.05245 | 6.71 | 00-043-1022; 00-004-0864 |
| 94.4708 | 1964.46 | 0.2400 | 1.04924 | 5.40 | 00-004-0587; 00-004-0864 |
| 95.2475 | 692.14 | 0.1920 | 1.04273 | 1.90 | |
| 99.3427 | 1097.45 | 0.3360 | 1.01043 | 3.02 | |
| 101.1167 | 1737.02 | 0.1920 | 0.99745 | 4.78 | |
| 101.4434 | 1230.89 | 0.1680 | 0.99759 | 3.39 | |
| 104.7029 | 145.49 | 0.5760 | 0.97288 | 0.40 | |
| 105.8657 | 241.62 | 0.5760 | 0.96537 | 0.66 | 00-043-1022; 00-004-0864 |
| 107.7641 | 945.02 | 0.1680 | 0.95357 | 2.60 | |
| 109.7735 | 1914.33 | 0.0360 | 0.94167 | 5.26 | 00-043-1022 |

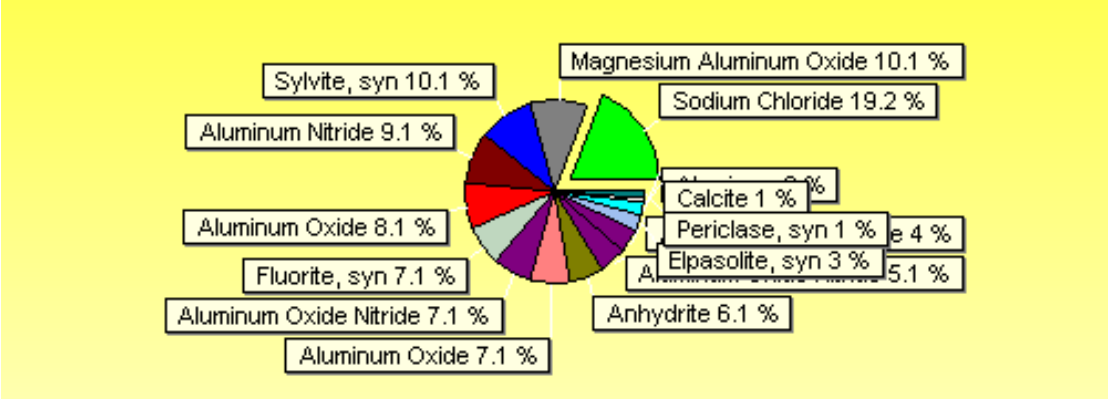
## 8.3: 2486-L-SC

### 8.3.1: Measurement Conditions of 2486-L

| | |
|---|---|
| Dataset Name | 2486-L-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2486-L-SC, no cover.sd |
| Sample Identification | 2486-L-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 9/21/2010 4:12:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 8.3.2: Main Graphics, Analyze View of 2486-L


Counts
2486-L-SC, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Periclase, syn (00-043-1022)
60000
40000
20000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Sylvite, syn 10.1 %
Magnesium Aluminum Oxide 10.1 %
Sodium Chloride 19.2 %
Aluminum Nitride 9.1 %
Calcite 1 %
Periclase, syn 1 %
Aluminum Oxide 8.1 %
Elpasolite, syn 3 %
Fluorite, syn 7.1 %
Aluminum Oxide Nitride 7.1 %
Anhydrite 6.1 %
Aluminum Oxide 7.1 %

**8.3.3: Pattern List of 2486-L**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 54 | Aluminum Oxide | Al2 O3 | 8 | 23 | 0 |
| 00-004-0587 | 42 | Sylvite, syn | K Cl | 10 | 10 | 0 |
| 01-077-2064 | 41 | Sodium Chloride | Na Cl | 19 | 9 | 0 |
| 01-074-1132 | 44 | Magnesium Aluminum Oxide | Mg Al2 O4 | 10 | 14 | 0 |
| 01-075-1620 | 39 | Aluminum Nitride | Al N | 9 | 11 | 0 |
| 01-080-2171 | 18 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 7 | 12 | 0 |
| 01-080-2172 | 20 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 5 | 12 | 0 |
| 01-080-2173 | 19 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 4 | 11 | 0 |
| 01-085-1327 | 30 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 27 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 19 | Fluorite, syn | Ca F2 | 7 | 5 | 0 |
| 00-022-1235 | 32 | Elpasolite, syn | K2 Na Al F6 | 3 | 15 | 0 |
| 01-086-2270 | 26 | Anhydrite | Ca ( S O4 ) | 6 | 39 | 0 |
| 01-080-1385 | 20 | Aluminum Oxide | Al2.667 O4 | 7 | 12 | 0 |
| 01-086-2340 | 12 | Calcite | Ca ( C O3 ) | 1 | 18 | 0 |

**8.3.4: Peak List of 2486-L**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 11.0974 | 38.92 | 0.9446 | 7.97314 | 0.06 | |
| 16.2435 | 70.19 | 0.4723 | 5.45693 | 0.11 | |
| 18.8818 | 2217.30 | 0.0720 | 4.69608 | 3.53 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 18.9432 | 2565.64 | 0.0689 | 4.68487 | 4.08 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 21.0380 | 228.34 | 0.3149 | 4.22288 | 0.36 | |
| 23.0137 | 314.11 | 0.1968 | 3.86463 | 0.50 | 01-086-2270; 01-086-2340 |
| 23.8019 | 323.60 | 0.1968 | 3.73842 | 0.51 | |
| 24.4061 | 369.53 | 0.1181 | 3.64721 | 0.59 | |
| 25.4559 | 2776.25 | 0.0720 | 3.49624 | 4.42 | 01-075-1862; 01-086-2270 |
| 25.5136 | 2862.00 | 0.0590 | 3.49136 | 4.55 | 01-075-1862; 01-086-2270 |
| 26.5549 | 837.77 | 0.0984 | 3.35677 | 1.33 | |
| 27.2561 | 6023.62 | 0.1181 | 3.27198 | 9.58 | 01-077-2064 |
| 28.2676 | 25602.28 | 0.1574 | 3.15715 | 40.72 | 00-004-0587; 00-004-0864 |
| 29.3195 | 1215.10 | 0.1574 | 3.04624 | 1.93 | 01-086-2340 |
| 31.1727 | 5081.80 | 0.1181 | 2.86924 | 8.08 | 01-074-1132; 00-022-1235; 01-086-2340 |
| 31.6095 | 62878.02 | 0.1574 | 2.83058 | 100.00 | 01-077-2064 |
| 33.0803 | 8939.73 | 0.0886 | 2.70801 | 14.22 | 01-075-1620 |
| 33.7313 | 2221.09 | 0.1378 | 2.65723 | 3.53 | |
| 35.0599 | 5387.06 | 0.0984 | 2.55952 | 8.57 | 01-075-1862 |
| 35.8917 | 6203.36 | 0.0394 | 2.50209 | 9.87 | 01-075-1620; 01-086-2340 |
| 36.7987 | 10779.92 | 0.1279 | 2.44247 | 17.14 | 01-074-1132; 00-043-1022 |
| 37.7720 | 7980.34 | 0.0600 | 2.37977 | 12.69 | 01-075-1862; 01-075-1620 |
| 37.8583 | 6874.35 | 0.0590 | 2.37651 | 10.93 | 01-075-1862; 01-075-1620 |
| 38.4179 | 2869.15 | 0.1968 | 2.34317 | 4.56 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.4080 | 12753.38 | 0.1574 | 2.23225 | 20.28 | 00-004-0587 |
| 41.7138 | 993.51 | 0.1574 | 2.16534 | 1.58 | 01-075-1862 |
| 42.8199 | 2168.34 | 0.1181 | 2.11193 | 3.45 | 00-043-1022 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 43.2748 | 5510.31 | 0.1476 | 2.09078 | 8.76 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 44.6770 | 6287.95 | 0.0787 | 2.02837 | 10.00 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3343 | 33921.75 | 0.1800 | 1.99882 | 53.95 | 01-077-2064; 01-086-2270 |
| 45.4402 | 22831.59 | 0.0960 | 1.99936 | 36.31 | |
| 46.8868 | 933.23 | 0.2880 | 1.93619 | 1.48 | 00-004-0864; 01-086-2270 |
| 49.6939 | 2555.77 | 0.1200 | 1.83319 | 4.06 | 01-075-1620 |
| 50.0758 | 3736.10 | 0.0720 | 1.82010 | 5.94 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 52.4290 | 2066.65 | 0.0960 | 1.74381 | 3.29 | 01-075-1862; 01-086-2270 |
| 53.7665 | 1375.89 | 0.0840 | 1.70355 | 2.19 | 01-077-2064 |
| 54.9060 | 449.79 | 0.2880 | 1.67086 | 0.72 | |
| 55.6027 | 1072.63 | 0.2880 | 1.65156 | 1.71 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.3308 | 8643.86 | 0.1200 | 1.63193 | 13.75 | 01-077-2064 |
| 56.5090 | 5058.80 | 0.0840 | 1.63125 | 8.05 | |
| 57.3692 | 3979.31 | 0.1560 | 1.60483 | 6.33 | 01-075-1862; 01-086-2340 |
| 57.5593 | 2530.01 | 0.0960 | 1.60396 | 4.02 | |
| 58.5303 | 2156.66 | 0.1440 | 1.57573 | 3.43 | 00-004-0587 |
| 59.2282 | 6163.91 | 0.2640 | 1.55882 | 9.80 | 01-074-1132; 01-075-1620; 00-022-1235; 01-086-2270 |
| 60.3472 | 1485.84 | 0.2400 | 1.53256 | 2.36 | 01-080-2171; 01-080-2172; 01-080-1385 |
| 61.1844 | 867.86 | 0.1920 | 1.51358 | 1.38 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 62.2101 | 913.96 | 0.3360 | 1.49107 | 1.45 | 00-043-1022; 01-086-2270 |
| 65.1512 | 6000.02 | 0.1920 | 1.43069 | 9.54 | 01-074-1132; 01-085-1327 |
| 65.3173 | 5805.26 | 0.1440 | 1.43100 | 9.23 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 66.0890 | 6803.63 | 0.0840 | 1.41264 | 10.82 | 01-077-2064; 01-075-1620 |
| 66.2624 | 6527.04 | 0.0960 | 1.41287 | 10.38 | |
| 68.1164 | 2233.90 | 0.0720 | 1.37545 | 3.55 | 01-075-1862; 00-022-1235 |
| 68.2714 | 1523.40 | 0.0960 | 1.37270 | 2.42 | 01-075-1862; 00-022-1235 |
| 69.5627 | 642.41 | 0.1920 | 1.35034 | 1.02 | 01-074-1132; 01-075-1620 |
| 70.4707 | 686.48 | 0.3840 | 1.33515 | 1.09 | 01-075-1862 |
| 71.2121 | 1845.49 | 0.0960 | 1.32306 | 2.94 | 01-075-1620; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 71.3126 | 1884.33 | 0.0720 | 1.32144 | 3.00 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 72.4843 | 922.59 | 0.1920 | 1.30294 | 1.47 | 01-075-1620 |
| 73.5983 | 1891.49 | 0.1080 | 1.28595 | 3.01 | 00-004-0587; 00-022-1235; 01-086-2340 |
| 73.8056 | 1608.36 | 0.1440 | 1.28285 | 2.56 | 00-004-0587; 00-022-1235; 01-086-2340 |
| 75.1396 | 7570.65 | 0.1320 | 1.26335 | 12.04 | 01-077-2064 |
| 75.3574 | 4283.81 | 0.0720 | 1.26337 | 6.81 | |
| 76.7270 | 872.54 | 0.1440 | 1.24112 | 1.39 | 01-075-1862; 01-086-2340 |
| 77.2730 | 921.51 | 0.3840 | 1.23371 | 1.47 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.1327 | 687.00 | 0.1920 | 1.22227 | 1.09 | 01-085-1327; 00-022-1235 |
| 80.9317 | 356.77 | 0.2880 | 1.18691 | 0.57 | 01-075-1620 |
| 82.5348 | 515.05 | 0.2400 | 1.16787 | 0.82 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.8500 | 4674.62 | 0.1080 | 1.15287 | 7.43 | 01-077-2064; 01-086-2340 |
| 84.1032 | 2433.55 | 0.1200 | 1.15290 | 3.87 | |
| 85.8574 | 193.86 | 0.4320 | 1.13098 | 0.31 | 01-074-1132; 01-075-1620; |

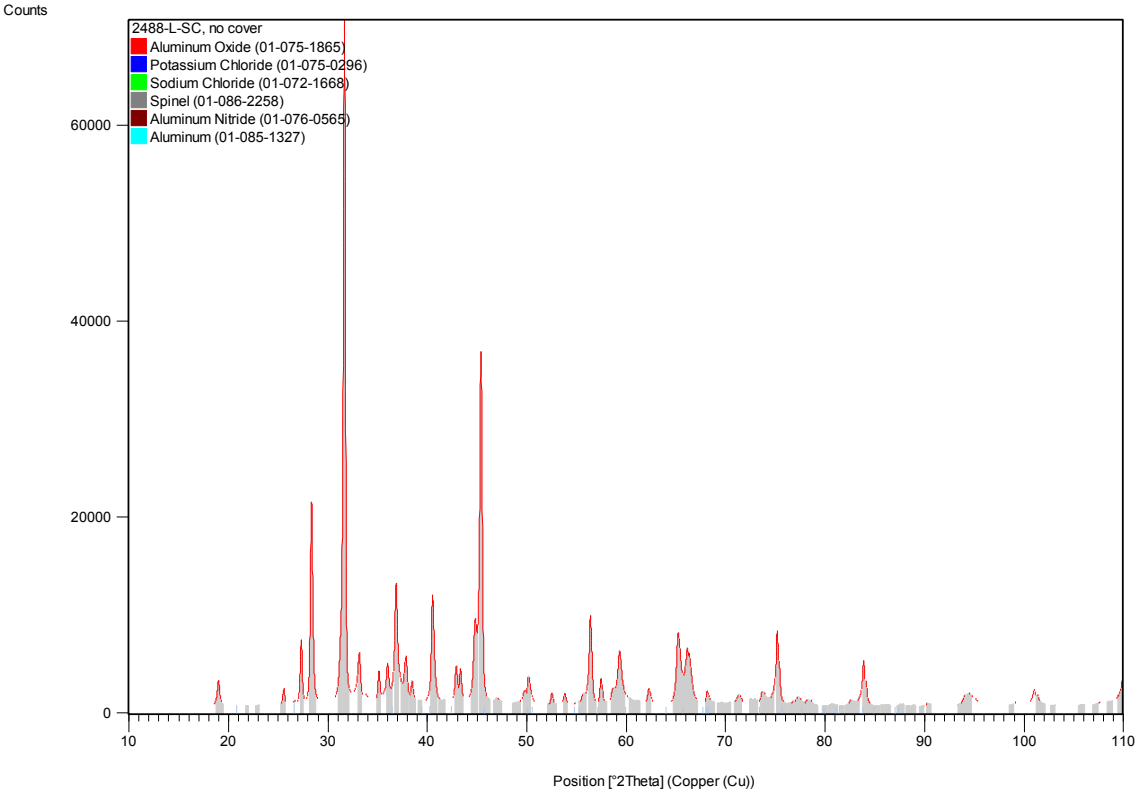
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| | | | | | 01-086-2270; 01-086-2340 |
| 86.2816 | 195.87 | 0.3840 | 1.12651 | 0.31 | 01-075-1862 |
| 87.5269 | 331.26 | 0.2400 | 1.11366 | 0.53 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385 |
| 88.8470 | 206.57 | 0.1920 | 1.10050 | 0.33 | 01-075-1862; 01-086-2270 |
| 90.2516 | 362.61 | 0.0960 | 1.08698 | 0.58 | 00-022-1235 |
| 90.5052 | 275.86 | 0.1440 | 1.08460 | 0.44 | 00-022-1235 |
| 91.1040 | 424.53 | 0.2640 | 1.07902 | 0.68 | |
| 93.9747 | 860.17 | 0.2400 | 1.05347 | 1.37 | 00-043-1022 |
| 94.4142 | 1499.98 | 0.2160 | 1.04972 | 2.39 | 00-004-0587; 00-004-0864 |
| 94.6861 | 1425.28 | 0.1440 | 1.05002 | 2.27 | |
| 95.0850 | 1047.41 | 0.1920 | 1.04408 | 1.67 | |
| 98.1469 | 201.52 | 0.3840 | 1.01952 | 0.32 | |
| 99.2347 | 362.05 | 0.6720 | 1.01124 | 0.58 | |
| 100.9637 | 1882.91 | 0.0960 | 0.99854 | 2.99 | |
| 101.3170 | 1355.04 | 0.1680 | 0.99849 | 2.16 | |
| 104.5693 | 110.46 | 0.4800 | 0.97376 | 0.18 | |
| 107.6114 | 477.33 | 0.1200 | 0.95450 | 0.76 | |
| 108.4108 | 424.10 | 0.1920 | 0.94967 | 0.67 | 00-004-0587 |
| 109.8302 | 2563.80 | 0.0600 | 0.94134 | 4.08 | 00-043-1022 |
| 109.9223 | 2103.32 | 0.0840 | 0.94081 | 3.35 | 00-043-1022 |

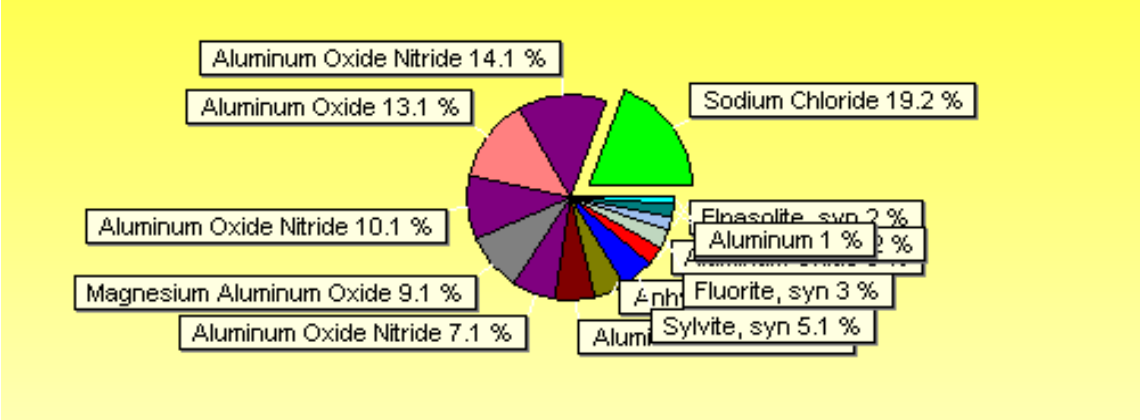
## 8.4: 2488-L-SC

### 8.4.1: Measurement Conditions of 2488-L

| | |
|---|---|
| Dataset Name | 2488-L-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2488-L-SC, no cover.sd |
| Sample Identification | 2488-L-SC, no cover |
| Comment | Exported by X'Pert SW Generated by Ford in project SAP. |
| Measurement Date / Time | 9/21/2010 9:26:00 PM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 8.4.2: Main Graphics, Analyze View of 2488-L


Counts
2488-L-SC, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
60000
40000
20000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Aluminum Oxide Nitride 14.1 %
Aluminum Oxide 13.1 %
Sodium Chloride 19.2 %
Aluminum Oxide Nitride 10.1 %
Aluminum 1 %
Fluorite, syn 3 %
Magnesium Aluminum Oxide 9.1 %
Aluminum Oxide Nitride 7.1 %
Sylvite, syn 5.1 %

**8.4.3: Pattern List of 2488-L**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 40 | Aluminum Oxide | Al2 O3 | 3 | 18 | 0 |
| 00-004-0587 | 44 | Sylvite, syn | K Cl | 5 | 10 | 0 |
| 01-077-2064 | 57 | Sodium Chloride | Na Cl | 19 | 9 | 0 |
| 01-074-1132 | 53 | Magnesium Aluminum Oxide | Mg Al2 O4 | 9 | 16 | 0 |
| 01-075-1620 | 50 | Aluminum Nitride | Al N | 6 | 11 | 0 |
| 01-080-2171 | 16 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 14 | 13 | 0 |
| 01-080-2172 | 17 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 10 | 13 | 0 |
| 01-080-2173 | 18 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 7 | 12 | 0 |
| 01-085-1327 | 32 | Aluminum | Al | 1 | 5 | 0 |
| 00-043-1022 | 33 | Periclase, syn | Mg O | 2 | 6 | 0 |
| 00-004-0864 | 20 | Fluorite, syn | Ca F2 | 3 | 5 | 0 |
| 00-022-1235 | 20 | Elpasolite, syn | K2 Na Al F6 | 2 | 14 | 0 |
| 01-086-2270 | 17 | Anhydrite | Ca ( S O4 ) | 5 | 38 | 0 |
| 01-080-1385 | 17 | Aluminum Oxide | Al2.667 O4 | 13 | 13 | 0 |

**8.4.4: Peak List of 2488-L**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 9.9836 | 368.80 | 0.0295 | 8.86002 | 0.53 | |
| 10.1092 | 363.58 | 0.2362 | 8.75022 | 0.52 | |
| 18.9958 | 2611.99 | 0.1771 | 4.67201 | 3.76 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0858 | 115.11 | 0.2362 | 3.85273 | 0.17 | 01-086-2270 |
| 25.5650 | 1805.22 | 0.0492 | 3.48445 | 2.60 | 01-075-1862; 01-086-2270 |
| 26.6043 | 625.39 | 0.1378 | 3.35065 | 0.90 | |
| 27.3506 | 6587.71 | 0.0787 | 3.26089 | 9.48 | 01-077-2064 |
| 28.3280 | 20731.34 | 0.1968 | 3.15056 | 29.84 | 00-004-0587; 00-004-0864 |
| 29.3837 | 706.88 | 0.1574 | 3.03973 | 1.02 | |
| 31.6768 | 69470.19 | 0.1968 | 2.82472 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.1465 | 5498.52 | 0.0787 | 2.70276 | 7.91 | 01-075-1620 |
| 33.7963 | 1176.64 | 0.2362 | 2.65226 | 1.69 | |
| 35.1003 | 3462.70 | 0.0492 | 2.55666 | 4.98 | 01-075-1862 |
| 35.9975 | 4301.70 | 0.1968 | 2.49497 | 6.19 | 01-075-1620 |
| 36.7969 | 11361.08 | 0.0960 | 2.44056 | 16.35 | 01-074-1132; 00-043-1022 |
| 36.8542 | 12682.59 | 0.0590 | 2.43892 | 18.26 | 01-074-1132; 00-043-1022 |
| 37.8759 | 4885.30 | 0.2755 | 2.37545 | 7.03 | 01-075-1862; 01-075-1620 |
| 38.4732 | 2554.39 | 0.1771 | 2.33993 | 3.68 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 40.5299 | 11292.30 | 0.1476 | 2.22581 | 16.25 | 00-004-0587 |
| 41.7476 | 699.59 | 0.3149 | 2.16367 | 1.01 | 01-075-1862 |
| 42.8747 | 4075.19 | 0.1771 | 2.10936 | 5.87 | 00-043-1022 |
| 43.3154 | 3774.52 | 0.0787 | 2.08892 | 5.43 | 01-075-1862; 01-086-2270 |
| 44.7716 | 8715.42 | 0.1378 | 2.02430 | 12.55 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3896 | 36414.25 | 0.1560 | 1.99651 | 52.42 | 01-077-2064; 01-086-2270 |
| 45.4503 | 30910.50 | 0.0480 | 1.99894 | 44.49 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 46.9450 | 891.36 | 0.2400 | 1.93393 | 1.28 | 00-004-0864; 01-086-2270 |
| 49.7428 | 1589.17 | 0.1680 | 1.83150 | 2.29 | 01-075-1620 |
| 50.1205 | 2911.19 | 0.0960 | 1.81858 | 4.19 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 52.4943 | 1327.64 | 0.2160 | 1.74180 | 1.91 | 01-075-1862 |
| 53.7825 | 1294.62 | 0.0840 | 1.70308 | 1.86 | 01-077-2064 |
| 54.9262 | 500.91 | 0.2880 | 1.67029 | 0.72 | |
| 55.6555 | 1278.42 | 0.2400 | 1.65012 | 1.84 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.3745 | 9156.96 | 0.1560 | 1.63076 | 13.18 | 01-077-2064 |
| 56.5651 | 4937.78 | 0.0960 | 1.62976 | 7.11 | |
| 57.4318 | 2796.43 | 0.1680 | 1.60323 | 4.03 | 01-075-1862 |
| 58.6125 | 1805.32 | 0.1680 | 1.57372 | 2.60 | 00-004-0587 |
| 59.2815 | 5536.44 | 0.1920 | 1.55754 | 7.97 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 61.2395 | 597.07 | 0.1920 | 1.51235 | 0.86 | 01-075-1862; 01-086-2270 |
| 62.2524 | 1807.38 | 0.1920 | 1.49016 | 2.60 | 00-043-1022; 01-086-2270 |
| 65.1719 | 7344.82 | 0.0960 | 1.43028 | 10.57 | 01-074-1132; 01-085-1327 |
| 65.3614 | 6387.29 | 0.1680 | 1.42659 | 9.19 | 01-074-1132; 01-086-2270 |
| 66.1205 | 5914.47 | 0.0840 | 1.41204 | 8.51 | 01-077-2064; 01-075-1620 |
| 66.3260 | 5370.27 | 0.0960 | 1.41167 | 7.73 | |
| 68.1175 | 1573.61 | 0.1680 | 1.37543 | 2.27 | 01-075-1862; 00-022-1235 |
| 69.6324 | 420.23 | 0.2880 | 1.34916 | 0.60 | 01-074-1132; 01-075-1620 |
| 71.2904 | 1263.66 | 0.1200 | 1.32180 | 1.82 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 72.5322 | 774.78 | 0.2880 | 1.30220 | 1.12 | 01-075-1620 |
| 73.6103 | 1457.80 | 0.2160 | 1.28577 | 2.10 | 00-004-0587; 00-022-1235 |
| 73.8729 | 1366.56 | 0.1440 | 1.28503 | 1.97 | |

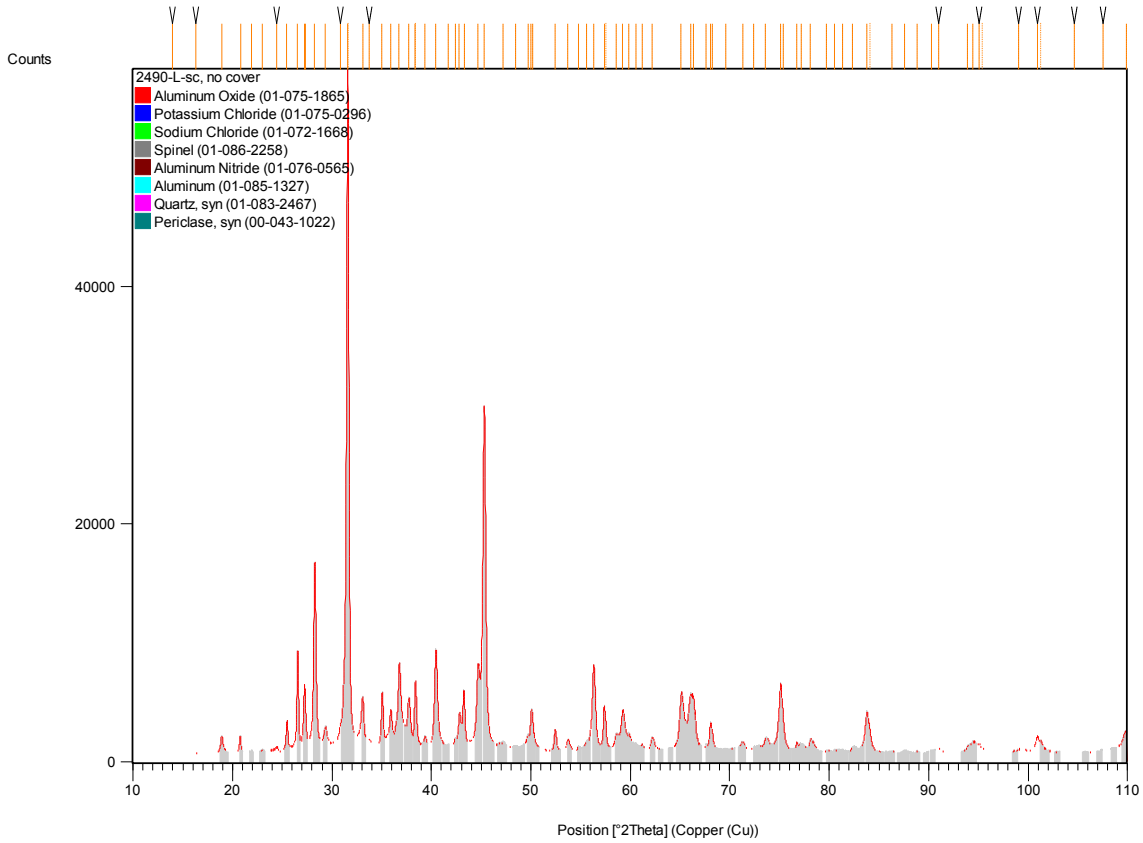
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 75.1704 | 7629.55 | 0.1560 | 1.26291 | 10.98 | 01-077-2064 |
| 75.4116 | 4067.26 | 0.1200 | 1.26260 | 5.85 | |
| 76.7748 | 617.23 | 0.1440 | 1.24047 | 0.89 | 01-075-1862 |
| 77.2771 | 984.14 | 0.2880 | 1.23365 | 1.42 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.1806 | 713.16 | 0.1920 | 1.22164 | 1.03 | 01-085-1327 |
| 78.5484 | 632.00 | 0.2400 | 1.21684 | 0.91 | 01-074-1132; 00-043-1022; 01-086-2270 |
| 80.9134 | 210.23 | 0.6720 | 1.18713 | 0.30 | 01-075-1620 |
| 82.5592 | 685.68 | 0.2400 | 1.16759 | 0.99 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.8836 | 4642.22 | 0.1320 | 1.15249 | 6.68 | 01-077-2064 |
| 84.1275 | 2499.40 | 0.1080 | 1.15263 | 3.60 | |
| 85.7196 | 181.62 | 0.3840 | 1.13245 | 0.26 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 87.5887 | 296.89 | 0.2160 | 1.11303 | 0.43 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385 |
| 88.9180 | 132.56 | 0.1920 | 1.09980 | 0.19 | 01-075-1862; 01-086-2270 |
| 90.2602 | 315.87 | 0.1920 | 1.08690 | 0.45 | 00-022-1235 |
| 91.0329 | 392.10 | 0.3840 | 1.07968 | 0.56 | |
| 94.0144 | 1110.96 | 0.2160 | 1.05313 | 1.60 | 00-043-1022; 00-004-0864 |
| 94.4416 | 1337.13 | 0.2880 | 1.04949 | 1.92 | 00-004-0587; 00-004-0864 |
| 95.1194 | 812.34 | 0.1440 | 1.04379 | 1.17 | |
| 98.1511 | 142.95 | 0.3840 | 1.01949 | 0.21 | |
| 99.2404 | 483.55 | 0.2400 | 1.01120 | 0.70 | |
| 100.9959 | 1618.40 | 0.1680 | 0.99831 | 2.33 | |
| 101.3700 | 1072.07 | 0.1680 | 0.99811 | 1.54 | |
| 104.6935 | 60.87 | 0.7680 | 0.97294 | 0.09 | |
| 107.6151 | 268.40 | 0.1440 | 0.95448 | 0.39 | |
| 108.5395 | 46.84 | 0.2880 | 0.94891 | 0.07 | 00-004-0587 |

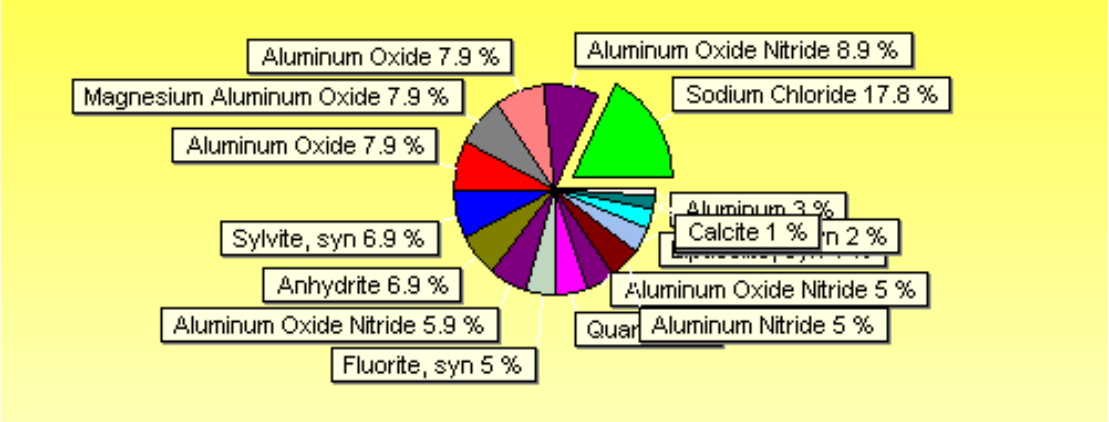
## 8.5: 2490-L-SC

### 8.5.1: Measurement Conditions of 2490-L

| | |
|---|---|
| Dataset Name | 2490-L-sc, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2490-L-sc, no cover.sd |
| Sample Identification | 2490-L-sc, no cover |
| Comment | Exported by X'Pert SW |
| Generated by todd in project Ft. Devens. | |
| Measurement Date / Time | 9/5/2010 8:05:00 PM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 8.5.2: Main Graphics, Analyze View of 2490-L


Counts
2490-L-sc, no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
Quartz, syn (01-083-2467)
Periclase, syn (00-043-1022)
40000
20000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Aluminum Oxide 7.9 %
Aluminum Oxide Nitride 8.9 %
Magnesium Aluminum Oxide 7.9 %
Sodium Chloride 17.8 %
Aluminum Oxide 7.9 %
Calcite 1 %
Sylvite, syn 6.9 %
Anhydrite 6.9 %
Aluminum Oxide Nitride 5 %
Aluminum Oxide Nitride 5.9 %
Aluminum Nitride 5 %
Fluorite, syn 5 %

**8.5.3: Pattern List of 2490-L**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 56 | Aluminum Oxide | Al2 O3 | 8 | 21 | 0 |
| 00-004-0587 | 50 | Sylvite, syn | K Cl | 7 | 10 | 0 |
| 01-077-2064 | 45 | Sodium Chloride | Na Cl | 18 | 9 | 0 |
| 01-074-1132 | 39 | Magnesium Aluminum Oxide | Mg Al2 O4 | 8 | 12 | 0 |
| 01-075-1620 | 35 | Aluminum Nitride | Al N | 5 | 10 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 9 | 12 | 0 |
| 01-080-2172 | 19 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 13 | 0 |
| 01-080-2173 | 20 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 13 | 0 |
| 01-085-1327 | 39 | Aluminum | Al | 3 | 5 | 0 |
| 00-043-1022 | 26 | Periclase, syn | Mg O | 2 | 7 | 0 |
| 00-004-0864 | 16 | Fluorite, syn | Ca F2 | 5 | 5 | 0 |
| 00-022-1235 | 27 | Elpasolite, syn | K2 Na Al F6 | 4 | 16 | 0 |
| 01-086-2270 | 21 | Anhydrite | Ca ( S O4 ) | 7 | 35 | 0 |
| 01-080-1385 | 17 | Aluminum Oxide | Al2.667 O4 | 8 | 13 | 0 |
| 01-086-2340 | 20 | Calcite | Ca ( C O3 ) | 1 | 17 | 0 |
| 01-085-0930 | 36 | Quartz | Si O2 | 5 | 24 | 0 |

**8.5.4: Peak List of 2490-L**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 13.9434 | 72.51 | 0.3149 | 6.35148 | 0.13 | |
| 16.3217 | 204.79 | 0.1378 | 5.43095 | 0.35 | |
| 18.9503 | 1634.08 | 0.1574 | 4.68314 | 2.83 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.7789 | 1570.76 | 0.0394 | 4.27495 | 2.72 | 01-085-0930 |
| 21.9484 | 358.61 | 0.1574 | 4.04974 | 0.62 | 00-022-1235 |
| 22.9884 | 447.68 | 0.2362 | 3.86883 | 0.77 | 01-086-2270; 01-086-2340 |
| 24.4591 | 682.07 | 0.1378 | 3.63944 | 1.18 | |
| 25.4737 | 2820.43 | 0.0886 | 3.49673 | 4.88 | 01-075-1862; 01-086-2270 |
| 26.5291 | 8441.70 | 0.1476 | 3.35998 | 14.61 | 01-085-0930 |
| 27.2518 | 5870.92 | 0.0960 | 3.26978 | 10.16 | 01-077-2064 |
| 27.2912 | 5480.58 | 0.0689 | 3.26785 | 9.49 | 01-077-2064 |
| 28.2530 | 15819.85 | 0.1279 | 3.15875 | 27.38 | 00-004-0587; 00-004-0864 |
| 29.3434 | 2420.24 | 0.2362 | 3.04381 | 4.19 | 01-086-2340 |
| 30.8345 | 2588.91 | 0.1181 | 2.89993 | 4.48 | |
| 31.5939 | 57775.62 | 0.1560 | 2.82960 | 100.00 | 01-077-2064 |
| 31.6526 | 48911.32 | 0.0960 | 2.83151 | 84.66 | |
| 33.1128 | 4839.31 | 0.2400 | 2.70319 | 8.38 | 01-075-1620 |
| 33.7065 | 1303.03 | 0.2880 | 2.65692 | 2.26 | |
| 35.0331 | 5144.83 | 0.1440 | 2.55929 | 8.90 | 01-075-1862 |
| 35.9131 | 3743.25 | 0.1920 | 2.49857 | 6.48 | 01-075-1620; 01-086-2340 |
| 36.7538 | 7538.44 | 0.1680 | 2.44332 | 13.05 | 01-074-1132; 00-043-1022 |
| 37.6834 | 4656.64 | 0.1080 | 2.38516 | 8.06 | 01-075-1862; 01-080-2172; 01-080-2173; 01-080-1385 |
| 38.3717 | 6108.86 | 0.1200 | 2.34395 | 10.57 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 38.4486 | 5791.40 | 0.0720 | 2.34525 | 10.02 | |
| 39.3759 | 1514.11 | 0.1680 | 2.28645 | 2.62 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-086-2340; 01-085-0930 |
| 40.4343 | 8875.04 | 0.0720 | 2.22901 | 15.36 | 00-004-0587; 01-085-0930 |
| 41.7013 | 856.98 | 0.2880 | 2.16417 | 1.48 | 01-075-1862 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 42.3730 | 1343.82 | 0.1920 | 2.13140 | 2.33 | 01-085-0930 |
| 42.7898 | 3335.86 | 0.2160 | 2.11160 | 5.77 | 00-043-1022 |
| 43.2697 | 5380.39 | 0.1320 | 2.08928 | 9.31 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 44.6701 | 7222.10 | 0.2400 | 2.02698 | 12.50 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3316 | 29137.50 | 0.2040 | 1.99893 | 50.43 | 01-077-2064; 01-086-2270 |
| 47.2041 | 1051.08 | 0.2880 | 1.92391 | 1.82 | 00-004-0864; 01-086-2270; 01-086-2340 |
| 48.4757 | 693.00 | 0.2880 | 1.87637 | 1.20 | 01-086-2340 |
| 49.7326 | 1609.69 | 0.2400 | 1.83185 | 2.79 | 01-075-1620 |
| 50.0286 | 3643.58 | 0.0840 | 1.82171 | 6.31 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 50.1639 | 3407.65 | 0.0960 | 1.81711 | 5.90 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.4356 | 2110.76 | 0.0960 | 1.74361 | 3.65 | 01-075-1862; 01-086-2270 |
| 53.7319 | 1248.28 | 0.1920 | 1.70456 | 2.16 | 01-077-2064 |
| 54.7447 | 661.55 | 0.1440 | 1.67540 | 1.15 | 01-085-0930 |
| 55.5834 | 1076.34 | 0.1920 | 1.65209 | 1.86 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.3382 | 7481.57 | 0.1080 | 1.63173 | 12.95 | 01-077-2064 |
| 57.3637 | 4055.92 | 0.1560 | 1.60497 | 7.02 | 01-075-1862; 01-086-2340; 01-085-0930 |
| 57.5393 | 2673.22 | 0.1080 | 1.60447 | 4.63 | |
| 58.5857 | 1700.09 | 0.2880 | 1.57437 | 2.94 | 00-004-0587 |
| 59.2462 | 3692.65 | 0.2160 | 1.55839 | 6.39 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 59.8436 | 1812.01 | 0.1440 | 1.54425 | 3.14 | 01-075-1862; 01-085-0930 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 60.5408 | 891.27 | 0.2880 | 1.52812 | 1.54 | 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385; 01-086-2340 |
| 61.1862 | 805.13 | 0.1680 | 1.51354 | 1.39 | 01-075-1862; 01-086-2270; 01-086-2340 |
| 62.1769 | 1396.48 | 0.1680 | 1.49178 | 2.42 | 00-043-1022; 01-086-2270 |
| 65.0974 | 4917.93 | 0.0960 | 1.43174 | 8.51 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2340 |
| 66.0759 | 5090.58 | 0.1680 | 1.41289 | 8.81 | 01-077-2064; 01-075-1620 |
| 66.3728 | 4590.83 | 0.2400 | 1.40729 | 7.95 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 67.6041 | 809.10 | 0.1440 | 1.38462 | 1.40 | 01-085-0930 |
| 68.0419 | 2497.18 | 0.0960 | 1.37677 | 4.32 | 01-075-1862; 01-085-0930 |
| 68.2619 | 1806.20 | 0.1200 | 1.37287 | 3.13 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 69.5553 | 429.66 | 0.3840 | 1.35047 | 0.74 | 01-074-1132; 01-075-1620; 00-022-1235 |
| 71.2727 | 998.69 | 0.3840 | 1.32209 | 1.73 | 01-075-1620; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 72.4147 | 581.20 | 0.2880 | 1.30402 | 1.01 | 01-075-1620 |
| 73.5976 | 1339.57 | 0.1920 | 1.28596 | 2.32 | 00-004-0587; 00-022-1235; 01-086-2340; 01-085-0930 |
| 75.1463 | 5856.04 | 0.0960 | 1.26325 | 10.14 | 01-077-2064 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 75.3723 | 3345.79 | 0.1440 | 1.26003 | 5.79 | 01-077-2064; 01-080-2171 |
| 76.7245 | 839.25 | 0.1680 | 1.24115 | 1.45 | 01-075-1862; 01-086-2340 |
| 77.1631 | 815.86 | 0.4800 | 1.23519 | 1.41 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.1213 | 1226.80 | 0.0960 | 1.22242 | 2.12 | 01-085-1327; 00-022-1235 |
| 79.7424 | 276.10 | 0.1920 | 1.20160 | 0.48 | 01-080-2171; 01-086-2270; 01-085-0930 |
| 80.5440 | 318.87 | 0.1920 | 1.19164 | 0.55 | 01-075-1862; 01-086-2340 |
| 81.3327 | 321.37 | 0.1920 | 1.18207 | 0.56 | 01-086-2340; 01-085-0930 |
| 82.3686 | 564.82 | 0.4800 | 1.16981 | 0.98 | 01-085-1327; 00-022-1235 |
| 83.8093 | 3538.21 | 0.1920 | 1.15333 | 6.12 | 01-077-2064; 01-086-2340; 01-085-0930 |
| 84.1029 | 1934.09 | 0.1920 | 1.15290 | 3.35 | |
| 86.3134 | 166.50 | 0.3840 | 1.12617 | 0.29 | 01-075-1862 |
| 87.6025 | 213.83 | 0.6720 | 1.11289 | 0.37 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 88.8485 | 198.55 | 0.1920 | 1.10048 | 0.34 | 01-075-1862; 01-086-2270 |
| 90.2461 | 198.66 | 0.5760 | 1.08704 | 0.34 | 00-022-1235 |
| 91.0015 | 415.85 | 0.1680 | 1.07997 | 0.72 | |
| 93.8955 | 586.15 | 0.2880 | 1.05415 | 1.01 | 00-043-1022 |
| 94.4412 | 954.62 | 0.3840 | 1.04949 | 1.65 | 00-004-0587; 00-004-0864 |
| 95.0828 | 847.05 | 0.2160 | 1.04410 | 1.47 | |
| 95.4108 | 410.11 | 0.1440 | 1.04396 | 0.71 | |
| 99.0685 | 261.86 | 0.5760 | 1.01249 | 0.45 | |
| 100.9613 | 1303.77 | 0.2640 | 0.99856 | 2.26 | |
| 101.2834 | 953.31 | 0.2400 | 0.99873 | 1.65 | |
| 104.6490 | 41.09 | 0.7680 | 0.97323 | 0.07 | |
| 107.5405 | 308.58 | 0.4800 | 0.95493 | 0.53 | |
| 109.9041 | 1640.49 | 0.0600 | 0.94091 | 2.84 | 00-043-1022 |

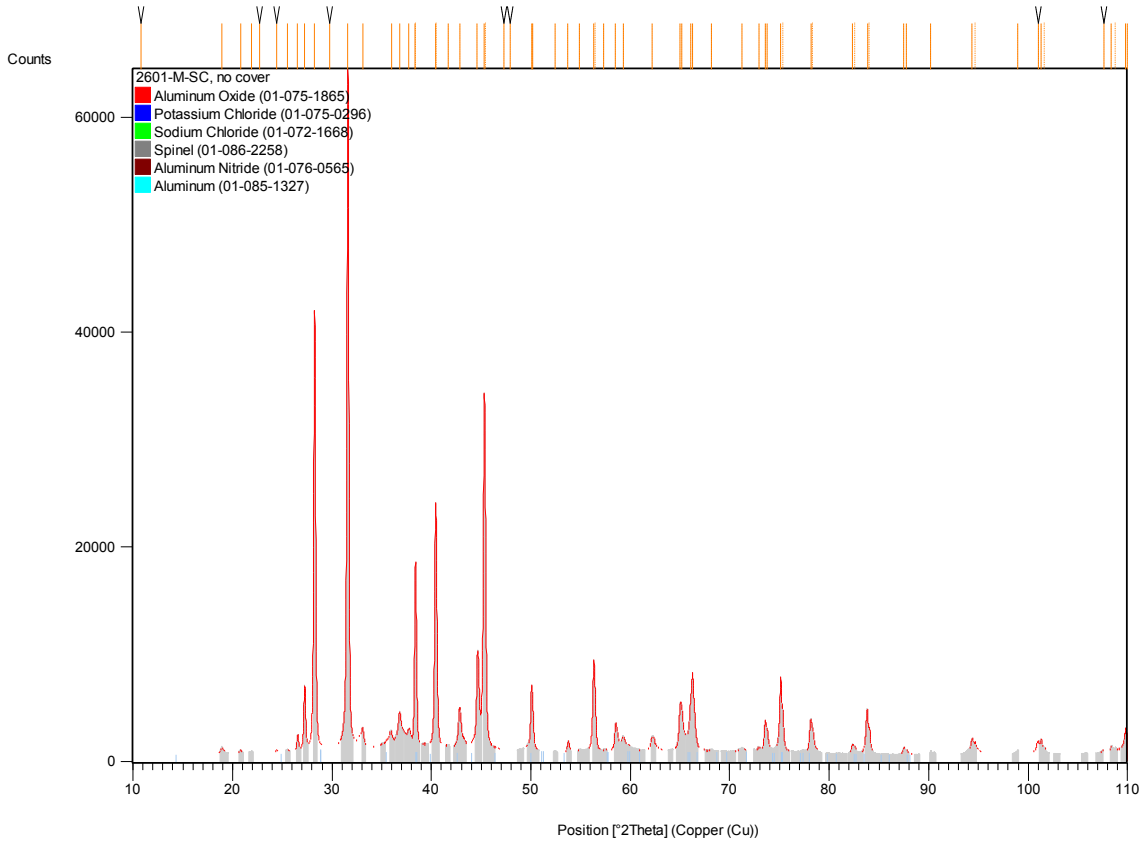
# FACILITY M

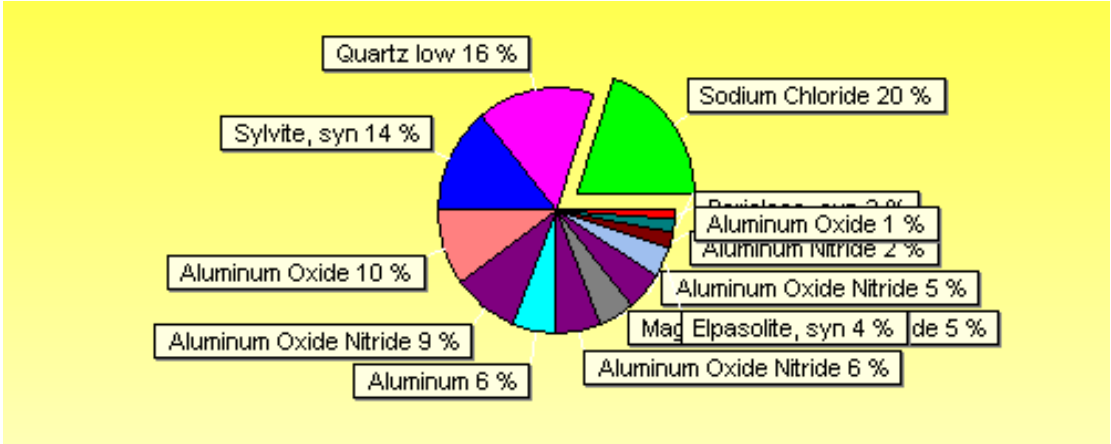
## 9.1: 2601-M-SC

### 9.1.1: Measurement Conditions of 2601-M

| | |
|---|---|
| Dataset Name | 2601-M-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2601-M-SC, no cover.udf |
| Sample Identification | 2601-M-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

**9.1.2: Main Graphics, Analyze View of 2601-M**




**9.1.3: Pattern List of 2601-M**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 13 | Aluminum Oxide | Al2 O3 | 1 | 13 | 0 |
| 00-004-0587 | 59 | Sylvite, syn | K Cl | 14 | 10 | 0 |
| 01-077-2064 | 57 | Sodium Chloride | Na Cl | 20 | 9 | 0 |
| 01-074-1132 | 37 | Magnesium Aluminum Oxide | Mg Al2 O4 | 5 | 11 | 0 |
| 01-075-1620 | 40 | Aluminum Nitride | Al N | 2 | 8 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 9 | 13 | 0 |
| 01-080-2172 | 17 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 17 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 5 | 11 | 0 |
| 01-085-1327 | 53 | Aluminum | Al | 6 | 5 | 0 |
| 00-043-1022 | 43 | Periclase, syn | Mg O | 2 | 7 | 0 |
| 00-022-1235 | 25 | Elpasolite, syn | K2 Na Al F6 | 4 | 15 | 0 |
| 01-080-1385 | 18 | Aluminum Oxide | Al2.667 O4 | 10 | 12 | 0 |
| 01-085-0335 | 27 | Quartz low | Si O2 | 16 | 18 | 0 |

**9.1.4: Peak List of 2601-M**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.7952 | 80.99 | 0.9446 | 8.19563 | 0.13 | |
| 18.9091 | 691.40 | 0.1181 | 4.69325 | 1.08 | 01-074-1132; 00-022-1235 |
| 20.7829 | 415.33 | 0.1181 | 4.27415 | 0.65 | 01-085-0335 |
| 21.9201 | 259.53 | 0.4723 | 4.05491 | 0.41 | 00-022-1235 |
| 22.7481 | 269.83 | 0.3149 | 3.90916 | 0.42 | |
| 24.4017 | 346.93 | 0.1574 | 3.64786 | 0.54 | |
| 25.5133 | 392.43 | 0.1574 | 3.49140 | 0.61 | 01-075-1862 |
| 26.5207 | 1777.72 | 0.1082 | 3.36101 | 2.79 | 01-085-0335 |
| 27.2758 | 6310.01 | 0.1771 | 3.26966 | 9.89 | 01-077-2064 |
| 28.2594 | 41263.36 | 0.1870 | 3.15806 | 64.65 | 00-004-0587 |
| 29.7320 | 779.94 | 0.1181 | 3.00490 | 1.22 | |
| 31.6092 | 63827.51 | 0.2165 | 2.83061 | 100.00 | 01-077-2064; 01-080-2171 |
| 33.0930 | 2367.24 | 0.1378 | 2.70700 | 3.71 | 01-075-1620 |
| 35.9800 | 2054.53 | 0.1574 | 2.49614 | 3.22 | 01-075-1620 |
| 36.7924 | 3876.62 | 0.1574 | 2.44287 | 6.07 | 01-074-1132; 00-043-1022 |
| 37.7448 | 2254.15 | 0.1574 | 2.38340 | 3.53 | 01-075-1862; 01-075-1620; 01-080-2173 |
| 38.3758 | 17098.49 | 0.1560 | 2.34371 | 26.79 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 38.4685 | 14052.68 | 0.0960 | 2.34408 | 22.02 | |
| 40.4187 | 23329.81 | 0.1440 | 2.22984 | 36.55 | 00-004-0587; 01-085-0335 |
| 40.4728 | 21384.02 | 0.0840 | 2.23251 | 33.50 | |
| 41.7279 | 739.73 | 0.3840 | 2.16285 | 1.16 | 01-075-1862 |
| 42.8273 | 4173.59 | 0.1920 | 2.10984 | 6.54 | 00-043-1022 |
| 44.6131 | 8824.10 | 0.1320 | 2.02944 | 13.82 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3198 | 33122.64 | 0.1680 | 1.99943 | 51.89 | 01-077-2064 |
| 45.4466 | 20558.48 | 0.0600 | 1.99910 | 32.21 | |
| 47.3086 | 508.13 | 0.2880 | 1.91991 | 0.80 | |
| 50.0631 | 6294.47 | 0.0600 | 1.82053 | 9.86 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0335 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 50.1750 | 5041.16 | 0.0960 | 1.81674 | 7.90 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0335 |
| 52.4665 | 113.20 | 0.2880 | 1.74265 | 0.18 | 01-075-1862 |
| 53.7233 | 998.41 | 0.1680 | 1.70481 | 1.56 | 01-077-2064 |
| 54.8308 | 294.64 | 0.3360 | 1.67297 | 0.46 | 01-085-0335 |
| 56.3108 | 8250.92 | 0.1560 | 1.63246 | 12.93 | 01-077-2064 |
| 56.4589 | 5877.35 | 0.0960 | 1.63257 | 9.21 | |
| 57.3551 | 303.99 | 0.1920 | 1.60519 | 0.48 | 01-075-1862; 01-085-0335 |
| 58.5222 | 2670.71 | 0.1320 | 1.57593 | 4.18 | 00-004-0587 |
| 59.2552 | 1490.20 | 0.1440 | 1.55817 | 2.33 | 01-074-1132; 01-075-1620; 00-022-1235 |
| 62.1827 | 1505.85 | 0.1200 | 1.49166 | 2.36 | 00-043-1022 |
| 65.0280 | 4612.61 | 0.1080 | 1.43310 | 7.23 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 65.1757 | 4066.73 | 0.1680 | 1.43021 | 6.37 | 01-074-1132; 01-085-1327 |
| 66.0611 | 5074.08 | 0.1200 | 1.41317 | 7.95 | 01-077-2064; 01-075-1620 |
| 66.2463 | 7380.27 | 0.1320 | 1.40967 | 11.56 | 01-075-1862; 00-004-0587; 01-077-2064; 01-075-1620; 01-080-2171 |
| 68.1213 | 183.32 | 0.2880 | 1.37536 | 0.29 | 01-075-1862; 00-022-1235; 01-085-0335 |
| 71.2607 | 350.59 | 0.1920 | 1.32228 | 0.55 | 01-075-1620; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 72.8957 | 442.95 | 0.1440 | 1.29660 | 0.69 | 01-077-2064 |
| 73.5631 | 2939.97 | 0.0720 | 1.28648 | 4.61 | 00-004-0587; 00-022-1235; 01-085-0335 |
| 73.7680 | 2100.24 | 0.1200 | 1.28341 | 3.29 | 00-004-0587; 00-022-1235 |

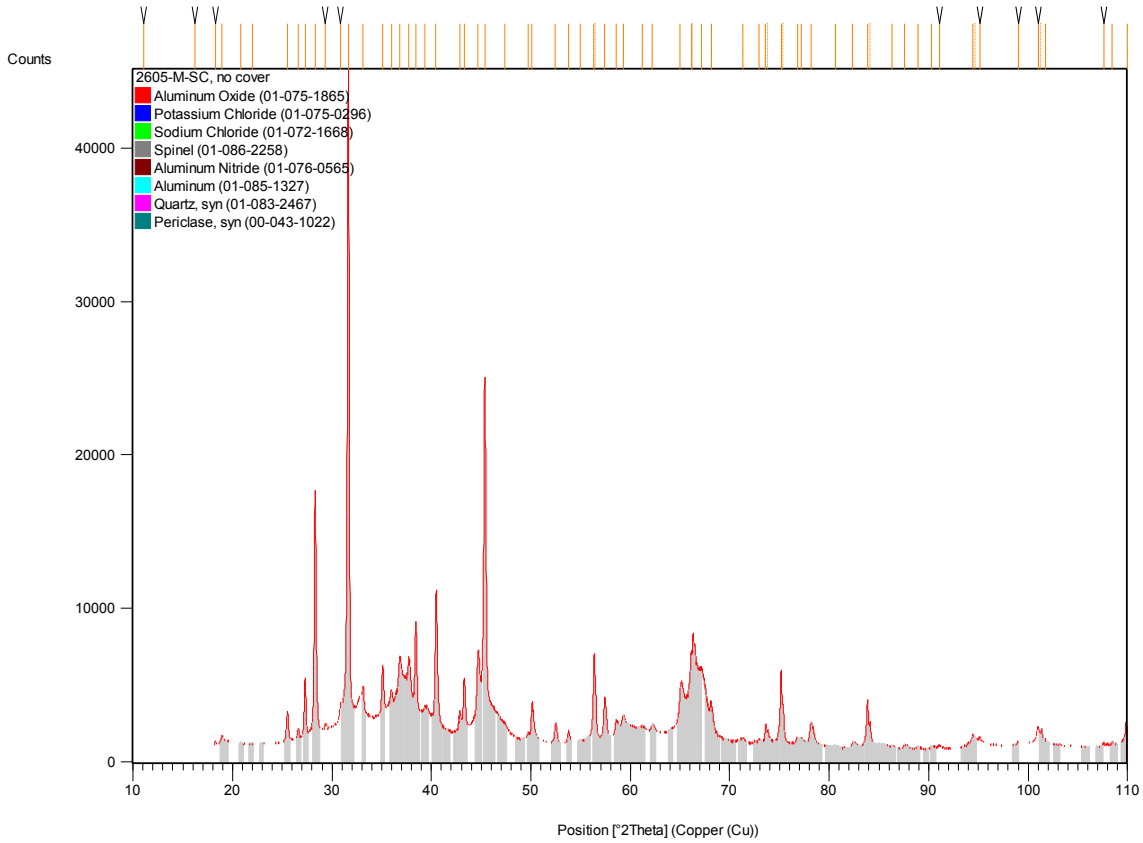
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 75.1293 | 7015.62 | 0.1680 | 1.26350 | 10.99 | 01-077-2064 |
| 75.3467 | 3864.85 | 0.1200 | 1.26352 | 6.06 | |
| 78.1484 | 3172.29 | 0.1800 | 1.22206 | 4.97 | 01-085-1327; 00-022-1235 |
| 78.3788 | 2081.36 | 0.1440 | 1.22208 | 3.26 | |
| 82.3598 | 884.16 | 0.1920 | 1.16991 | 1.39 | 01-085-1327; 00-022-1235 |
| 82.5884 | 639.50 | 0.1440 | 1.17015 | 1.00 | |
| 83.8364 | 4132.72 | 0.1440 | 1.15302 | 6.47 | 01-077-2064; 01-085-0335 |
| 84.0893 | 2201.84 | 0.1200 | 1.15306 | 3.45 | |
| 87.4951 | 637.35 | 0.1200 | 1.11398 | 1.00 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0335 |
| 87.7896 | 409.99 | 0.1440 | 1.11100 | 0.64 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 90.2314 | 296.50 | 0.1440 | 1.08717 | 0.46 | 00-022-1235 |
| 94.3412 | 1445.67 | 0.2400 | 1.05034 | 2.26 | 00-004-0587 |
| 94.6883 | 1005.44 | 0.1920 | 1.05001 | 1.58 | |
| 98.9889 | 360.91 | 0.1920 | 1.01309 | 0.57 | 00-022-1235 |
| 101.0011 | 1207.88 | 0.1680 | 0.99827 | 1.89 | |
| 101.3365 | 1327.86 | 0.2160 | 0.99588 | 2.08 | 00-004-0587 |
| 101.6759 | 565.79 | 0.1920 | 0.99594 | 0.89 | |
| 107.6004 | 368.51 | 0.1680 | 0.95457 | 0.58 | |
| 108.3654 | 659.71 | 0.2160 | 0.94995 | 1.03 | 00-004-0587 |
| 108.7931 | 533.28 | 0.1920 | 0.94975 | 0.84 | |
| 109.8130 | 2400.84 | 0.0840 | 0.94144 | 3.76 | 00-043-1022 |
| 109.9386 | 1722.45 | 0.0600 | 0.94071 | 2.70 | 00-043-1022 |

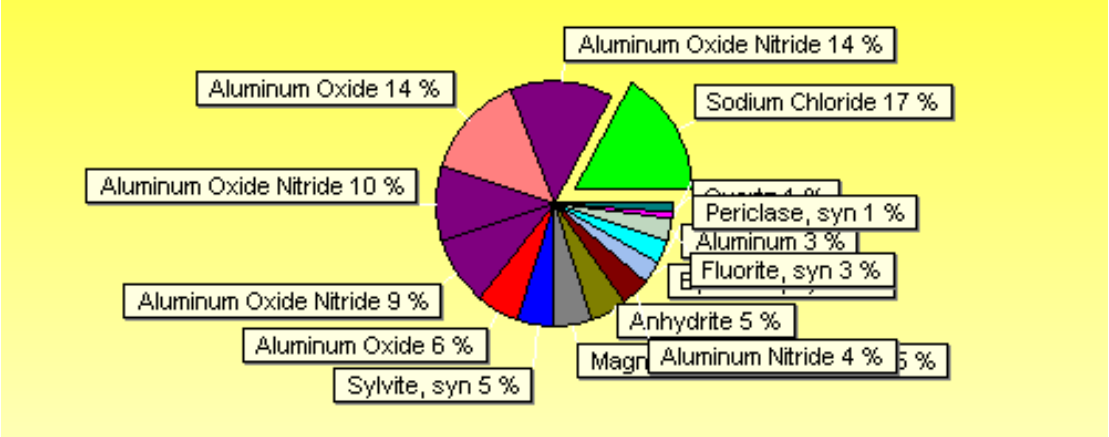
## 9.2: 2605-M-SC

### 9.2.1: Measurement Conditions of 2605-M

| | |
|---|---|
| Dataset Name | 2605-M-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2605-M-SC, no cover.udf |
| Sample Identification | 2605-M-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 9.2.2: Main Graphics, Analyze View of 2605-M




**9.2.3: Pattern List of 2605-M**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 48 | Aluminum Oxide | Al2 O3 | 6 | 21 | 0 |
| 00-004-0587 | 46 | Sylvite, syn | K Cl | 5 | 10 | 0 |
| 01-077-2064 | 58 | Sodium Chloride | Na Cl | 17 | 9 | 0 |
| 01-074-1132 | 42 | Magnesium Aluminum Oxide | Mg Al2 O4 | 5 | 14 | 0 |
| 01-075-1620 | 35 | Aluminum Nitride | Al N | 4 | 8 | 0 |
| 01-080-2171 | 20 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 14 | 13 | 0 |
| 01-080-2172 | 20 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 10 | 13 | 0 |
| 01-080-2173 | 20 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 9 | 13 | 0 |
| 01-085-1327 | 50 | Aluminum | Al | 3 | 5 | 0 |
| 00-043-1022 | 34 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 18 | Fluorite, syn | Ca F2 | 3 | 5 | 0 |
| 00-022-1235 | 25 | Elpasolite, syn | K2 Na Al F6 | 3 | 14 | 0 |
| 01-086-2270 | 16 | Anhydrite | Ca ( S O4 ) | 5 | 37 | 0 |
| 01-080-1385 | 22 | Aluminum Oxide | Al2.667 O4 | 14 | 13 | 0 |
| 01-085-0930 | 18 | Quartz | Si O2 | 1 | 21 | 0 |

**9.2.4: Peak List of 2605-M**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 11.0434 | 71.69 | 0.6298 | 8.01202 | 0.16 | |
| 16.2083 | 35.85 | 0.4723 | 5.46870 | 0.08 | |
| 18.2540 | 450.49 | 0.1181 | 4.86019 | 1.02 | |
| 18.9663 | 804.77 | 0.1181 | 4.67922 | 1.83 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.8276 | 218.70 | 0.2362 | 4.26507 | 0.50 | 01-085-0930 |
| 22.0289 | 87.69 | 0.4723 | 4.03513 | 0.20 | 00-022-1235 |
| 25.5302 | 2054.42 | 0.1771 | 3.48913 | 4.66 | 01-075-1862; 01-086-2270 |
| 26.5930 | 968.70 | 0.1181 | 3.35204 | 2.20 | 01-085-0930 |
| 27.3005 | 4298.76 | 0.1476 | 3.26676 | 9.76 | 01-077-2064 |
| 28.3000 | 16362.88 | 0.1771 | 3.15361 | 37.15 | 00-004-0587; 00-004-0864 |
| 29.3555 | 1297.92 | 0.1574 | 3.04258 | 2.95 | |
| 30.8699 | 2660.29 | 0.1574 | 2.89669 | 6.04 | |
| 31.6428 | 44042.39 | 0.1181 | 2.82768 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 33.1367 | 3685.76 | 0.1574 | 2.70354 | 8.37 | 01-075-1620 |
| 35.0696 | 4811.97 | 0.0984 | 2.55883 | 10.93 | 01-075-1862 |
| 35.9591 | 3458.83 | 0.1181 | 2.49755 | 7.85 | 01-075-1620 |
| 36.8139 | 5612.81 | 0.2165 | 2.44150 | 12.74 | 01-074-1132; 00-043-1022 |
| 37.7218 | 5518.87 | 0.1378 | 2.38479 | 12.53 | 01-075-1862; 01-075-1620; 01-080-2172; 01-080-2173 |
| 38.4300 | 7905.10 | 0.1181 | 2.34246 | 17.95 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 39.3523 | 2361.27 | 0.2362 | 2.28966 | 5.36 | 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0930 |
| 40.4376 | 9446.08 | 0.0886 | 2.23068 | 21.45 | 00-004-0587; 01-085-0930 |
| 42.8607 | 1976.19 | 0.1968 | 2.11002 | 4.49 | 00-043-1022 |
| 43.2767 | 4102.97 | 0.0492 | 2.09070 | 9.32 | 01-075-1862; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 44.6647 | 5732.21 | 0.1771 | 2.02890 | 13.02 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3579 | 23614.84 | 0.1181 | 1.99949 | 53.62 | 01-077-2064; 01-086-2270 |
| 47.3863 | 1223.31 | 0.4723 | 1.91853 | 2.78 | 01-086-2270 |
| 49.7019 | 609.59 | 0.1181 | 1.83443 | 1.38 | 01-075-1620 |
| 50.0879 | 2436.18 | 0.0590 | 1.82120 | 5.53 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.4502 | 1205.26 | 0.1181 | 1.74460 | 2.74 | 01-075-1862; 01-086-2270 |
| 53.7818 | 696.33 | 0.1181 | 1.70451 | 1.58 | 01-077-2064 |
| 54.9415 | 137.78 | 0.2362 | 1.67124 | 0.31 | 01-085-0930 |
| 56.3460 | 5522.21 | 0.1560 | 1.63152 | 12.54 | 01-077-2064 |
| 56.5358 | 3136.56 | 0.0960 | 1.63053 | 7.12 | |
| 57.4147 | 2839.89 | 0.1320 | 1.60367 | 6.45 | 01-075-1862; 01-085-0930 |
| 58.5601 | 1363.59 | 0.1200 | 1.57500 | 3.10 | 00-004-0587 |
| 59.2742 | 1735.90 | 0.3360 | 1.55772 | 3.94 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 61.1885 | 1008.87 | 0.2880 | 1.51349 | 2.29 | 01-075-1862; 01-086-2270 |
| 62.2261 | 1096.10 | 0.3840 | 1.49072 | 2.49 | 00-043-1022; 01-086-2270 |
| 65.0238 | 3365.46 | 0.1200 | 1.43318 | 7.64 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 66.1384 | 5859.34 | 0.0840 | 1.41171 | 13.30 | 01-077-2064; 01-075-1620 |
| 66.2911 | 6849.69 | 0.0960 | 1.41232 | 15.55 | |
| 67.1271 | 4700.37 | 0.4800 | 1.39329 | 10.67 | 01-086-2270 |
| 68.1396 | 2504.70 | 0.1440 | 1.37504 | 5.69 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 71.3250 | 265.82 | 0.3840 | 1.32125 | 0.60 | 01-075-1620; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 72.9472 | 265.01 | 0.1440 | 1.29581 | 0.60 | 01-077-2064; 01-086-2270 |

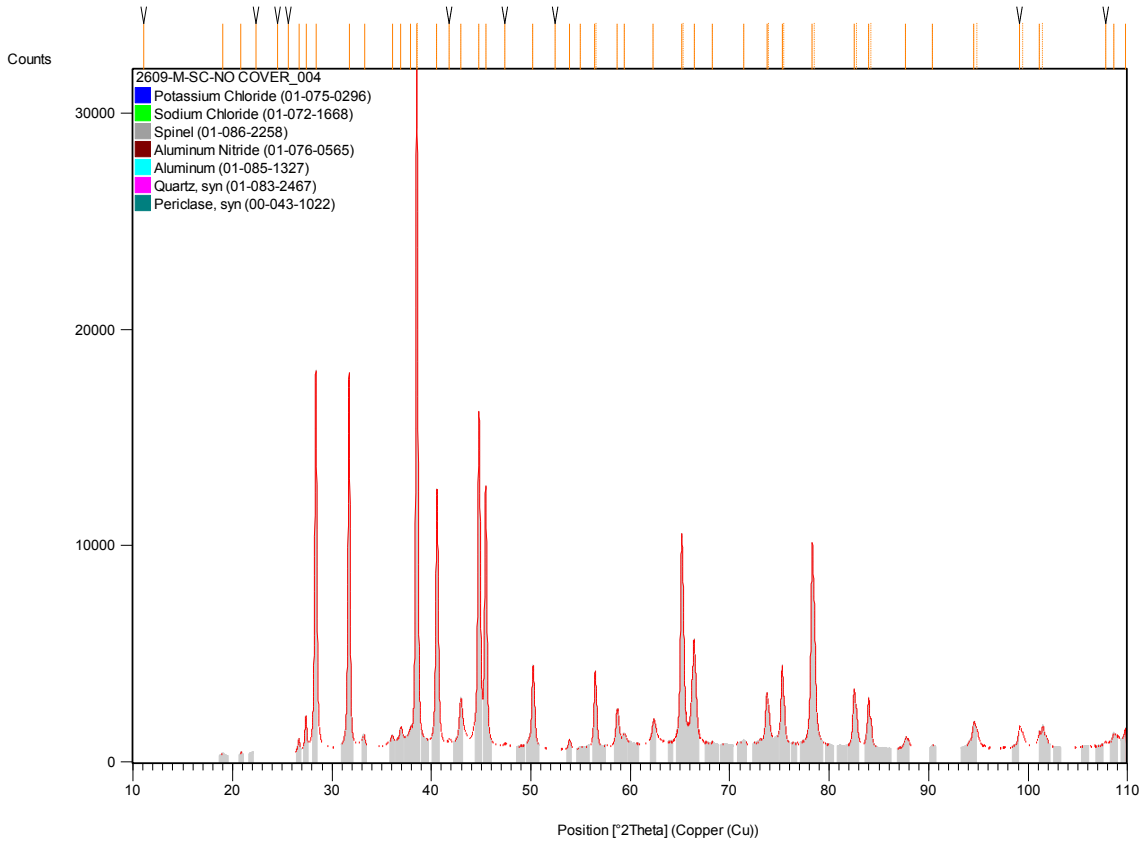
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 73.6084 | 1272.26 | 0.1200 | 1.28580 | 2.89 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 73.8133 | 869.04 | 0.0960 | 1.28592 | 1.97 | |
| 75.1673 | 4856.19 | 0.1560 | 1.26295 | 11.03 | 01-077-2064 |
| 75.4010 | 2425.76 | 0.0600 | 1.26275 | 5.51 | |
| 76.7785 | 525.79 | 0.1680 | 1.24042 | 1.19 | 01-075-1862 |
| 77.1643 | 456.38 | 0.2880 | 1.23517 | 1.04 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 78.1334 | 1517.48 | 0.1680 | 1.22226 | 3.45 | 01-085-1327; 00-022-1235 |
| 80.5968 | 148.68 | 0.2880 | 1.19100 | 0.34 | 01-075-1862 |
| 82.3550 | 387.56 | 0.4320 | 1.16997 | 0.88 | 01-085-1327; 00-022-1235 |
| 83.8607 | 3141.00 | 0.1320 | 1.15275 | 7.13 | 01-077-2064; 01-085-0930 |
| 84.1195 | 1684.14 | 0.0960 | 1.15272 | 3.82 | |
| 86.3348 | 179.02 | 0.2880 | 1.12595 | 0.41 | 01-075-1862 |
| 87.5260 | 307.09 | 0.1680 | 1.11367 | 0.70 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 88.9110 | 165.94 | 0.2400 | 1.09987 | 0.38 | 01-075-1862; 01-086-2270 |
| 90.2532 | 195.66 | 0.1920 | 1.08697 | 0.44 | 00-022-1235 |
| 91.0674 | 216.39 | 0.1440 | 1.07936 | 0.49 | |
| 94.4347 | 771.06 | 0.1680 | 1.04955 | 1.75 | 00-004-0587; 00-004-0864 |
| 94.7286 | 596.36 | 0.1440 | 1.04967 | 1.35 | |
| 95.1218 | 534.45 | 0.1920 | 1.04377 | 1.21 | |
| 99.0492 | 208.15 | 0.5760 | 1.01264 | 0.47 | |
| 101.0024 | 1222.30 | 0.2160 | 0.99826 | 2.78 | |
| 101.3244 | 1058.33 | 0.1440 | 0.99844 | 2.40 | |
| 101.7300 | 386.57 | 0.1920 | 0.99309 | 0.88 | 00-004-0587; 00-022-1235 |
| 107.6132 | 277.77 | 0.1920 | 0.95449 | 0.63 | |
| 108.4422 | 331.97 | 0.1920 | 0.94949 | 0.75 | 00-004-0587 |
| 109.9386 | 1260.07 | 0.0240 | 0.94071 | 2.86 | 00-043-1022 |

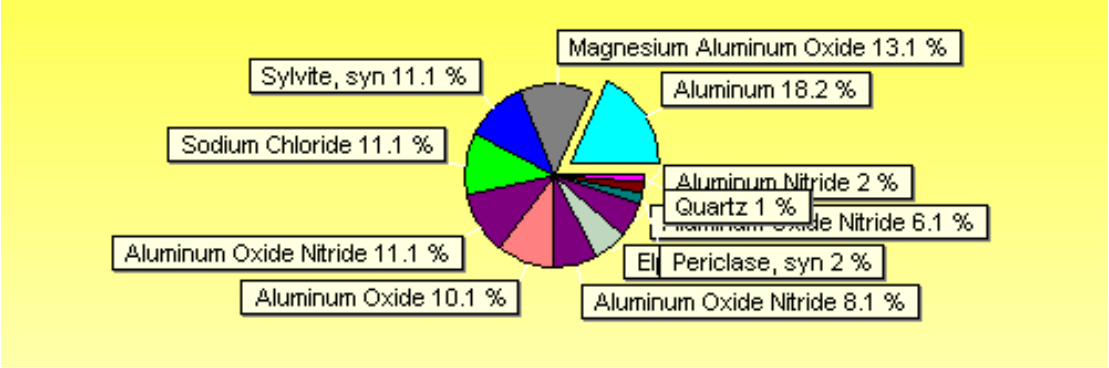
## 9.3: 2609-M-SC

### 9.3.1: Measurement Conditions of 2609-M

| | |
|---|---|
| Dataset Name | 2609-M-SC-NO COVER_004 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2609-M-SC-NO COVER_004.udf |
| Sample Identification | 2609-M-SC-NO COVER |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 9.3.2: Main Graphics, Analyze View of 2609-M




**9.3.3: Pattern List of 2609-M**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 00-004-0587 | 59 | Sylvite, syn | K Cl | 11 | 10 | 0 |
| 01-077-2064 | 61 | Sodium Chloride | Na Cl | 11 | 9 | 0 |
| 01-074-1132 | 17 | Magnesium Aluminum Oxide | Mg Al2 O4 | 13 | 10 | 0 |
| 01-075-1620 | 43 | Aluminum Nitride | Al N | 2 | 7 | 0 |
| 01-080-2171 | 20 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 11 | 11 | 0 |
| 01-080-2172 | 18 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 8 | 11 | 0 |
| 01-080-2173 | 19 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 6 | 11 | 0 |
| 01-085-1327 | 48 | Aluminum | Al | 18 | 5 | 0 |
| 00-043-1022 | 38 | Periclase, syn | Mg O | 2 | 7 | 0 |
| 00-022-1235 | 24 | Elpasolite, syn | K2 Na Al F6 | 6 | 15 | 0 |
| 01-080-1385 | 19 | Aluminum Oxide | Al2.667 O4 | 10 | 11 | 0 |
| 01-085-0930 | 31 | Quartz | Si O2 | 1 | 18 | 0 |

**9.3.4: Peak List of 2609-M**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 11.0271 | 106.72 | 0.5510 | 8.02385 | 0.34 | |
| 18.9736 | 137.21 | 0.1574 | 4.67743 | 0.44 | 01-074-1132; 00-022-1235 |
| 20.8501 | 117.52 | 0.1181 | 4.26051 | 0.38 | 01-085-0930 |
| 22.3149 | 164.78 | 0.1574 | 3.98406 | 0.53 | |
| 24.5188 | 63.06 | 0.1574 | 3.63070 | 0.20 | |
| 25.5722 | 35.54 | 0.2362 | 3.48349 | 0.11 | |
| 26.6616 | 589.51 | 0.0984 | 3.34358 | 1.88 | 01-085-0930 |
| 27.4165 | 1490.28 | 0.1279 | 3.25320 | 4.76 | 01-077-2064 |
| 28.3666 | 17468.05 | 0.1574 | 3.14637 | 55.79 | 00-004-0587 |
| 31.7237 | 17206.39 | 0.1771 | 2.82065 | 54.95 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 33.2442 | 573.62 | 0.1968 | 2.69504 | 1.83 | 01-075-1620 |
| 36.0661 | 568.56 | 0.1574 | 2.49038 | 1.82 | 01-075-1620 |
| 36.9418 | 984.11 | 0.2362 | 2.43334 | 3.14 | 01-074-1132; 00-043-1022 |
| 37.9159 | 1015.45 | 0.1968 | 2.37303 | 3.24 | 01-075-1620 |
| 38.5207 | 31310.20 | 0.1560 | 2.33522 | 100.00 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 38.5978 | 25117.45 | 0.0720 | 2.33653 | 80.22 | |
| 40.5313 | 11725.92 | 0.2280 | 2.22390 | 37.45 | 00-004-0587; 01-085-0930 |
| 41.8019 | 468.69 | 0.2400 | 2.15919 | 1.50 | |
| 42.9720 | 2345.03 | 0.1920 | 2.10307 | 7.49 | 00-043-1022 |
| 44.7760 | 15574.33 | 0.2040 | 2.02244 | 49.74 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.4388 | 12040.88 | 0.1680 | 1.99447 | 38.46 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-1385 |
| 47.4168 | 222.49 | 0.3840 | 1.91578 | 0.71 | |
| 50.2206 | 3870.50 | 0.1800 | 1.81519 | 12.36 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.4305 | 55.64 | 0.5760 | 1.74376 | 0.18 | |
| 53.8529 | 428.85 | 0.1440 | 1.70102 | 1.37 | 01-077-2064 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 54.9566 | 71.53 | 0.2880 | 1.66944 | 0.23 | 01-085-0930 |
| 56.4391 | 3411.50 | 0.1800 | 1.62905 | 10.90 | 01-077-2064; 01-080-2171 |
| 56.6254 | 2092.83 | 0.0960 | 1.62817 | 6.68 | |
| 58.6533 | 1757.27 | 0.0960 | 1.57272 | 5.61 | 00-004-0587 |
| 59.3971 | 641.59 | 0.3840 | 1.55479 | 2.05 | 01-074-1132; 01-075-1620 |
| 62.3101 | 1185.95 | 0.3360 | 1.48891 | 3.79 | 00-043-1022 |
| 65.1589 | 9795.49 | 0.1680 | 1.43054 | 31.29 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 65.3442 | 6634.39 | 0.1440 | 1.43047 | 21.19 | |
| 66.4043 | 4930.29 | 0.1560 | 1.40670 | 15.75 | 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 68.2147 | 113.68 | 0.3360 | 1.37371 | 0.36 | 00-022-1235; 01-085-0930 |
| 71.4365 | 214.83 | 0.2880 | 1.31946 | 0.69 | 01-075-1620; 01-080-2173 |
| 73.7290 | 2420.82 | 0.1920 | 1.28399 | 7.73 | 00-004-0587; 00-022-1235 |
| 73.9363 | 1654.13 | 0.1440 | 1.28409 | 5.28 | |
| 75.2877 | 3662.02 | 0.1080 | 1.26123 | 11.70 | 01-077-2064; 01-080-2171 |
| 75.4827 | 2081.48 | 0.0960 | 1.26158 | 6.65 | |
| 78.2995 | 9333.36 | 0.1560 | 1.22008 | 29.81 | 01-074-1132; 01-085-1327 |
| 78.5343 | 5930.83 | 0.1680 | 1.22005 | 18.94 | |
| 82.4912 | 2623.05 | 0.2400 | 1.16838 | 8.38 | 01-074-1132; 01-085-1327 |
| 82.7939 | 1450.86 | 0.1680 | 1.16777 | 4.63 | |
| 83.9821 | 2285.27 | 0.1920 | 1.15139 | 7.30 | 01-077-2064; 01-085-0930 |
| 84.2358 | 1219.05 | 0.1200 | 1.15143 | 3.89 | |
| 87.6639 | 530.50 | 0.2400 | 1.11227 | 1.69 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0930 |
| 90.3969 | 180.38 | 0.1440 | 1.08561 | 0.58 | 00-022-1235 |
| 94.5101 | 1141.18 | 0.2160 | 1.04891 | 3.64 | 00-004-0587 |

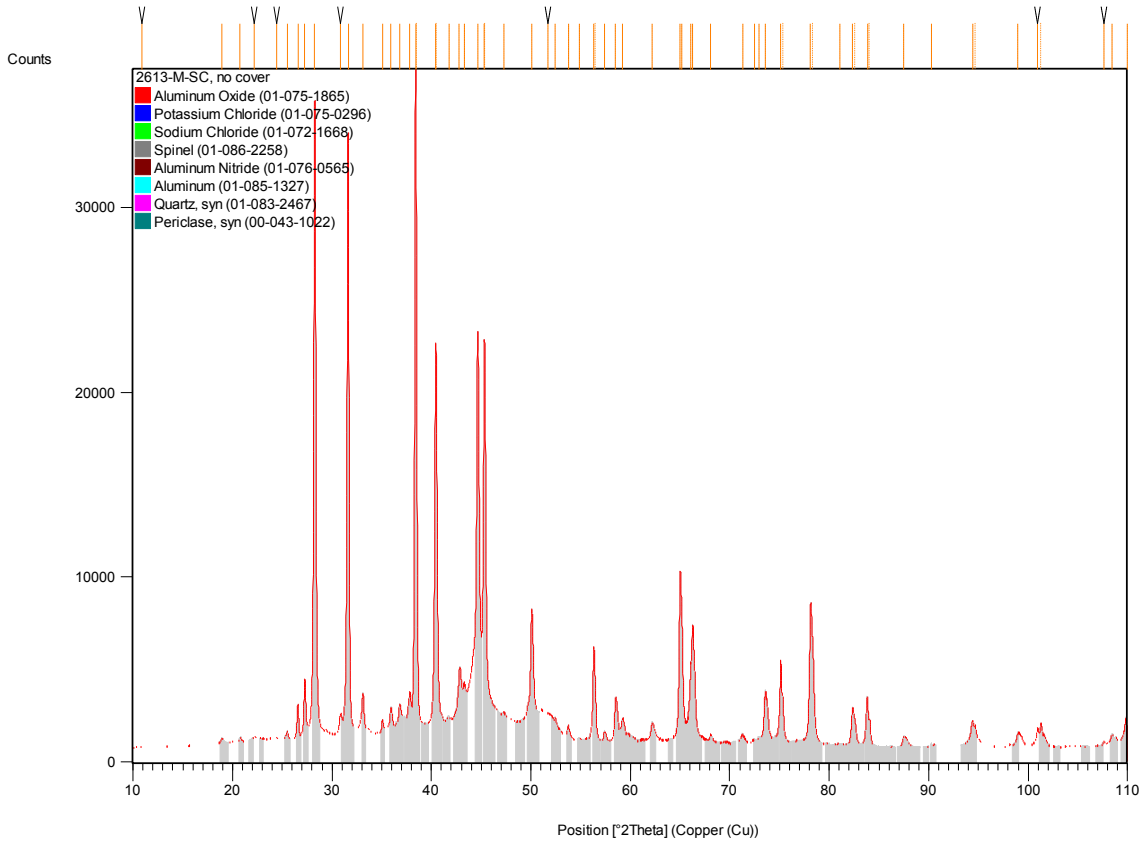
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 94.8816 | 803.37 | 0.1920 | 1.04838 | 2.57 | |
| 99.1251 | 894.93 | 0.1680 | 1.01207 | 2.86 | |
| 99.4947 | 651.96 | 0.1440 | 1.01181 | 2.08 | |
| 101.1521 | 762.15 | 0.0960 | 0.99719 | 2.43 | 00-004-0587 |
| 101.4865 | 1028.08 | 0.1680 | 0.99728 | 3.28 | |
| 107.7619 | 359.21 | 0.1920 | 0.95358 | 1.15 | |
| 108.5761 | 787.44 | 0.2400 | 0.94869 | 2.51 | 00-004-0587 |
| 109.8260 | 1282.58 | 0.0600 | 0.94136 | 4.10 | 00-043-1022 |

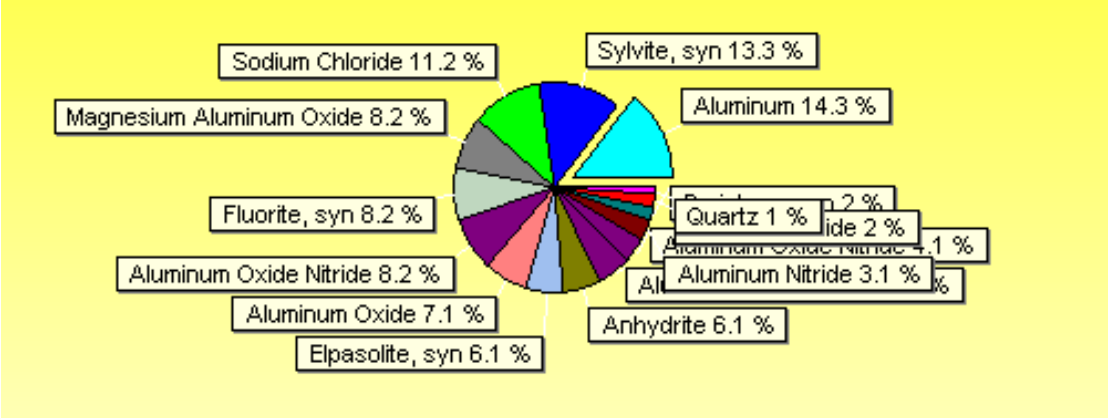
## 9.4: 2613-M-SC

### 9.4.1: Measurement Conditions of 2613-M

| | |
|---|---|
| Dataset Name | 2613-M-SC, no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2613-M-SC, no cover.udf |
| Sample Identification | 2613-M-SC, no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 9.4.2: Main Graphics, Analyze View of 2613-M




**9.4.3: Pattern List of 2613-M**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 31 | Aluminum Oxide | Al2 O3 | 2 | 13 | 0 |
| 00-004-0587 | 60 | Sylvite, syn | K Cl | 13 | 10 | 0 |
| 01-077-2064 | 60 | Sodium Chloride | Na Cl | 11 | 9 | 0 |
| 01-074-1132 | 27 | Magnesium Aluminum Oxide | Mg Al2 O4 | 8 | 10 | 0 |
| 01-075-1620 | 45 | Aluminum Nitride | Al N | 3 | 9 | 0 |
| 01-080-2171 | 19 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 8 | 11 | 0 |
| 01-080-2172 | 18 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 5 | 11 | 0 |
| 01-080-2173 | 16 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 4 | 10 | 0 |
| 01-085-1327 | 54 | Aluminum | Al | 14 | 5 | 0 |
| 00-043-1022 | 38 | Periclase, syn | Mg O | 2 | 7 | 0 |
| 00-004-0864 | 11 | Fluorite, syn | Ca F2 | 8 | 4 | 0 |
| 00-022-1235 | 22 | Elpasolite, syn | K2 Na Al F6 | 6 | 15 | 0 |
| 01-086-2270 | 15 | Anhydrite | Ca ( S O4 ) | 6 | 29 | 0 |
| 01-080-1385 | 19 | Aluminum Oxide | Al2.667 O4 | 7 | 11 | 0 |
| 01-085-0930 | 35 | Quartz | Si O2 | 1 | 20 | 0 |

**9.4.4: Peak List of 2613-M**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 10.8753 | 77.78 | 0.5510 | 8.13545 | 0.22 | |
| 18.9541 | 333.80 | 0.1968 | 4.68221 | 0.93 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.7727 | 396.97 | 0.1574 | 4.27623 | 1.11 | 01-085-0930 |
| 22.2170 | 419.25 | 0.2362 | 4.00138 | 1.17 | |
| 24.4069 | 403.09 | 0.1574 | 3.64710 | 1.12 | |
| 25.5042 | 739.96 | 0.0787 | 3.49262 | 2.06 | 01-075-1862; 01-086-2270 |
| 26.5600 | 2136.08 | 0.1574 | 3.35613 | 5.96 | 01-085-0930 |
| 27.2728 | 3485.10 | 0.0689 | 3.27001 | 9.72 | 01-077-2064 |
| 28.2718 | 34898.57 | 0.1771 | 3.15670 | 97.36 | 00-004-0587; 00-004-0864 |
| 30.8285 | 1658.69 | 0.1574 | 2.90048 | 4.63 | |
| 31.6184 | 33109.27 | 0.1771 | 2.82981 | 92.37 | 01-077-2064; 01-080-2171 |
| 33.1255 | 2715.09 | 0.1771 | 2.70442 | 7.57 | 01-075-1620 |
| 35.0621 | 1274.61 | 0.1378 | 2.55936 | 3.56 | 01-075-1862 |
| 35.9197 | 1982.82 | 0.0886 | 2.50020 | 5.53 | 01-075-1620 |
| 36.8277 | 2143.27 | 0.1279 | 2.44062 | 5.98 | 01-074-1132; 00-043-1022 |
| 37.8248 | 2787.10 | 0.2362 | 2.37854 | 7.78 | 01-075-1862; 01-075-1620 |
| 38.3942 | 35843.59 | 0.1680 | 2.34263 | 100.00 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 38.4825 | 29718.26 | 0.0720 | 2.34326 | 82.91 | |
| 40.4331 | 21572.14 | 0.2160 | 2.22907 | 60.18 | 00-004-0587; 01-085-0930 |
| 40.5197 | 17682.13 | 0.0480 | 2.23003 | 49.33 | |
| 41.7410 | 1493.96 | 0.2640 | 2.16220 | 4.17 | 01-075-1862 |
| 42.8030 | 3984.38 | 0.2400 | 2.11098 | 11.12 | 00-043-1022 |
| 43.2797 | 3268.84 | 0.1920 | 2.08883 | 9.12 | 01-075-1862; 01-086-2270 |
| 44.6402 | 21868.60 | 0.2040 | 2.02827 | 61.01 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3184 | 21623.98 | 0.1560 | 1.99949 | 60.33 | 01-077-2064; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 45.4210 | 17215.03 | 0.0960 | 2.00016 | 48.03 | |
| 47.2957 | 1662.81 | 0.3840 | 1.92040 | 4.64 | 01-086-2270 |
| 50.0962 | 7283.83 | 0.1080 | 1.81941 | 20.32 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 51.7508 | 1639.07 | 0.8640 | 1.76506 | 4.57 | |
| 52.4394 | 1381.99 | 0.2400 | 1.74349 | 3.86 | 01-075-1862; 01-086-2270 |
| 53.7463 | 974.70 | 0.1080 | 1.70414 | 2.72 | 01-077-2064 |
| 54.8338 | 268.47 | 0.2880 | 1.67288 | 0.75 | 01-085-0930 |
| 56.3302 | 5217.73 | 0.1560 | 1.63194 | 14.56 | 01-077-2064 |
| 56.5045 | 2900.22 | 0.1200 | 1.63136 | 8.09 | |
| 57.3798 | 687.38 | 0.1200 | 1.60456 | 1.92 | 01-075-1862; 01-085-0930 |
| 58.5292 | 2506.84 | 0.2160 | 1.57576 | 6.99 | 00-004-0587 |
| 59.2470 | 1359.80 | 0.1920 | 1.55837 | 3.79 | 01-074-1132; 01-075-1620; 00-022-1235; 01-086-2270 |
| 62.2321 | 1107.71 | 0.1680 | 1.49059 | 3.09 | 00-043-1022; 01-086-2270 |
| 64.9783 | 8149.34 | 0.0840 | 1.43408 | 22.74 | 01-085-1327; 00-022-1235 |
| 65.0323 | 9266.65 | 0.0600 | 1.43301 | 25.85 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 65.1830 | 6942.95 | 0.1920 | 1.43007 | 19.37 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 66.0632 | 3970.63 | 0.0720 | 1.41313 | 11.08 | 01-077-2064; 01-075-1620; 01-085-0930 |
| 66.2862 | 6356.56 | 0.1200 | 1.40891 | 17.73 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-1385 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 68.0560 | 408.60 | 0.1920 | 1.37652 | 1.14 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 71.2720 | 501.78 | 0.1920 | 1.32210 | 1.40 | 01-075-1620; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 72.4562 | 238.71 | 0.2880 | 1.30338 | 0.67 | 01-075-1620 |
| 72.9047 | 356.42 | 0.1440 | 1.29646 | 0.99 | 01-077-2064; 01-086-2270 |
| 73.5537 | 2809.07 | 0.1680 | 1.28662 | 7.84 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 75.1236 | 4503.63 | 0.1200 | 1.26358 | 12.56 | 01-077-2064 |
| 75.3626 | 2393.45 | 0.1440 | 1.26330 | 6.68 | |
| 78.1198 | 7426.22 | 0.1800 | 1.22244 | 20.72 | 01-085-1327; 00-022-1235 |
| 78.3976 | 4771.55 | 0.1680 | 1.22183 | 13.31 | |
| 81.0453 | 86.77 | 0.5760 | 1.18553 | 0.24 | 01-075-1620; 01-085-0930 |
| 82.3432 | 2092.80 | 0.2160 | 1.17010 | 5.84 | 01-085-1327; 00-022-1235 |
| 82.6258 | 1241.98 | 0.1920 | 1.16972 | 3.47 | |
| 83.8250 | 2689.96 | 0.1200 | 1.15315 | 7.50 | 01-077-2064; 01-085-0930 |
| 84.0847 | 1454.45 | 0.0960 | 1.15311 | 4.06 | |
| 87.4761 | 585.59 | 0.1200 | 1.11417 | 1.63 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 90.2426 | 176.75 | 0.1920 | 1.08707 | 0.49 | 00-022-1235 |
| 94.4121 | 1392.59 | 0.1680 | 1.04974 | 3.89 | 00-004-0587; 00-004-0864 |
| 94.6972 | 998.42 | 0.1440 | 1.04993 | 2.79 | |
| 98.9376 | 749.04 | 0.1920 | 1.01348 | 2.09 | 00-022-1235 |
| 100.9349 | 960.38 | 0.1680 | 0.99875 | 2.68 | |
| 101.3004 | 1257.34 | 0.1200 | 0.99861 | 3.51 | |
| 107.5799 | 270.18 | 0.1920 | 0.95469 | 0.75 | |
| 108.4313 | 653.04 | 0.2400 | 0.94955 | 1.82 | 00-004-0587 |
| 109.9364 | 1261.24 | 0.0840 | 0.94073 | 3.52 | 00-043-1022 |

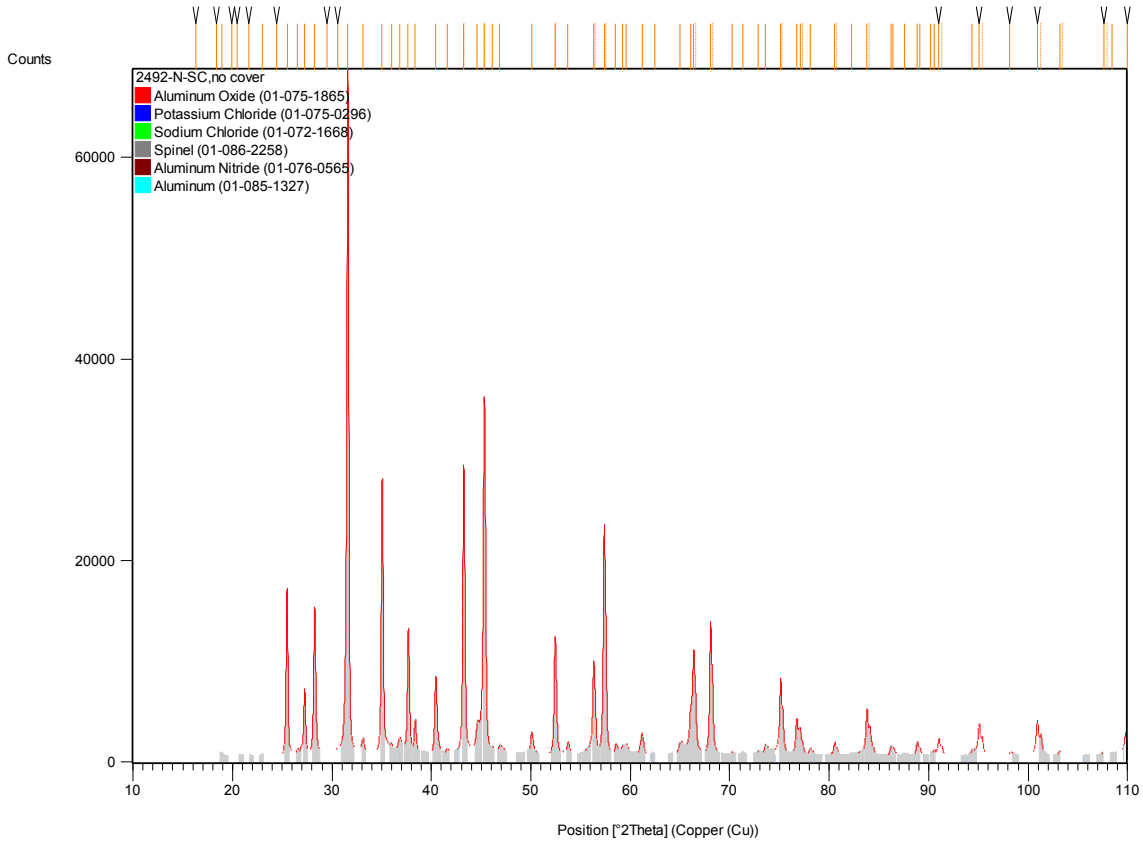
# FACILITY N

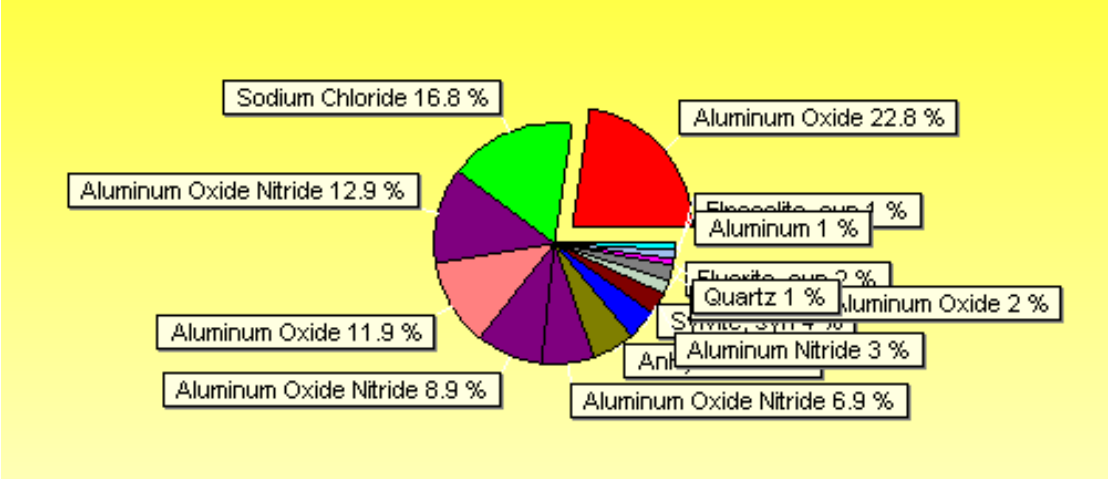
## 10.1: 2492-N-SC

### 10.1.1: Measurement Conditions of 2492-N

| | |
|---|---|
| Dataset Name | 2492-N-SC,no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2492-N-SC,no cover.sd |
| Sample Identification | 2492-N-SC,no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 9/24/2010 6:39:00 PM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 10.1.2: Main Graphics, Analyze View of 2492-N




**10.1.3: Pattern List of 2492-N**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 62 | Aluminum Oxide | Al2 O3 | 23 | 24 | 0 |
| 00-004-0587 | 47 | Sylvite, syn | K Cl | 4 | 10 | 0 |
| 01-077-2064 | 59 | Sodium Chloride | Na Cl | 17 | 9 | 0 |
| 01-074-1132 | 30 | Magnesium Aluminum Oxide | Mg Al2 O4 | 2 | 13 | 0 |
| 01-075-1620 | 27 | Aluminum Nitride | Al N | 3 | 9 | 0 |
| 01-080-2171 | 16 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 13 | 9 | 0 |
| 01-080-2172 | 18 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 9 | 9 | 0 |
| 01-080-2173 | 18 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 7 | 9 | 0 |
| 01-085-1327 | 49 | Aluminum | Al | 1 | 5 | 0 |
| 00-004-0864 | 25 | Fluorite, syn | Ca F2 | 2 | 6 | 0 |
| 00-022-1235 | 23 | Elpasolite, syn | K2 Na Al F6 | 1 | 13 | 0 |
| 01-086-2270 | 21 | Anhydrite | Ca ( S O4 ) | 6 | 34 | 0 |
| 01-080-1385 | 19 | Aluminum Oxide | Al2.667 O4 | 12 | 9 | 0 |
| 01-085-0930 | 20 | Quartz | Si O2 | 1 | 18 | 0 |

**10.1.4: Peak List of 2492-N**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 16.3163 | 81.99 | 0.1574 | 5.43274 | 0.12 | |
| 18.3861 | 248.06 | 0.3149 | 4.82555 | 0.36 | |
| 18.9152 | 388.61 | 0.1181 | 4.69174 | 0.57 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 19.9003 | 179.85 | 0.1574 | 4.46166 | 0.26 | |
| 20.4906 | 103.86 | 0.6298 | 4.33444 | 0.15 | |
| 21.6006 | 117.08 | 0.2362 | 4.11416 | 0.17 | |
| 23.0249 | 155.17 | 0.1968 | 3.86277 | 0.23 | 01-086-2270 |
| 24.4109 | 275.06 | 0.1574 | 3.64651 | 0.40 | |
| 25.4742 | 16493.36 | 0.1673 | 3.49667 | 24.20 | 01-075-1862; 01-086-2270 |
| 26.5467 | 718.20 | 0.1378 | 3.35778 | 1.05 | 01-085-0930 |
| 27.2471 | 6516.87 | 0.1476 | 3.27304 | 9.56 | 01-077-2064 |
| 28.2539 | 14624.11 | 0.1673 | 3.15866 | 21.46 | 00-004-0587; 00-004-0864 |
| 29.5366 | 441.35 | 0.1181 | 3.02434 | 0.65 | |
| 30.5451 | 790.50 | 0.1181 | 2.92675 | 1.16 | |
| 31.5897 | 68156.91 | 0.1771 | 2.83231 | 100.00 | 01-077-2064 |
| 33.1383 | 1671.56 | 0.1771 | 2.70341 | 2.45 | 01-075-1620 |
| 35.0213 | 25634.49 | 0.2066 | 2.56225 | 37.61 | 01-075-1862 |
| 35.9626 | 1130.97 | 0.1968 | 2.49731 | 1.66 | 01-075-1620 |
| 36.8134 | 1739.19 | 0.1968 | 2.44153 | 2.55 | 01-074-1132 |
| 37.6590 | 12190.62 | 0.1378 | 2.38863 | 17.89 | 01-075-1862; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 38.3883 | 3479.72 | 0.1378 | 2.34491 | 5.11 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.4014 | 7320.23 | 0.1771 | 2.23259 | 10.74 | 00-004-0587; 01-085-0930 |
| 41.5952 | 514.61 | 0.1968 | 2.17124 | 0.76 | 01-075-1862 |
| 43.2197 | 27070.55 | 0.1800 | 2.09159 | 39.72 | 01-075-1862; 01-086-2270 |
| 43.3362 | 20988.35 | 0.0960 | 2.09142 | 30.79 | |
| 44.5837 | 2982.66 | 0.1200 | 2.03071 | 4.38 | 01-085-1327; 00-022-1235 |
| 45.3159 | 35338.67 | 0.1920 | 1.99959 | 51.85 | 01-077-2064; 01-086-2270 |
| 45.4338 | 23127.41 | 0.0840 | 1.99963 | 33.93 | |
| 46.1020 | 745.47 | 0.1920 | 1.96731 | 1.09 | 01-075-1862 |
| 46.8599 | 884.29 | 0.2400 | 1.93724 | 1.30 | 00-004-0864; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 50.0799 | 2072.29 | 0.1440 | 1.81996 | 3.04 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.4276 | 11582.11 | 0.1920 | 1.74385 | 16.99 | 01-075-1862; 01-086-2270 |
| 52.5641 | 7777.18 | 0.0840 | 1.74397 | 11.41 | |
| 53.7391 | 1194.56 | 0.1320 | 1.70435 | 1.75 | 01-077-2064 |
| 56.3210 | 9184.80 | 0.1800 | 1.63219 | 13.48 | 01-077-2064 |
| 56.4710 | 5946.84 | 0.0960 | 1.63225 | 8.73 | |
| 57.3781 | 22783.80 | 0.1680 | 1.60460 | 33.43 | 01-075-1862; 01-085-0930 |
| 57.5496 | 12745.58 | 0.1320 | 1.60420 | 18.70 | |
| 58.5210 | 976.79 | 0.0960 | 1.57596 | 1.43 | 00-004-0587 |
| 59.2229 | 926.00 | 0.1680 | 1.55894 | 1.36 | 01-074-1132; 01-075-1620; 00-022-1235; 01-086-2270 |
| 59.6122 | 997.05 | 0.1440 | 1.54969 | 1.46 | 01-075-1862 |
| 61.1847 | 2108.09 | 0.1200 | 1.51358 | 3.09 | 01-075-1862; 01-086-2270 |
| 62.4511 | 148.77 | 0.6720 | 1.48589 | 0.22 | 01-086-2270 |
| 64.9599 | 1112.49 | 0.1200 | 1.43444 | 1.63 | 01-085-1327; 00-022-1235 |
| 66.0894 | 5050.93 | 0.1440 | 1.41263 | 7.41 | 01-077-2064; 01-075-1620 |
| 66.3728 | 10389.96 | 0.1440 | 1.40729 | 15.24 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 66.5734 | 5883.09 | 0.0840 | 1.40702 | 8.63 | |
| 68.0659 | 13139.20 | 0.1560 | 1.37635 | 19.28 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.3043 | 6236.42 | 0.1320 | 1.37553 | 9.15 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 70.2688 | 305.60 | 0.1920 | 1.33849 | 0.45 | 01-075-1862; 01-080-2173; 01-086-2270 |
| 71.2872 | 230.42 | 0.3840 | 1.32185 | 0.34 | 01-075-1620; 01-080-2171; 01-080-2172; 01-080-2173; 01-086-2270; 01-080-1385 |
| 72.8888 | 490.09 | 0.1440 | 1.29671 | 0.72 | 01-077-2064; 01-086-2270 |
| 73.5681 | 1073.82 | 0.1440 | 1.28640 | 1.58 | 00-004-0587; 00-022-1235; 01-085-0930 |
| 75.1355 | 7595.16 | 0.1680 | 1.26341 | 11.14 | 01-077-2064 |
| 75.3286 | 4607.32 | 0.0960 | 1.26378 | 6.76 | |
| 76.7179 | 3559.52 | 0.1440 | 1.24124 | 5.22 | 01-075-1862 |
| 77.0987 | 2472.49 | 0.2400 | 1.23606 | 3.63 | 01-075-1862; 01-086-2270 |
| 77.3242 | 1206.61 | 0.1200 | 1.23608 | 1.77 | |
| 78.1111 | 733.52 | 0.1440 | 1.22255 | 1.08 | 01-085-1327; 00-022-1235 |
| 80.5340 | 1310.14 | 0.1920 | 1.19177 | 1.92 | 01-075-1862 |
| 80.7984 | 757.89 | 0.1200 | 1.19149 | 1.11 | |
| 82.2755 | 267.76 | 0.1920 | 1.17090 | 0.39 | 01-085-1327; 00-022-1235 |
| 83.8006 | 4662.35 | 0.1440 | 1.15342 | 6.84 | 01-077-2064; 01-085-0930 |
| 84.0345 | 3039.65 | 0.1320 | 1.15367 | 4.46 | |
| 86.1757 | 858.33 | 0.1680 | 1.12762 | 1.26 | 01-075-1862 |
| 86.4035 | 877.29 | 0.1920 | 1.12523 | 1.29 | 01-075-1862 |
| 87.5444 | 263.30 | 0.1920 | 1.11348 | 0.39 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 88.8084 | 1303.32 | 0.0960 | 1.10087 | 1.91 | 01-075-1862; 01-086-2270 |
| 89.1034 | 837.97 | 0.1680 | 1.09799 | 1.23 | 01-075-1862 |
| 90.2136 | 405.03 | 0.1920 | 1.08734 | 0.59 | 00-022-1235 |
| 90.5727 | 598.91 | 0.2160 | 1.08396 | 0.88 | 00-022-1235 |
| 91.0402 | 1756.22 | 0.1200 | 1.07961 | 2.58 | |
| 91.3190 | 934.55 | 0.1680 | 1.07972 | 1.37 | |

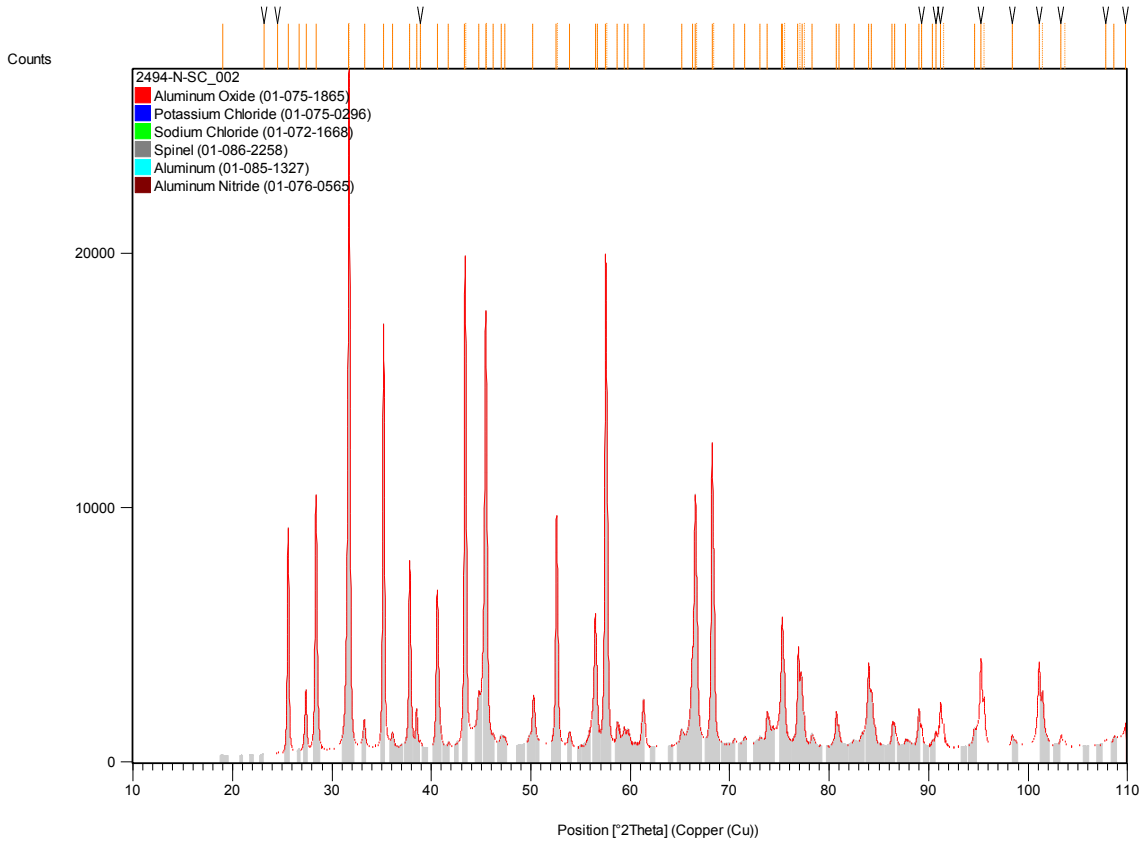
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 94.3868 | 671.47 | 0.1440 | 1.04995 | 0.99 | 00-004-0587; 00-004-0864 |
| 95.0852 | 3333.32 | 0.1680 | 1.04408 | 4.89 | |
| 95.4021 | 1880.05 | 0.1440 | 1.04403 | 2.76 | |
| 98.1606 | 359.21 | 0.2160 | 1.01942 | 0.53 | |
| 100.9480 | 3419.73 | 0.0960 | 0.99866 | 5.02 | |
| 101.2998 | 2067.28 | 0.1320 | 0.99861 | 3.03 | |
| 103.1709 | 417.80 | 0.1920 | 0.98311 | 0.61 | 00-022-1235 |
| 103.4990 | 218.51 | 0.1440 | 0.98332 | 0.32 | |
| 107.6014 | 291.14 | 0.2640 | 0.95456 | 0.43 | |
| 107.9613 | 108.47 | 0.1920 | 0.95474 | 0.16 | |
| 108.4580 | 95.63 | 0.1920 | 0.94939 | 0.14 | 00-004-0587 |
| 109.9257 | 701.65 | 0.1080 | 0.94079 | 1.03 | |

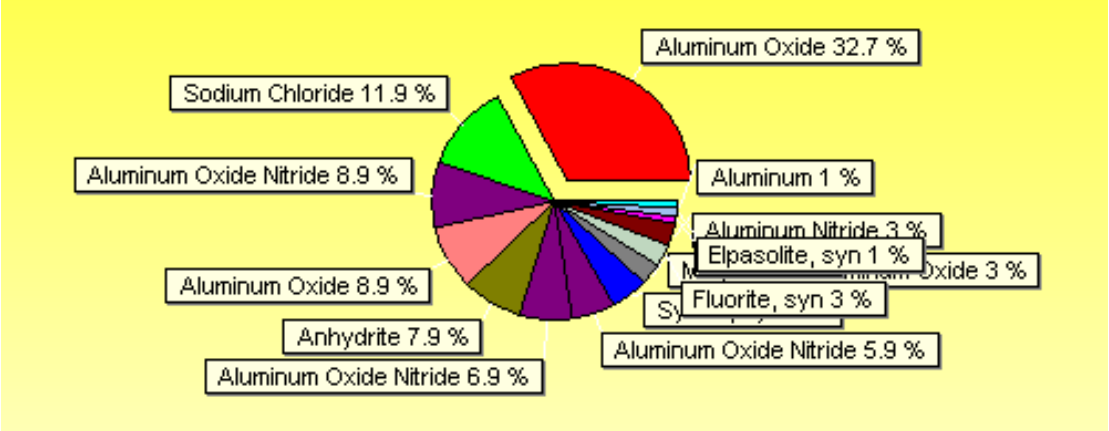
## 10.2: 2494-N-SC

### 10.2.1: Measurement Conditions of 2494-N

| | |
|---|---|
| Dataset Name | 2494-N-SC_002 |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\9-24-10\2494-N-SC_002.sd |
| Sample Identification | 2494-N-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Measurement Date / Time | 8/2/2010 11:34:00 AM |
| Raw Data Origin | PHILIPS-binary (scan) (.SD) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 0.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

### 10.2.2:Main Graphics, Analyze View of 2494-N




**10.2.3: Pattern List of 2494-N**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 67 | Aluminum Oxide | Al2 O3 | 33 | 24 | 0 |
| 00-004-0587 | 54 | Sylvite, syn | K Cl | 5 | 10 | 0 |
| 01-077-2064 | 53 | Sodium Chloride | Na Cl | 12 | 9 | 0 |
| 01-074-1132 | 15 | Magnesium Aluminum Oxide | Mg Al2 O4 | 3 | 11 | 1 |
| 01-075-1620 | 32 | Aluminum Nitride | Al N | 3 | 9 | 0 |
| 01-080-2171 | 14 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 9 | 10 | 0 |
| 01-080-2172 | 14 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 7 | 10 | 0 |
| 01-080-2173 | 14 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 6 | 10 | 0 |
| 01-085-1327 | 45 | Aluminum | Al | 1 | 5 | 0 |
| 00-004-0864 | 25 | Fluorite, syn | Ca F2 | 3 | 6 | 0 |
| 00-022-1235 | 22 | Elpasolite, syn | K2 Na Al F6 | 1 | 14 | 0 |
| 01-086-2270 | 24 | Anhydrite | Ca ( S O4 ) | 8 | 34 | 0 |
| 01-080-1385 | 15 | Aluminum Oxide | Al2.667 O4 | 9 | 10 | 0 |
| 01-085-0930 | 5 | Quartz | Si O2 | 1 | 15 | 0 |

**10.2.4: Peak List of 2494-N**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9723 | 25.88 | 0.6298 | 4.67774 | 0.10 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.1407 | 44.03 | 0.3149 | 3.84371 | 0.17 | |
| 24.5492 | 92.12 | 0.1378 | 3.62629 | 0.35 | |
| 25.5838 | 8748.90 | 0.1574 | 3.48194 | 33.14 | 01-075-1862; 01-086-2270 |
| 26.6718 | 113.39 | 0.1181 | 3.34231 | 0.43 | 01-085-0930 |
| 27.3708 | 2425.94 | 0.0886 | 3.25852 | 9.19 | 01-077-2064 |
| 28.3735 | 9778.12 | 0.1279 | 3.14562 | 37.04 | 00-004-0587; 00-004-0864 |
| 31.6969 | 26401.62 | 0.1440 | 2.82064 | 100.00 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 31.7861 | 20534.50 | 0.0480 | 2.81991 | 77.78 | |
| 33.2816 | 1128.99 | 0.0960 | 2.68986 | 4.28 | 01-075-1620 |
| 35.1617 | 16163.75 | 0.1800 | 2.55023 | 61.22 | 01-075-1862 |
| 36.0583 | 626.85 | 0.1200 | 2.48884 | 2.37 | 01-075-1620 |
| 37.7833 | 7106.66 | 0.1440 | 2.37909 | 26.92 | 01-075-1862; 01-075-1620 |
| 38.4854 | 1505.70 | 0.2160 | 2.33728 | 5.70 | 01-074-1132; 01-085-1327; 00-022-1235; 01-086-2270 |
| 38.8903 | 264.50 | 0.2400 | 2.31388 | 1.00 | |
| 40.5618 | 6132.01 | 0.1320 | 2.22230 | 23.23 | 00-004-0587 |
| 41.7195 | 244.78 | 0.1920 | 2.16327 | 0.93 | 01-075-1862 |
| 43.3512 | 17981.77 | 0.1680 | 2.08555 | 68.11 | 01-075-1862; 01-086-2270 |
| 43.4833 | 13238.04 | 0.0600 | 2.08468 | 50.14 | |
| 44.7659 | 2216.87 | 0.1680 | 2.02287 | 8.40 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.4443 | 17004.93 | 0.1680 | 1.99424 | 64.41 | 01-077-2064; 01-080-2171; 01-086-2270; 01-080-1385 |
| 45.6012 | 9261.42 | 0.0960 | 1.99268 | 35.08 | |
| 46.2207 | 551.71 | 0.1920 | 1.96253 | 2.09 | 01-075-1862 |
| 46.9765 | 485.81 | 0.2400 | 1.93270 | 1.84 | 00-004-0864; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 47.3755 | 404.28 | 0.1920 | 1.91735 | 1.53 | 01-086-2270 |
| 50.2057 | 1844.62 | 0.2880 | 1.81570 | 6.99 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-085-0930 |
| 52.5438 | 8418.80 | 0.1560 | 1.74027 | 31.89 | 01-075-1862 |
| 52.7167 | 5729.39 | 0.0720 | 1.73928 | 21.70 | |
| 53.8731 | 631.84 | 0.1440 | 1.70043 | 2.39 | 01-077-2064 |
| 56.4827 | 5279.75 | 0.0840 | 1.62790 | 20.00 | 01-077-2064; 01-080-2171 |
| 56.6369 | 3194.93 | 0.1200 | 1.62383 | 12.10 | 01-077-2064; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 57.5038 | 18670.98 | 0.1320 | 1.60139 | 70.72 | 01-075-1862 |
| 57.6727 | 12290.50 | 0.0600 | 1.60107 | 46.55 | |
| 58.6679 | 888.05 | 0.2160 | 1.57236 | 3.36 | 00-004-0587 |
| 59.3732 | 740.18 | 0.1920 | 1.55536 | 2.80 | 01-074-1132; 01-075-1620 |
| 59.7429 | 709.07 | 0.1440 | 1.54661 | 2.69 | 01-075-1862 |
| 61.3327 | 1883.23 | 0.1320 | 1.51028 | 7.13 | 01-075-1862 |
| 65.1340 | 687.92 | 0.1680 | 1.43102 | 2.61 | 01-074-1132; 01-085-1327 |
| 66.2228 | 3622.68 | 0.1200 | 1.41011 | 13.72 | 00-004-0587; 01-077-2064; 01-075-1620 |
| 66.5075 | 9731.17 | 0.1680 | 1.40476 | 36.86 | 01-075-1862; 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 66.7162 | 5968.09 | 0.0720 | 1.40436 | 22.61 | |
| 68.2126 | 11705.47 | 0.1440 | 1.37374 | 44.34 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.4058 | 7403.69 | 0.1200 | 1.37374 | 28.04 | |
| 70.4454 | 338.50 | 0.1920 | 1.33557 | 1.28 | 01-075-1862 |
| 71.4996 | 432.59 | 0.2160 | 1.31845 | 1.64 | 01-075-1620; 01-086-2270 |
| 73.0558 | 403.59 | 0.1440 | 1.29415 | 1.53 | 01-077-2064; 01-086-2270 |
| 73.7507 | 1319.28 | 0.1920 | 1.28367 | 5.00 | 00-004-0587; 00-022-1235 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 75.2366 | 4682.80 | 0.0600 | 1.26196 | 17.74 | 01-077-2064 |
| 75.2926 | 5116.57 | 0.0720 | 1.26116 | 19.38 | 01-077-2064 |
| 75.5121 | 2969.84 | 0.1200 | 1.26117 | 11.25 | |
| 76.8400 | 3261.92 | 0.1440 | 1.23958 | 12.36 | 01-075-1862 |
| 77.1324 | 2953.03 | 0.0960 | 1.23867 | 11.19 | |
| 77.2465 | 2683.80 | 0.1440 | 1.23407 | 10.17 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 77.5034 | 1179.51 | 0.1200 | 1.23367 | 4.47 | |
| 78.2420 | 496.48 | 0.1680 | 1.22084 | 1.88 | 01-074-1132; 01-085-1327 |
| 80.7027 | 1413.24 | 0.0840 | 1.18970 | 5.35 | 01-075-1862 |
| 80.9567 | 840.02 | 0.1200 | 1.18661 | 3.18 | 01-075-1620; 01-085-0930 |
| 82.4669 | 275.58 | 0.3840 | 1.16866 | 1.04 | 01-074-1132; 01-085-1327 |
| 83.9432 | 3156.34 | 0.1200 | 1.15183 | 11.96 | 01-077-2064; 01-085-0930 |
| 84.2266 | 2243.89 | 0.1680 | 1.14867 | 8.50 | 01-075-1862; 01-080-2171; 01-080-2172; 01-086-2270; 01-080-1385 |
| 86.3274 | 837.93 | 0.1680 | 1.12603 | 3.17 | 01-075-1862 |
| 86.5787 | 861.88 | 0.1440 | 1.12340 | 3.26 | 01-075-1862 |
| 87.6659 | 316.48 | 0.1680 | 1.11225 | 1.20 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385; 01-085-0930 |
| 89.0015 | 1438.41 | 0.0840 | 1.09899 | 5.45 | 01-075-1862 |
| 89.3042 | 813.15 | 0.1440 | 1.09604 | 3.08 | |
| 90.3913 | 294.43 | 0.1440 | 1.08567 | 1.12 | 00-022-1235 |
| 90.7064 | 563.12 | 0.1200 | 1.08271 | 2.13 | |
| 91.1941 | 1750.05 | 0.1680 | 1.07819 | 6.63 | |
| 91.5051 | 910.28 | 0.1680 | 1.07801 | 3.45 | |
| 94.5773 | 703.63 | 0.1680 | 1.04834 | 2.67 | 00-004-0587 |
| 95.2765 | 3422.06 | 0.1680 | 1.04249 | 12.96 | |
| 95.5712 | 1949.04 | 0.1440 | 1.04264 | 7.38 | |
| 98.3968 | 461.87 | 0.1920 | 1.01760 | 1.75 | |
| 101.0981 | 3297.08 | 0.0720 | 0.99758 | 12.49 | |
| 101.4594 | 2079.88 | 0.1680 | 0.99747 | 7.88 | |
| 103.3218 | 463.80 | 0.1680 | 0.98208 | 1.76 | |
| 103.6939 | 197.67 | 0.1440 | 0.98201 | 0.75 | |

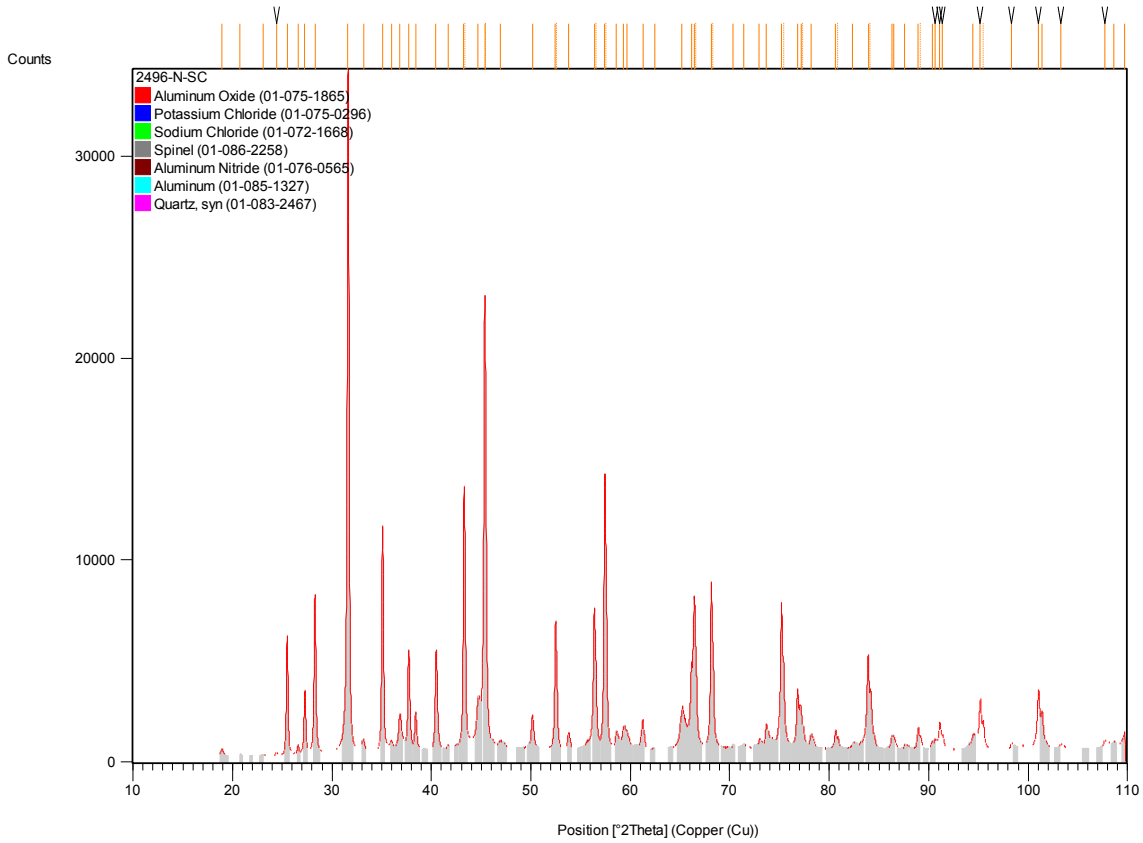
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 107.7621 | 296.26 | 0.1920 | 0.95358 | 1.12 | |
| 108.6384 | 373.90 | 0.2400 | 0.94832 | 1.42 | 00-004-0587 |
| 109.8280 | 1241.94 | 0.0480 | 0.94135 | 4.70 | |

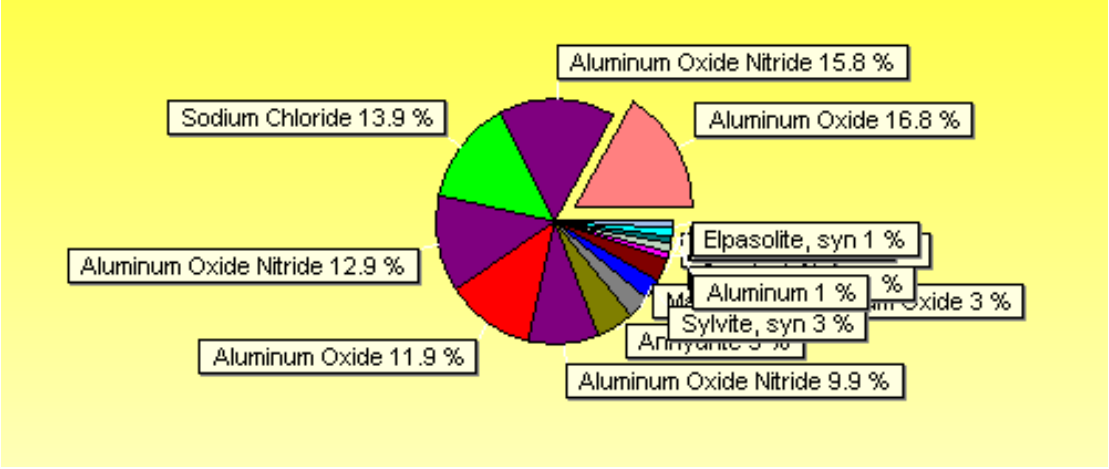
## 10.3: 2496-N-SC

### 10.3.1: Measurement Conditions of 2496-N

| | |
|---|---|
| Dataset Name | 2496-N-SC |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2496-N-SC.udf |
| Sample Identification | 2496-N-SC |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 10.3.2: Main Graphics, Analyze View OF 2496-N




**10.3.3: Pattern List OF 2496-N**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 43 | Aluminum Oxide | Al2 O3 | 12 | 21 | 0 |
| 00-004-0587 | 43 | Sylvite, syn | K Cl | 3 | 10 | 0 |
| 01-077-2064 | 55 | Sodium Chloride | Na Cl | 14 | 9 | 0 |
| 01-074-1132 | 35 | Magnesium Aluminum Oxide | Mg Al2 O4 | 3 | 13 | 0 |
| 01-075-1620 | 20 | Aluminum Nitride | Al N | 3 | 7 | 0 |
| 01-080-2171 | 12 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 16 | 10 | 0 |
| 01-080-2172 | 15 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 13 | 11 | 0 |
| 01-080-2173 | 15 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 10 | 11 | 0 |
| 01-085-1327 | 35 | Aluminum | Al | 1 | 5 | 0 |
| 00-043-1022 | 8 | Periclase, syn | Mg O | 1 | 6 | 1 |
| 00-004-0864 | 21 | Fluorite, syn | Ca F2 | 1 | 6 | 0 |
| 00-022-1235 | 17 | Elpasolite, syn | K2 Na Al F6 | 1 | 13 | 0 |
| 01-086-2270 | 17 | Anhydrite | Ca ( S O4 ) | 5 | 35 | 0 |
| 01-080-1385 | 15 | Aluminum Oxide | Al2.667 O4 | 17 | 10 | 0 |
| 01-085-0930 | 11 | Quartz | Si O2 | 1 | 18 | 0 |

**10.3.4: Peak List of 2496-N**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9545 | 377.81 | 0.1968 | 4.68211 | 1.13 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 20.7728 | 84.99 | 0.1574 | 4.27619 | 0.26 | 01-085-0930 |
| 23.0584 | 28.40 | 0.2362 | 3.85724 | 0.09 | 01-086-2270 |
| 24.4553 | 144.65 | 0.1378 | 3.64000 | 0.43 | |
| 25.4777 | 5649.54 | 0.1476 | 3.49620 | 16.97 | 01-075-1862; 01-086-2270 |
| 26.5677 | 443.78 | 0.1378 | 3.35517 | 1.33 | 01-085-0930 |
| 27.2798 | 3153.87 | 0.0886 | 3.26919 | 9.47 | 01-077-2064 |
| 28.2751 | 7611.40 | 0.1673 | 3.15633 | 22.86 | 00-004-0587; 00-004-0864 |
| 31.6065 | 33298.05 | 0.1771 | 2.83085 | 100.00 | 01-077-2064 |
| 33.1971 | 522.15 | 0.0984 | 2.69876 | 1.57 | 01-075-1620 |
| 35.0561 | 10459.47 | 0.1574 | 2.55978 | 31.41 | 01-075-1862 |
| 35.9535 | 492.08 | 0.1574 | 2.49793 | 1.48 | 01-075-1620 |
| 36.8105 | 1794.80 | 0.0886 | 2.44171 | 5.39 | 01-074-1132; 00-043-1022 |
| 37.6800 | 4649.32 | 0.1378 | 2.38735 | 13.96 | 01-075-1862; 01-080-2172; 01-080-2173 |
| 38.4279 | 1910.33 | 0.1673 | 2.34259 | 5.74 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.4386 | 4599.88 | 0.1574 | 2.23063 | 13.81 | 00-004-0587; 01-085-0930 |
| 41.6561 | 227.47 | 0.1378 | 2.16821 | 0.68 | 01-075-1862 |
| 43.2661 | 12638.05 | 0.1440 | 2.08945 | 37.95 | 01-075-1862; 01-086-2270 |
| 43.3594 | 10675.91 | 0.0600 | 2.09035 | 32.06 | |
| 44.7001 | 2595.87 | 0.1920 | 2.02569 | 7.80 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3495 | 21846.23 | 0.1560 | 1.99818 | 65.61 | 01-077-2064; 01-086-2270 |
| 45.4594 | 17034.76 | 0.0960 | 1.99856 | 51.16 | |
| 46.9340 | 406.30 | 0.2400 | 1.93435 | 1.22 | 00-004-0864; 01-086-2270 |
| 50.1338 | 1588.05 | 0.1920 | 1.81813 | 4.77 | 00-004-0587; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385; 01-085-0930 |
| 52.4748 | 6229.23 | 0.1320 | 1.74240 | 18.71 | 01-075-1862 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 52.6312 | 3712.31 | 0.1200 | 1.74191 | 11.15 | |
| 53.7861 | 813.58 | 0.1680 | 1.70297 | 2.44 | 01-077-2064 |
| 56.3718 | 6858.29 | 0.1440 | 1.63084 | 20.60 | 01-077-2064 |
| 56.5450 | 4314.63 | 0.0960 | 1.63029 | 12.96 | |
| 57.4075 | 12885.75 | 0.1440 | 1.60385 | 38.70 | 01-075-1862; 01-085-0930 |
| 57.5935 | 8233.12 | 0.1320 | 1.60309 | 24.73 | |
| 58.5949 | 894.19 | 0.1680 | 1.57415 | 2.69 | 00-004-0587 |
| 59.3301 | 1138.95 | 0.1680 | 1.55638 | 3.42 | 01-074-1132; 01-075-1620 |
| 59.6724 | 890.63 | 0.1440 | 1.54827 | 2.67 | 01-075-1862 |
| 61.2506 | 1453.20 | 0.1440 | 1.51211 | 4.36 | 01-075-1862; 01-086-2270 |
| 62.4304 | 42.14 | 0.5760 | 1.48634 | 0.13 | 00-043-1022 |
| 65.2055 | 1994.67 | 0.1440 | 1.42963 | 5.99 | 01-074-1132; 01-085-1327 |
| 66.1491 | 4283.99 | 0.1200 | 1.41150 | 12.87 | 01-077-2064; 01-075-1620 |
| 66.4597 | 7591.04 | 0.1200 | 1.40566 | 22.80 | 01-075-1862; 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 01-080-1385 |
| 66.6416 | 4000.28 | 0.1200 | 1.40575 | 12.01 | |
| 68.1371 | 8155.08 | 0.1560 | 1.37508 | 24.49 | 01-075-1862; 00-022-1235; 01-085-0930 |
| 68.3405 | 4741.32 | 0.0960 | 1.37489 | 14.24 | |
| 70.3216 | 220.17 | 0.2880 | 1.33762 | 0.66 | 01-075-1862; 01-086-2270 |
| 71.4325 | 236.99 | 0.3360 | 1.31952 | 0.71 | 01-075-1620; 01-086-2270 |
| 72.9792 | 493.47 | 0.1440 | 1.29532 | 1.48 | 01-077-2064; 01-086-2270 |
| 73.6705 | 1173.76 | 0.1680 | 1.28487 | 3.53 | 00-004-0587; 00-022-1235 |
| 75.2164 | 7216.69 | 0.1320 | 1.26225 | 21.67 | 01-077-2064 |
| 75.4566 | 3822.54 | 0.1200 | 1.26196 | 11.48 | |
| 76.8311 | 2989.00 | 0.0840 | 1.23970 | 8.98 | 01-075-1862 |
| 77.1798 | 1997.73 | 0.1440 | 1.23496 | 6.00 | 01-075-1862; 01-074-1132; 01-086-2270 |
| 77.3990 | 1139.55 | 0.1200 | 1.23507 | 3.42 | |
| 78.1740 | 736.44 | 0.2400 | 1.22173 | 2.21 | 01-085-1327; 00-022-1235 |

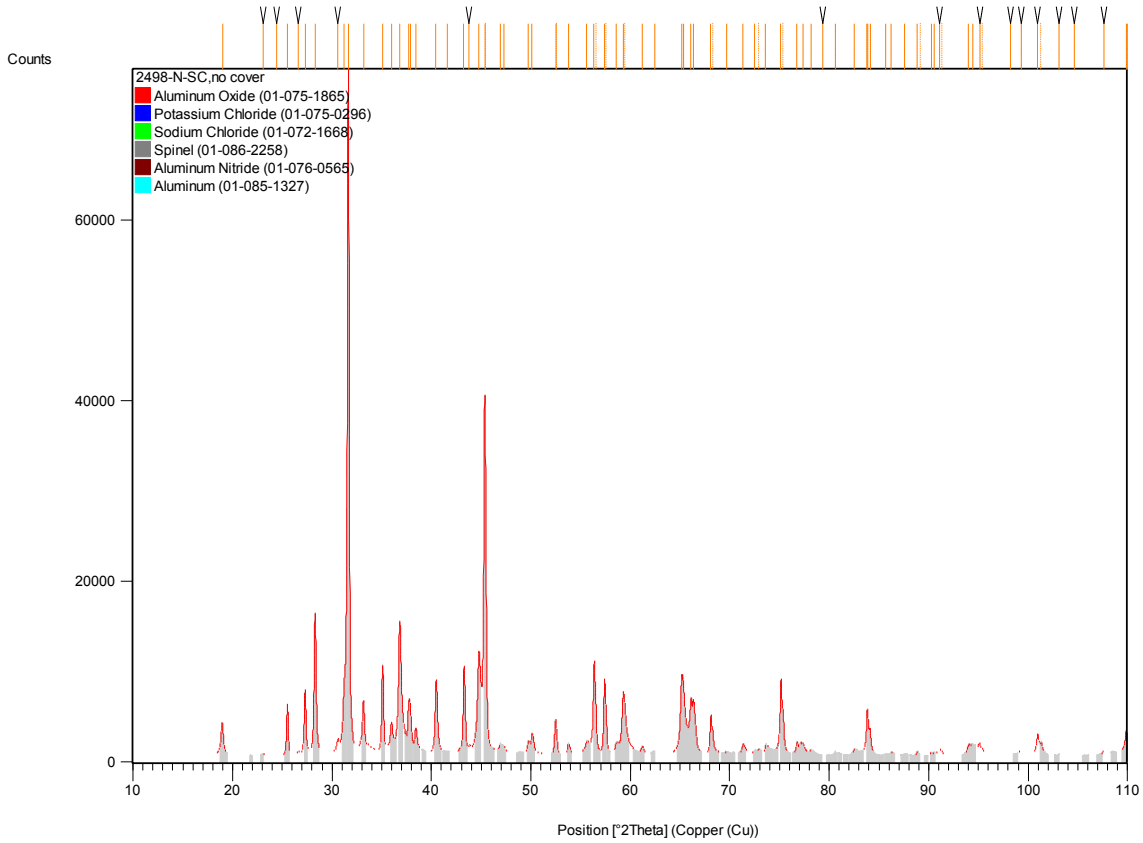
| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 80.6277 | 956.38 | 0.1440 | 1.19062 | 2.87 | 01-075-1862 |
| 80.8620 | 623.40 | 0.0960 | 1.19071 | 1.87 | |
| 82.3565 | 301.14 | 0.2880 | 1.16995 | 0.90 | 01-085-1327; 00-022-1235 |
| 83.9281 | 4680.93 | 0.1560 | 1.15200 | 14.06 | 01-077-2064; 01-085-0930 |
| 84.1731 | 2936.27 | 0.1200 | 1.15212 | 8.82 | |
| 86.2669 | 638.85 | 0.1440 | 1.12666 | 1.92 | 01-075-1862 |
| 86.5019 | 638.17 | 0.1920 | 1.12420 | 1.92 | 01-075-1862 |
| 87.5945 | 231.62 | 0.2400 | 1.11297 | 0.70 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385; 01-085-0930 |
| 88.9248 | 1083.03 | 0.1200 | 1.09973 | 3.25 | 01-075-1862; 01-086-2270 |
| 89.2208 | 606.42 | 0.1440 | 1.09958 | 1.82 | |
| 90.3401 | 431.48 | 0.0960 | 1.08615 | 1.30 | 00-022-1235 |
| 90.6808 | 511.42 | 0.1680 | 1.08295 | 1.54 | |
| 91.1224 | 1352.95 | 0.0960 | 1.07885 | 4.06 | |
| 91.4012 | 748.18 | 0.1440 | 1.07629 | 2.25 | |
| 94.4753 | 745.70 | 0.1920 | 1.04920 | 2.24 | 00-004-0587; 00-004-0864 |
| 95.1693 | 2464.44 | 0.0960 | 1.04338 | 7.40 | |
| 95.5075 | 1424.04 | 0.0960 | 1.04316 | 4.28 | |
| 98.3281 | 329.45 | 0.1680 | 1.01813 | 0.99 | |
| 101.0591 | 2977.73 | 0.0960 | 0.99786 | 8.94 | |
| 101.4194 | 1883.12 | 0.2160 | 0.99529 | 5.66 | 00-004-0587 |
| 103.2657 | 334.81 | 0.1680 | 0.98246 | 1.01 | |
| 107.6696 | 619.00 | 0.2160 | 0.95414 | 1.86 | |
| 108.5997 | 699.60 | 0.2400 | 0.94855 | 2.10 | 00-004-0587 |
| 109.7364 | 1408.40 | 0.0480 | 0.94188 | 4.23 | 00-043-1022 |

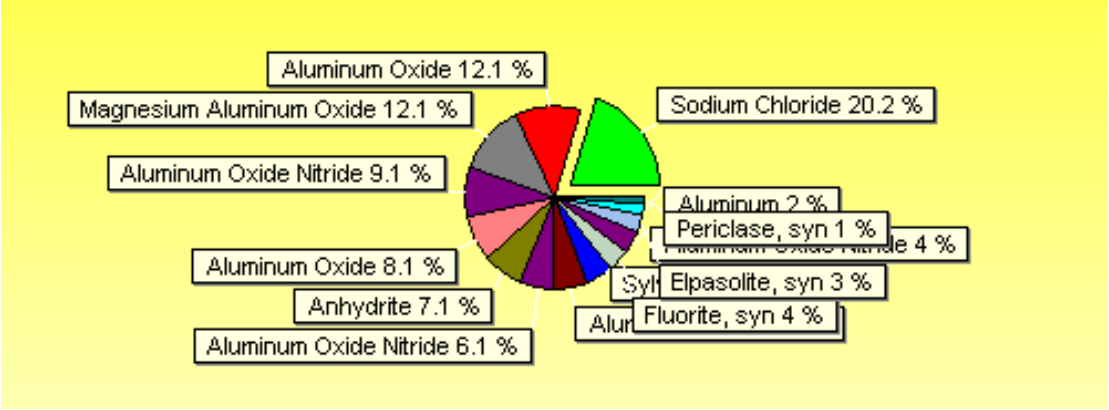
## 10.4: 2498-N-SC

### 10.4.1: Measurement Conditions of 2498-N

| | |
|---|---|
| Dataset Name | 2498-N-SC,no cover |
| File name | C:\Documents and Settings\xhuang\Desktop\SAP-xrd\SC-no cover\2498-N-SC,no cover.udf |
| Sample Identification | 2498-N-SC,no cover |
| Comment | Exported by X'Pert SW |
| Generated by Ford in project SAP. | |
| Raw Data Origin | PHILIPS-ASCII (.UDF) |
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 110.0000 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Divergence Slit Type | Automatic |
| Irradiated Length [mm] | 20.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 2.0000 |
| Measurement Temperature [°C] | 25.00 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPERT |
| Diffractometer Number | 1 |
| Goniometer Radius [mm] | 240.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | No |

### 10.4.2: Main Graphics, Analyze View of 2498-N


Counts
2498-N-SC,no cover
Aluminum Oxide (01-075-1865)
Potassium Chloride (01-075-0296)
Sodium Chloride (01-072-1668)
Spinel (01-086-2258)
Aluminum Nitride (01-076-0565)
Aluminum (01-085-1327)
60000
40000
20000
0
10
20
30
40
50
60
70
80
90
100
110
Position [°2Theta] (Copper (Cu))


Aluminum Oxide 12.1 %
Magnesium Aluminum Oxide 12.1 %
Sodium Chloride 20.2 %
Aluminum Oxide Nitride 9.1 %
Periclase, syn 1 %
Aluminum Oxide 8.1 %
Elpasolite, syn 3 %
Anhydrite 7.1 %
Fluorite, syn 4 %
Aluminum Oxide Nitride 6.1 %

**10.4.3: Pattern List of 2498-N**

| Ref. Code | Score | Compound Name | Chemical Formula | SemiQuant [%] | Matched Lines | Strong Unmatched Lines |
|---|---|---|---|---|---|---|
| 01-075-1862 | 59 | Aluminum Oxide | Al2 O3 | 12 | 22 | 0 |
| 00-004-0587 | 41 | Sylvite, syn | K Cl | 5 | 10 | 0 |
| 01-077-2064 | 48 | Sodium Chloride | Na Cl | 20 | 9 | 0 |
| 01-074-1132 | 53 | Magnesium Aluminum Oxide | Mg Al2 O4 | 12 | 15 | 0 |
| 01-075-1620 | 49 | Aluminum Nitride | Al N | 6 | 11 | 0 |
| 01-080-2171 | 17 | Aluminum Oxide Nitride | Al2.85 O3.45 N0.55 | 9 | 12 | 0 |
| 01-080-2172 | 18 | Aluminum Oxide Nitride | Al2.81 O3.56 N0.44 | 6 | 12 | 0 |
| 01-080-2173 | 19 | Aluminum Oxide Nitride | Al2.78 O3.65 N0.35 | 4 | 12 | 0 |
| 01-085-1327 | 29 | Aluminum | Al | 2 | 5 | 0 |
| 00-043-1022 | 10 | Periclase, syn | Mg O | 1 | 7 | 0 |
| 00-004-0864 | 20 | Fluorite, syn | Ca F2 | 4 | 5 | 0 |
| 00-022-1235 | 29 | Elpasolite, syn | K2 Na Al F6 | 3 | 17 | 0 |
| 01-086-2270 | 22 | Anhydrite | Ca ( S O4 ) | 7 | 38 | 0 |
| 01-080-1385 | 18 | Aluminum Oxide | Al2.667 O4 | 8 | 12 | 0 |

**10.4.4: Peak List of 2498-N**

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 18.9736 | 3575.44 | 0.0492 | 4.67744 | 4.77 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 23.0855 | 186.52 | 0.1574 | 3.85278 | 0.25 | |
| 24.4619 | 265.83 | 0.1574 | 3.63903 | 0.35 | |
| 25.5046 | 5490.32 | 0.1080 | 3.48968 | 7.32 | 01-075-1862; 01-086-2270 |
| 25.5568 | 5031.29 | 0.0360 | 3.49132 | 6.71 | |
| 26.5823 | 595.19 | 0.1200 | 3.35059 | 0.79 | |
| 27.3156 | 7241.97 | 0.1440 | 3.26228 | 9.66 | 01-077-2064 |
| 28.3223 | 15843.06 | 0.1800 | 3.14857 | 21.14 | 00-004-0587; 00-004-0864 |
| 30.5653 | 1827.60 | 0.1680 | 2.92244 | 2.44 | |
| 31.1937 | 7266.05 | 0.1440 | 2.86498 | 9.69 | 01-074-1132; 00-022-1235; 01-086-2270 |
| 31.6288 | 74958.65 | 0.2280 | 2.82656 | 100.00 | 01-077-2064 |
| 33.1889 | 5973.09 | 0.1440 | 2.69717 | 7.97 | 01-075-1620 |
| 35.1095 | 10026.84 | 0.1440 | 2.55390 | 13.38 | 01-075-1862 |
| 35.9841 | 3708.59 | 0.0960 | 2.49380 | 4.95 | 01-075-1620 |
| 36.7778 | 14048.43 | 0.1200 | 2.44178 | 18.74 | 01-074-1132; 00-043-1022 |
| 36.8301 | 14967.21 | 0.1080 | 2.43844 | 19.97 | 01-074-1132; 00-043-1022 |
| 37.7110 | 6025.68 | 0.0720 | 2.38348 | 8.04 | 01-075-1862; 01-080-2173 |
| 37.8823 | 5432.35 | 0.1920 | 2.37309 | 7.25 | 01-075-1862; 01-075-1620 |
| 38.4273 | 3022.09 | 0.2640 | 2.34068 | 4.03 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 40.4129 | 6828.79 | 0.0480 | 2.23014 | 9.11 | 00-004-0587 |
| 40.4627 | 8341.99 | 0.0480 | 2.23304 | 11.13 | |
| 41.6043 | 506.07 | 0.2880 | 2.16899 | 0.68 | 01-075-1862 |
| 43.2605 | 9465.14 | 0.1440 | 2.08971 | 12.63 | 01-075-1862; 01-086-2270 |
| 43.8080 | 1265.80 | 0.0720 | 2.06486 | 1.69 | |
| 44.7313 | 10465.37 | 0.1440 | 2.02435 | 13.96 | 01-074-1132; 01-085-1327; 00-022-1235 |
| 45.3572 | 39866.75 | 0.2160 | 1.99786 | 53.18 | 01-077-2064; 01-086-2270 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 45.4902 | 23286.25 | 0.0720 | 1.99728 | 31.07 | |
| 46.9545 | 1347.14 | 0.1680 | 1.93355 | 1.80 | 00-004-0864; 01-086-2270 |
| 47.3219 | 1055.61 | 0.1680 | 1.91940 | 1.41 | 01-086-2270 |
| 49.7617 | 1639.23 | 0.1440 | 1.83085 | 2.19 | 01-075-1620 |
| 50.1119 | 2379.38 | 0.0960 | 1.81888 | 3.17 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-022-1235; 01-080-1385 |
| 52.4982 | 3881.65 | 0.1440 | 1.74168 | 5.18 | 01-075-1862 |
| 52.6032 | 2469.82 | 0.0960 | 1.74277 | 3.29 | |
| 53.7729 | 1412.15 | 0.0840 | 1.70336 | 1.88 | 01-077-2064 |
| 55.6125 | 1629.71 | 0.2880 | 1.65129 | 2.17 | 01-074-1132; 00-004-0864; 01-086-2270 |
| 56.3407 | 9749.35 | 0.1680 | 1.63166 | 13.01 | 01-077-2064 |
| 56.5512 | 5435.00 | 0.1080 | 1.63013 | 7.25 | |
| 57.4141 | 8478.55 | 0.1440 | 1.60368 | 11.31 | 01-075-1862 |
| 57.5813 | 4667.90 | 0.0960 | 1.60340 | 6.23 | |
| 58.5765 | 1513.53 | 0.2400 | 1.57460 | 2.02 | 00-004-0587 |
| 59.2816 | 6816.08 | 0.2160 | 1.55754 | 9.09 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 59.4395 | 5811.97 | 0.1200 | 1.55764 | 7.75 | |
| 61.2350 | 1009.99 | 0.1080 | 1.51245 | 1.35 | 01-075-1862; 01-086-2270 |
| 62.4293 | 475.51 | 0.1920 | 1.48636 | 0.63 | 00-043-1022 |
| 65.1702 | 8801.68 | 0.1200 | 1.43032 | 11.74 | 01-074-1132; 01-085-1327 |
| 65.3505 | 7846.01 | 0.1440 | 1.42681 | 10.47 | 01-074-1132; 01-086-2270 |
| 66.1013 | 6379.70 | 0.0960 | 1.41241 | 8.51 | 01-077-2064; 01-075-1620 |
| 66.3551 | 6126.87 | 0.2400 | 1.40762 | 8.17 | 01-075-1862; 00-004-0587; 01-077-2064; 01-080-2171; 01-080-1385 |
| 68.1006 | 4506.46 | 0.1320 | 1.37573 | 6.01 | 01-075-1862; 00-022-1235 |
| 68.2971 | 2673.63 | 0.0960 | 1.37566 | 3.57 | |
| 69.6617 | 436.17 | 0.1920 | 1.34867 | 0.58 | 01-074-1132; 01-075-1620 |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 71.3553 | 1297.00 | 0.1920 | 1.32076 | 1.73 | 01-075-1620; 01-080-2173; 01-086-2270 |
| 72.5221 | 633.96 | 0.1920 | 1.30236 | 0.85 | 01-075-1620 |
| 72.9320 | 804.88 | 0.0960 | 1.29927 | 1.07 | |
| 73.5834 | 1150.84 | 0.1440 | 1.28617 | 1.54 | 00-004-0587; 00-022-1235 |
| 75.1491 | 8364.28 | 0.1560 | 1.26321 | 11.16 | 01-077-2064 |
| 75.3704 | 4975.27 | 0.0840 | 1.26318 | 6.64 | |
| 76.7562 | 1575.11 | 0.0960 | 1.24072 | 2.10 | 01-075-1862 |
| 77.3306 | 1407.18 | 0.1680 | 1.23293 | 1.88 | 01-074-1132 |
| 78.1541 | 678.24 | 0.1920 | 1.22199 | 0.90 | 01-085-1327; 00-022-1235 |
| 79.3808 | 198.86 | 0.1440 | 1.20616 | 0.27 | |
| 80.6048 | 464.76 | 0.1920 | 1.19090 | 0.62 | 01-075-1862 |
| 82.5277 | 764.95 | 0.1440 | 1.16796 | 1.02 | 01-074-1132; 01-085-1327; 01-086-2270 |
| 83.8185 | 5053.95 | 0.0840 | 1.15322 | 6.74 | 01-077-2064 |
| 83.8737 | 5009.65 | 0.0720 | 1.15261 | 6.68 | 01-077-2064 |
| 84.0965 | 3013.24 | 0.1440 | 1.15012 | 4.02 | 01-075-1862; 01-077-2064; 01-086-2270 |
| 85.6995 | 208.78 | 0.3840 | 1.13266 | 0.28 | 01-074-1132; 01-075-1620; 01-086-2270 |
| 86.2182 | 349.59 | 0.1920 | 1.12717 | 0.47 | 01-075-1862 |
| 87.5487 | 233.78 | 0.2400 | 1.11344 | 0.31 | 00-004-0587; 01-080-2171; 01-080-2172; 01-080-2173; 00-004-0864; 01-080-1385 |
| 88.8778 | 467.97 | 0.1680 | 1.10019 | 0.62 | 01-075-1862; 01-086-2270 |
| 89.1654 | 245.83 | 0.1440 | 1.10012 | 0.33 | |
| 90.2458 | 427.21 | 0.1200 | 1.08704 | 0.57 | 00-022-1235 |
| 90.5426 | 412.61 | 0.1440 | 1.08425 | 0.55 | 00-022-1235 |
| 91.0857 | 830.01 | 0.1200 | 1.07919 | 1.11 | |
| 91.3368 | 470.28 | 0.1440 | 1.07955 | 0.63 | |
| 93.9720 | 1143.02 | 0.2400 | 1.05349 | 1.52 | 00-043-1022 |
| 94.4249 | 1288.91 | 0.1920 | 1.04963 | 1.72 | 00-004-0587; 00-004-0864 |
| 95.1199 | 1461.80 | 0.1920 | 1.04379 | 1.95 | |
| 95.4011 | 810.94 | 0.1440 | 1.04404 | 1.08 | |
| 99.2707 | 620.05 | 0.3360 | 1.01097 | 0.83 | |

| Pos. [°2Th.] | Height [cts] | FWHM [°2Th.] | d-spacing [Å] | Rel. Int. [%] | Matched by |
|---|---|---|---|---|---|
| 100.9469 | 2304.68 | 0.2160 | 0.99866 | 3.07 | |
| 103.1483 | 191.31 | 0.1920 | 0.98326 | 0.26 | |
| 104.6818 | 98.64 | 0.4800 | 0.97302 | 0.13 | |
| 107.6057 | 593.61 | 0.2160 | 0.95453 | 0.79 | |
| 109.8479 | 2774.35 | 0.0840 | 0.94124 | 3.70 | 00-043-1022 |
| 109.9444 | 2114.96 | 0.0720 | 0.94068 | 2.82 | 00-043-1022 |

*Secondary Aluminum Processing Waste: Salt Cake Characterization and Reactivity*

## 10. APPENDIX D: Temperature Profiles of Salt Cake

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## Facility B

Facility B has four samples, which were collected on Feb 24, 2010 and March 19, 2010, respectively. The highest temperature increase was Sample 2478-B-SC (27.8 °C @ 50 °C, and 14.9 °C @ 37 °C).

**Table 10-1: The Temperature Pattern of Facility B**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2452-B-SC | 10.4 | 2.6 | 13.2 | 1.3 |
| 2454-B-SC | 8.5 | 2.3 | 13.1 | 1.2 |

| | | | | |
|---|---|---|---|---|
| 2468-B-SC | 11.2 | 1.5 | 18.2 | 0.8 |
| 2478-B-SC | 14.9 | 2.2 | 27.7 | 0.7 |
| Maximum | 14.9 | 2.6 | 27.7 | 1.3 |
| Minimum | 8.5 | 1.5 | 13.1 | 0.7 |
| Average | 11.3 | 2.1 | 18.1 | 1.0 |

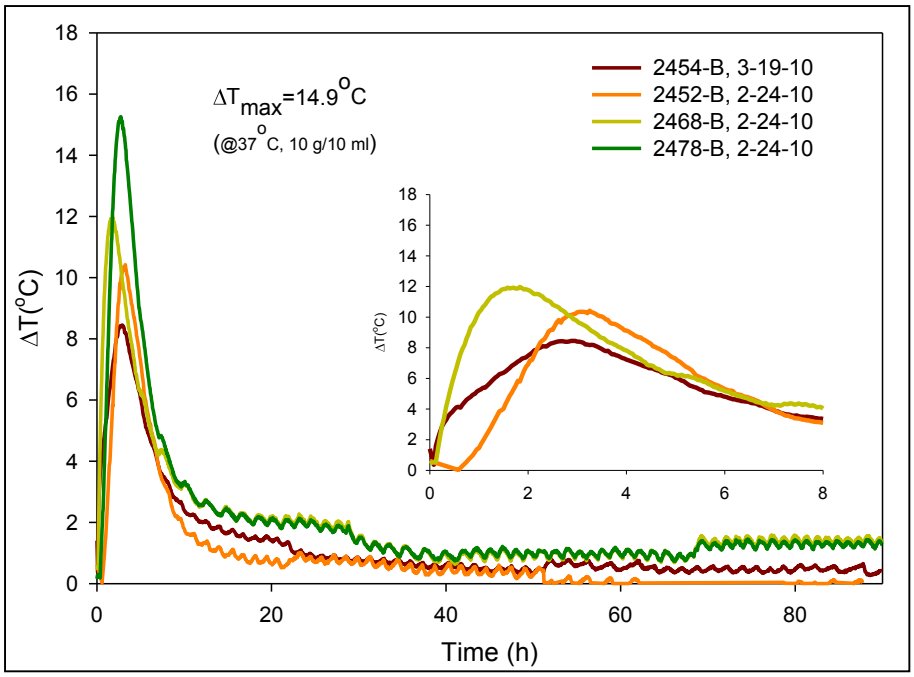



**Figure 10-1: Temperature profile of four salt cakes from Facility B at 37 °C.**

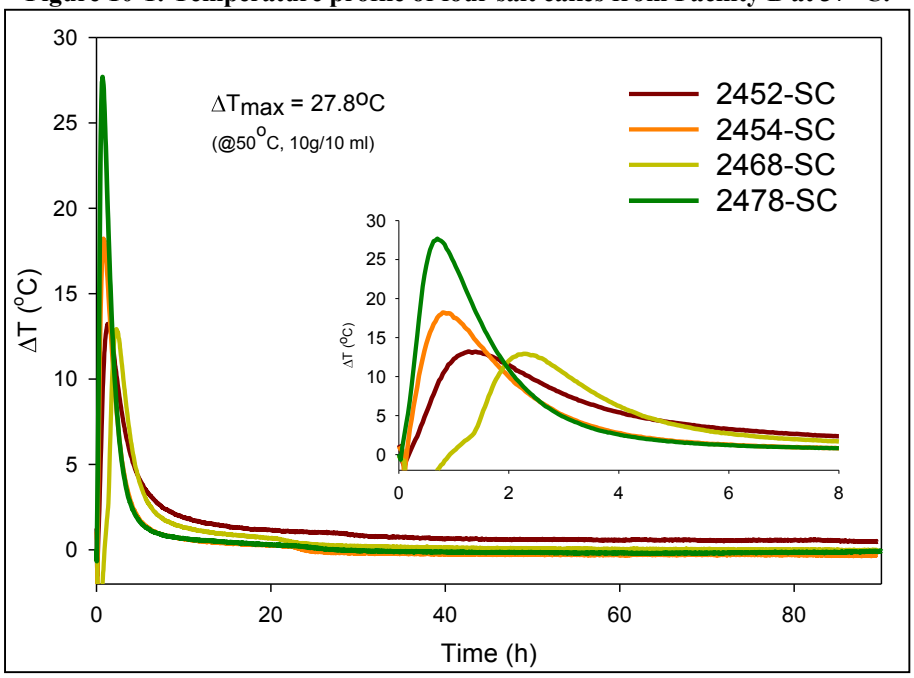



**Figure 10-2: Temperature profile of four salt cakes from Facility B at 50 °C.**

## Facility C

Facility C has two samples, which were collected on November 25 and December 28, 2009, respectively. The highest temperature increase was Sample 2435-C-SC (6.9 °C @ 50 °C, and 14.5 °C @ 37 °C).

**Table 10-2: The Temperature Pattern of Facility C**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max\text{-}T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max\text{-}T}$ (h) |
| 2433-C-SC | 3.4 | 4.1 | 4.4 | 2.0 |
| 2535-C-SC | 10.0 | 0.9 | 6.9 | 2.1 |
| Maximum | 10.0 | 4.1 | 6.9 | 2.1 |
| Minimum | 3.4 | 0.9 | 4.4 | 2.0 |
| Average | 6.7 | 2.5 | 5.7 | 2.0 |

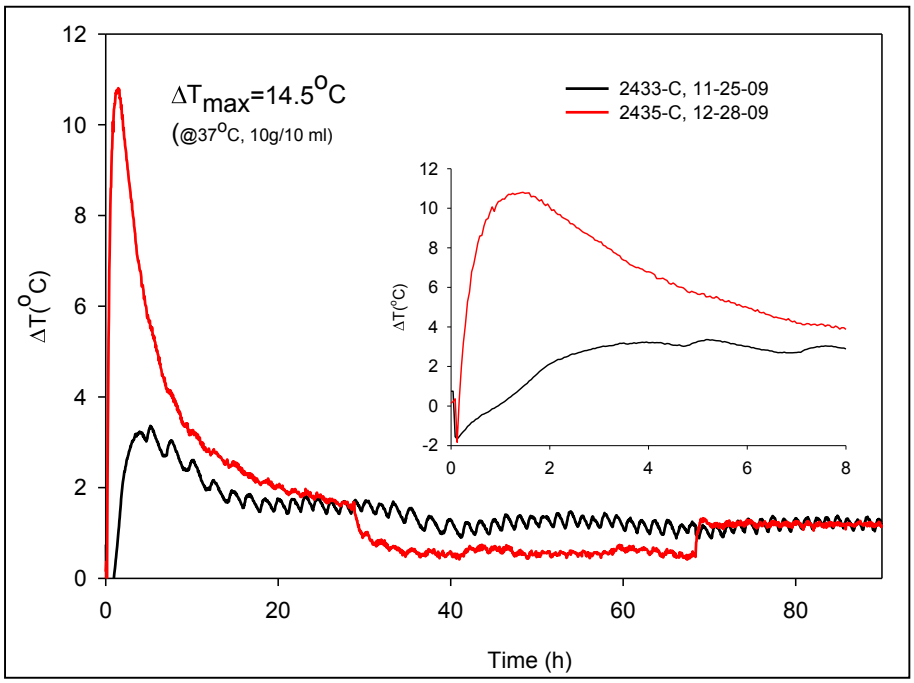



**Figure 10-3: Temperature profile of two salt cakes from Facility C at 37 °C.**

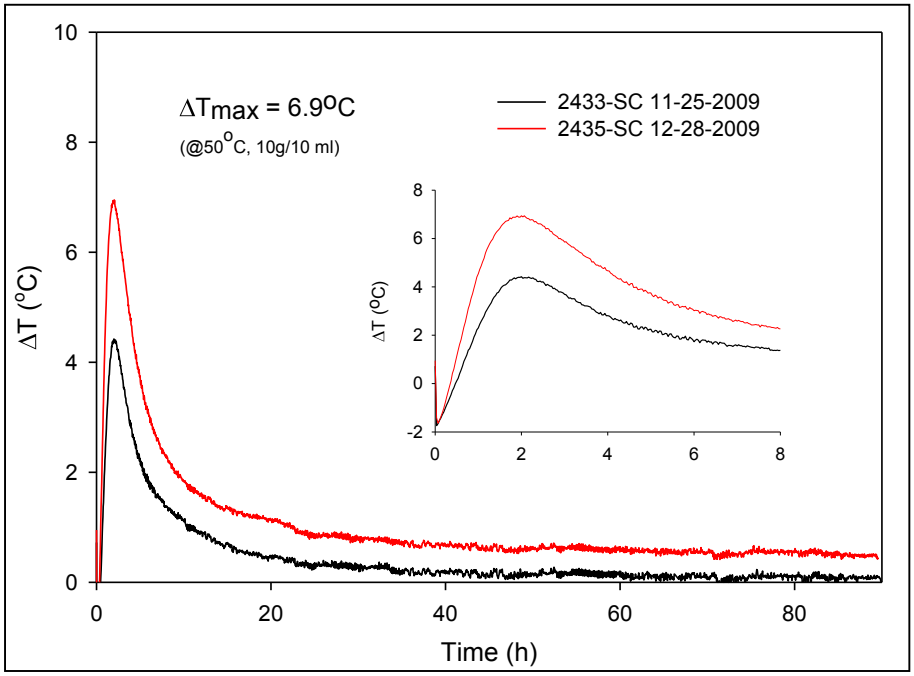



**Figure 10-4: Temperature profile of two salt cakes from Facility C at 50 °C.**

## Facility D

Facility D has four samples, which were collected on November 5, 2009, and January 15, February 24, and March 19, 2010, respectively. The highest temperature increase was Sample 2538-D-SC @ 50°C (20.4 °C) and Sample 2536-D-SC @ 37°C (14.4 °C).

**Table 10-3: The Temperature Pattern of Facility D**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2532-D-SC | 11.8 | 1.4 | 17.7 | 0.5 |
| 2536-D-SC | 14.4 | 1.7 | 17.1 | 0.7 |
| 2538-D-SC | 11.8 | 0.8 | 20.4 | 0.3 |
| 2540-D-SC | 10.4 | 1.7 | 17.7 | 0.6 |
| Maximum | 14.4 | 1.7 | 20.4 | 0.7 |
| Minimum | 10.4 | 0.8 | 17.1 | 0.3 |
| Average | 12.1 | 1.4 | 18.2 | 0.5 |

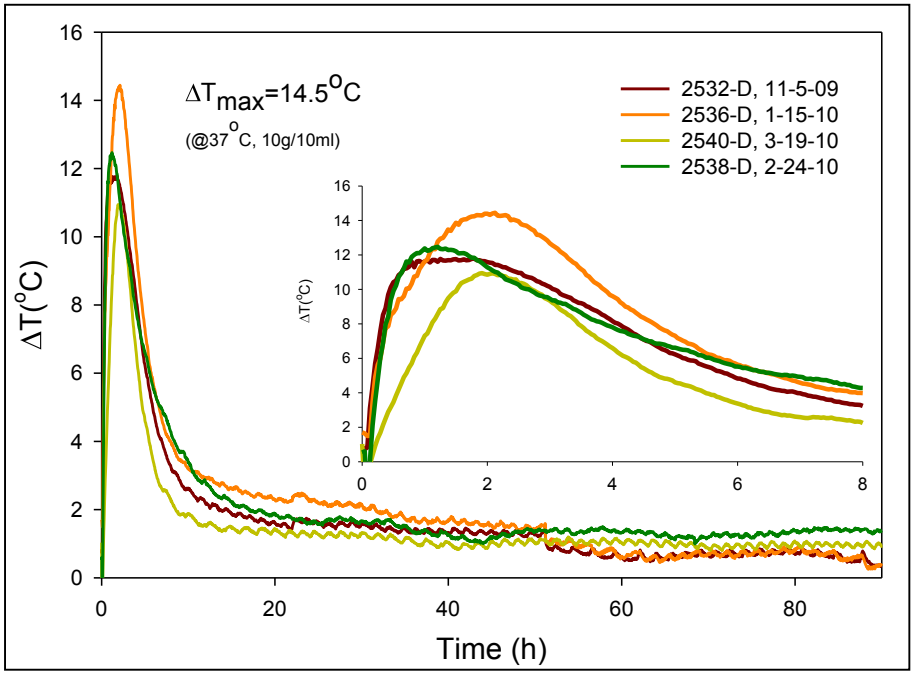



**Figure 10-5: Temperature profile of four salt cakes from Facility D at 37 °C.**

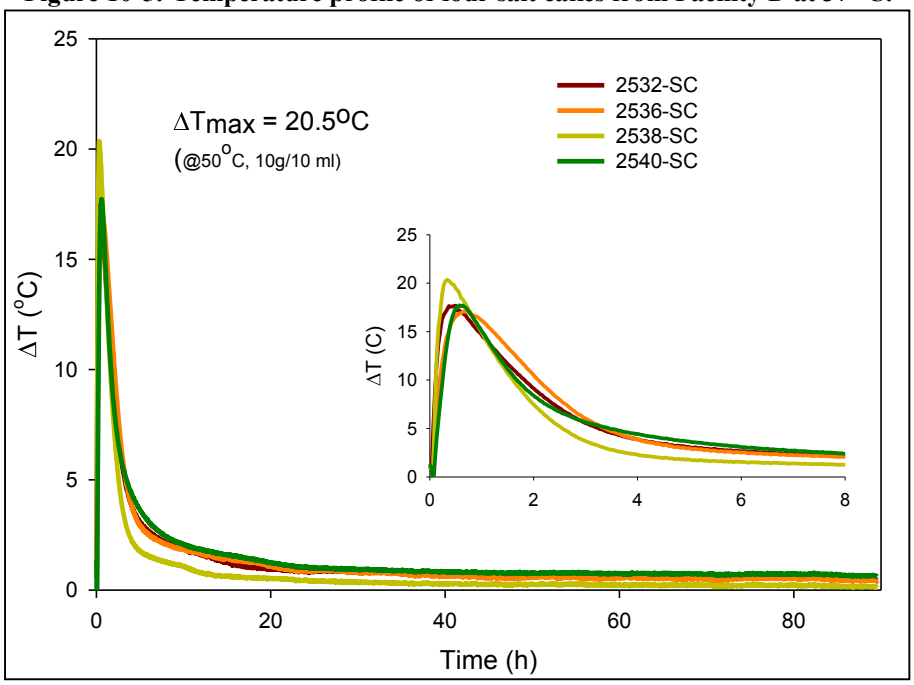



**Figure 10-6: Temperature profile of four salt cakes from Facility D at 50 °C.**

## Facility E

Facility E has two samples, which were collected on February 24 and 25, 2010, respectively. Their behaviors were very similar. The higher temperature increase was Sample 2437-E-SC (23.2 °C @ 50 °C) and Sample 2439-E-SC (16.1 °C @ 37 °C).

**Table 10-4: The Temperature Pattern of Facility E**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2437-E-SC | 16.0 | 1.0 | 23.2 | 0.7 |
| 2439-E-SC | 16.1 | 1.2 | 22.6 | 0.9 |
| Maximum | 16.1 | 1.2 | 23.2 | 0.9 |
| Minimum | 16.0 | 1.0 | 22.6 | 0.7 |
| Average | 16.1 | 1.1 | 22.9 | 0.8 |

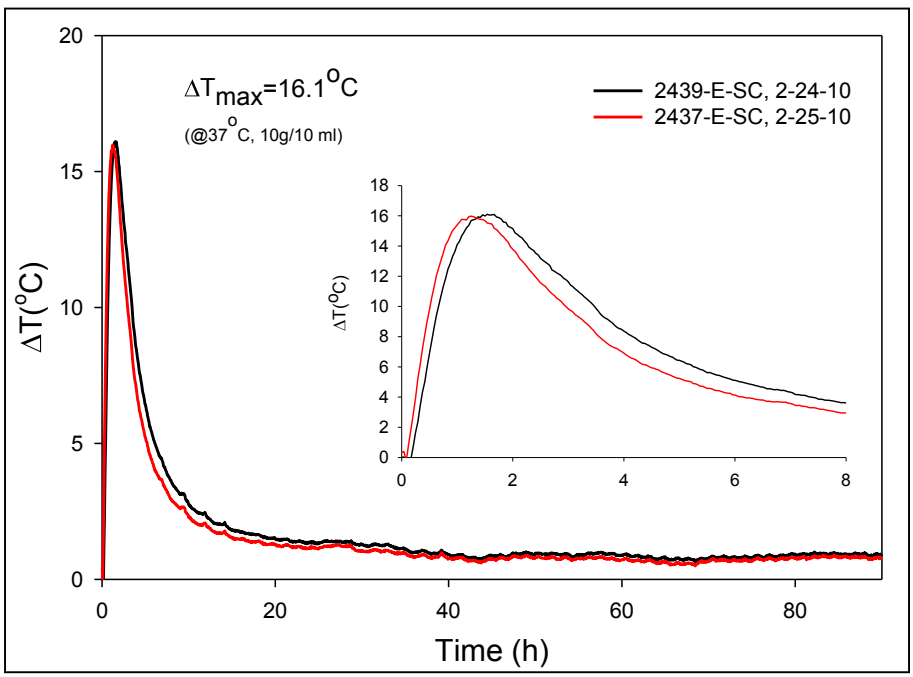



**Figure 10-7: Temperature profile of two salt cakes from Facility E at 37 °C.**

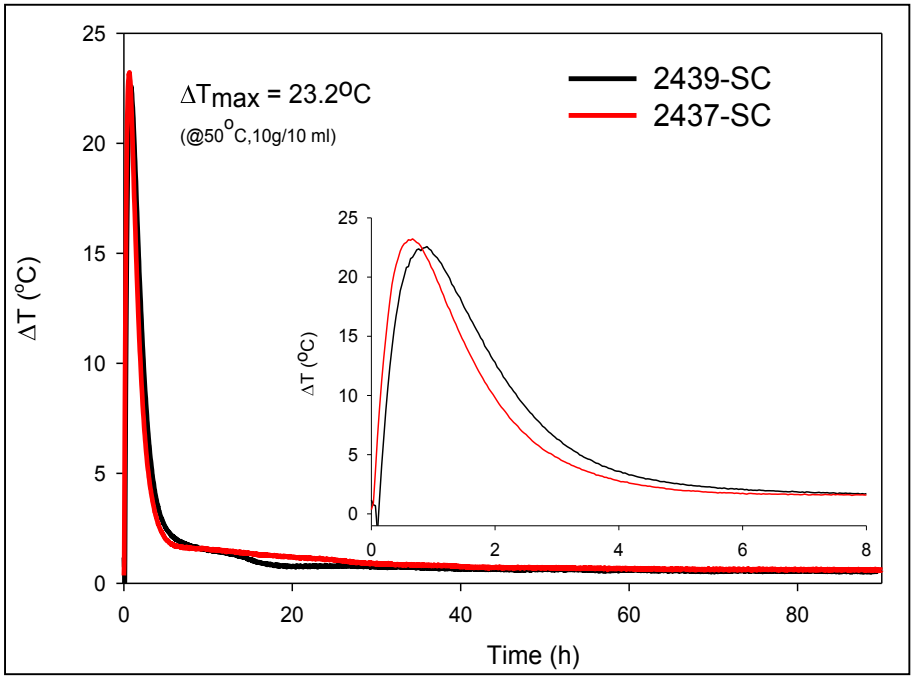



**Figure 10-8: Temperature profile of two salt cakes from Facility E at 50 °C.**

## Facility F

Facility F has five samples, which were collected from November 20, 2009 to April 6, 2010. They were very similar and the strongest temperature response was sample 2510-F-SC, which had more than 50 °C increase, both under 37 °C and 50 °C environmental conditions.

**Table 10-5: The Temperature Pattern of Facility F**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2502-F-SC | 50.9 | 1.1 | 48.7 | 0.6 |
| 2504-F-SC | 48.0 | 0.9 | 44.8 | 0.5 |
| 2506-F-SC | 52.9 | 0.7 | 47.4 | 0.4 |
| 2508-F-SC | 39.4 | 1.2 | 48.3 | 0.5 |
| 2510-F-SC | 56.1 | 0.8 | 52.4 | 0.4 |
| Maximum | 56.1 | 1.2 | 52.4 | 0.6 |
| Minimum | 39.4 | 0.7 | 44.8 | 0.4 |
| Average | 49.5 | 0.9 | 48.3 | 0.5 |

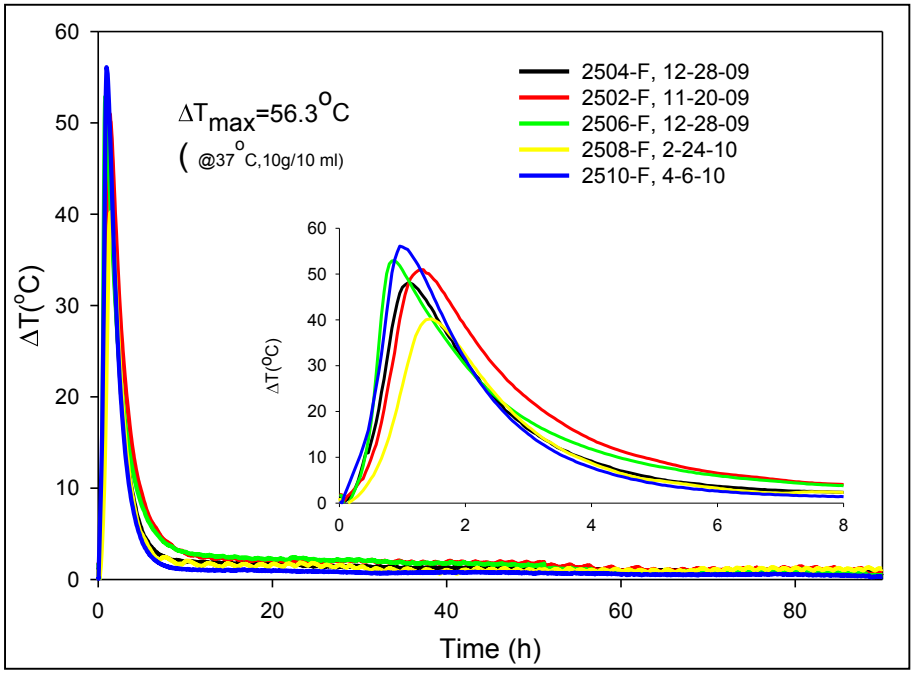



**Figure 10-9: Temperature profile of five salt cakes from Facility F at 37 °C.**

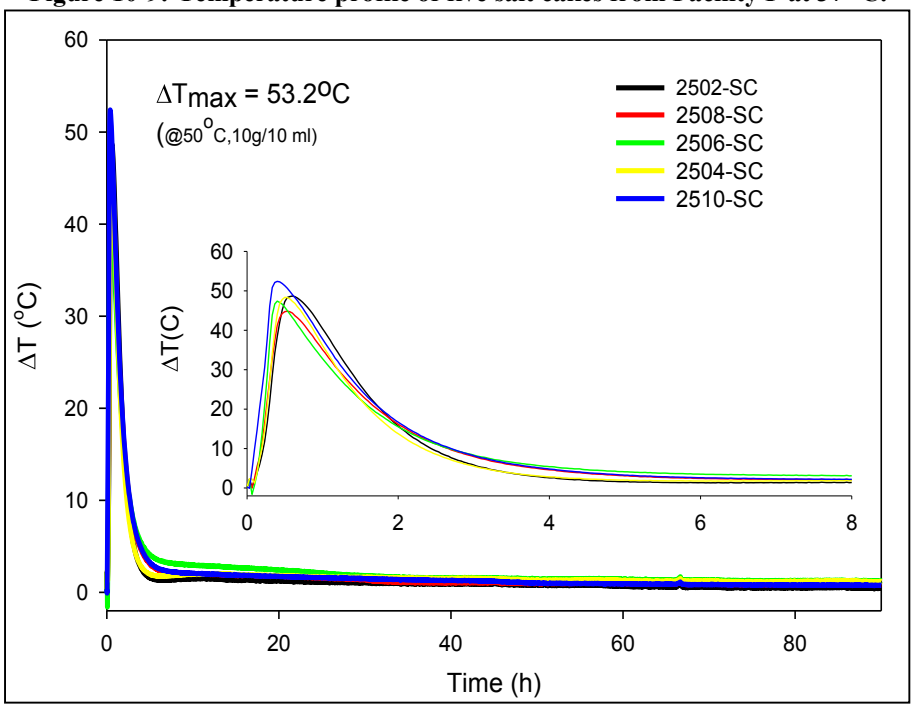
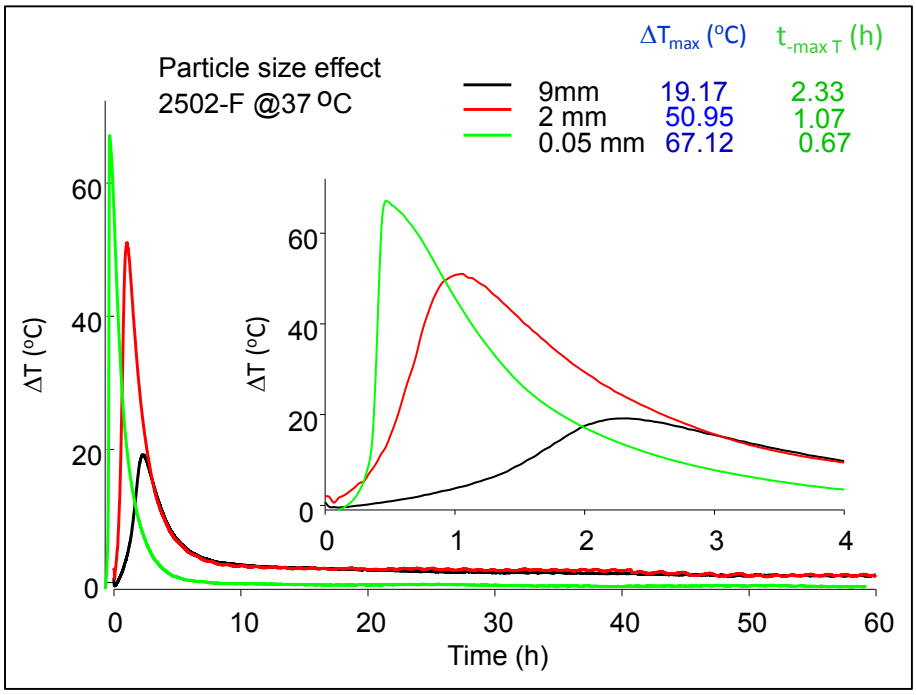



**Figure 10-10: Temperature profile of five salt cakes from Facility F at 50 °C.**

As expected, the particle size significantly influenced the reaction of salt cake with water, both the degree of temperature increases as well as the intensity of reaction (Table 6-2, Figure 5-3, and Figure 5-4).

**Figure 10-11: Effect of particle size of salt cake 2502-F on the temperature**




**profile at 37 °C.**

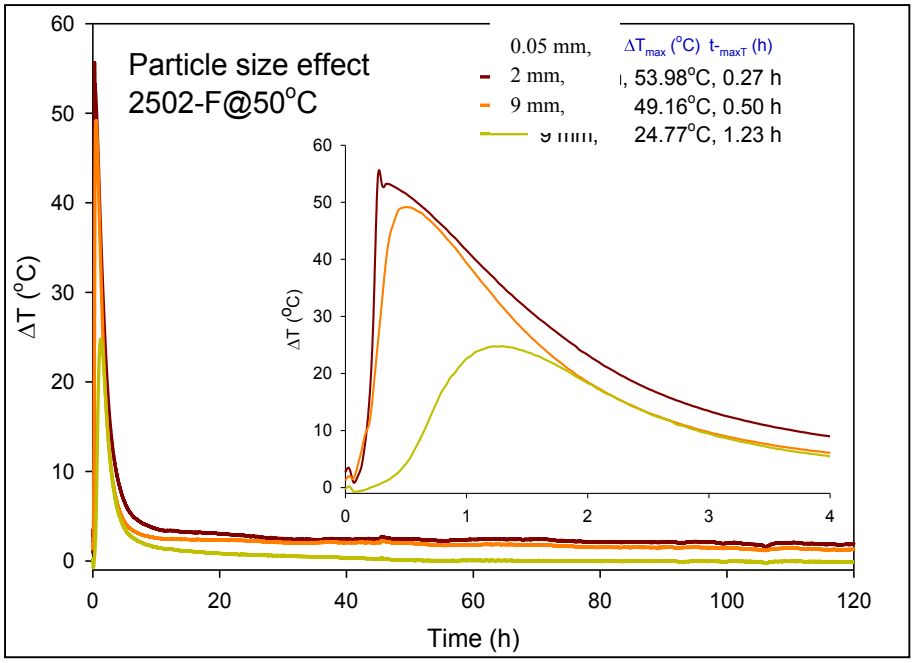



**Figure 10-12: Effect of particle size of salt cake 2502-F on the temperature profile at 50 °C.**

Some trail experiments were conducted to explore the effect of chemical composition of liquids on the temperature changes, including the pH effect (0.1 M HCl and 0.1 M $H_3PO_4$, acidic condition; 0.1 M NaOH, base condition), aluminum ligands effect, including phosphate (0.2 M $NaH_2PO_4$, and 0.2 M $Na_2HPO_4$), and a leachate from landfill. All of these primary results indicated that chemical composition of liquids can significantly change the temperature patterns of salt cake reaction with liquids (Figure 5-5, and Table 6-3).

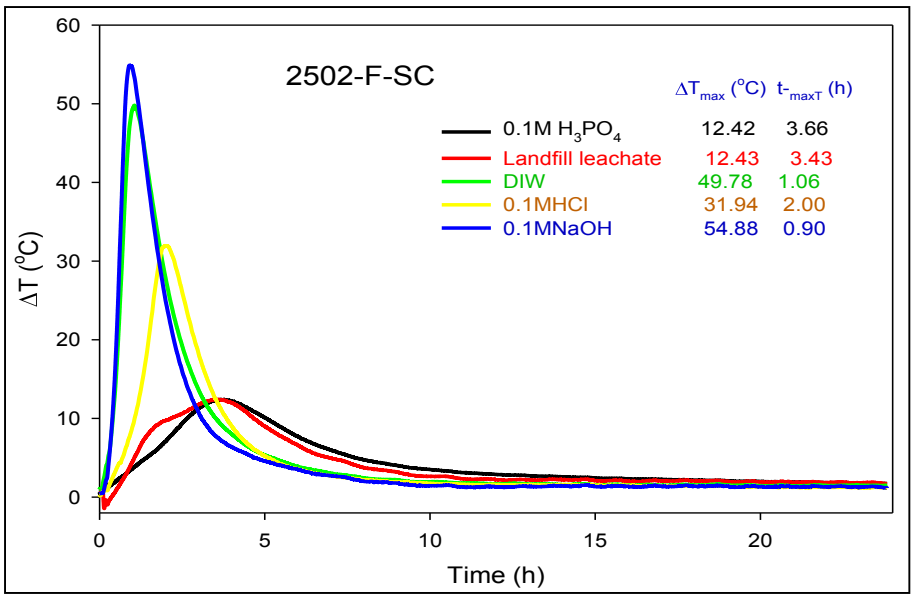



**Figure 10-13: Effect of chemical composition of liquids on the temperature profile of salt cake 2502-F at 37 °C.**

## Facility H

Facility H has five samples, which were collected from November 13, 2009 to March 12, 2010. The strongest temperature response was Sample 2046-H-SC (23.9 °C @ 37 °C; and 55.8 °C @ 50 °C). We also made four replications for sample 2046-H at 50 °C environment.

**Table 10-6: The Temperature Pattern of Facility H**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2046-H-SC | 23.9 | 1.7 | 55.8 | 0.8 |
| 2544-H-SC | 20.3 | 2.2 | 40.0 | 1.2 |
| 2555-H-SC | 16.5 | 2.7 | 33.7 | 1.0 |
| 2559-H-SC | 14.3 | 2.7 | 37.4 | 1.1 |
| 2568-H-SC | 16.7 | 2.9 | 34.8 | 0.9 |
| Maximum | 23.9 | 2.9 | 55.8 | 1.2 |
| Minimum | 14.3 | 1.7 | 33.7 | 0.8 |
| Average | 18.3 | 2.4 | 40.3 | 1.0 |

Similar to the trail experiment on 2502-F, the effect of particle size and chemical composition of liquids to the temperature for sample 2555-H or 2046-H at 37 °C were also conducted (Table 6-2 and 6-3).

**Table 10-7: Effect of Particle Size on the Temperature Change of Salt Cake at 37 °C**

| Sample ID | | $\Delta T_{max}$ (°C) | | | $t_{max-T}$ (h) | | |
|---|---|---|---|---|---|---|---|
| | | **2502-F** | **2512-J** | **2046-H** | **2502-F** | **2512-J** | **2046-H** |
| Particle size | 9 mm | 19.2 | 4.1 | 12.4 | 2.33 | 1.63 | 2.20 |
| | 2 mm | 50.9 | 9.2 | 23.9 | 1.07 | 0.81 | 1.33 |
| | 0.05 mm | 67.1 | 96.2 | 55.6 | 0.67 | 0.23 | 1.56 |

**Table 10-8: Effect of Chemical Composition of liquids on the Temperature Change of Salt Cake at 37 °C**

| Sample ID | | $\Delta T_{max}$ (°C) | | | $t_{max-T}$ (h) | | |
|---|---|---|---|---|---|---|---|
| | | **2502-F** | **2555-H** | **2613-M** | **2502-F** | **2555-H** | **2613-M** |
| Chemical treatment | 0.1 M HCl | 31.9 | 13.9 | 27.6 | 2.00 | 3.57 | 1.50 |
| | 0.1 M NaOH | 55.9 | 20.1 | 53.8 | 0.90 | 2.27 | 0.63 |
| | DIW | 49.8 | 19.1 | 43.0 | 1.06 | 2.73 | 0.97 |
| | 0.1M $H_3PO_4$ | 12.4 | 5.8 | 7.6 | 3.66 | 4.93 | 5.07 |
| | landfill leachate | 12.4 | 9.2 | 12.6 | 3.43 | 3.70 | 2.20 |

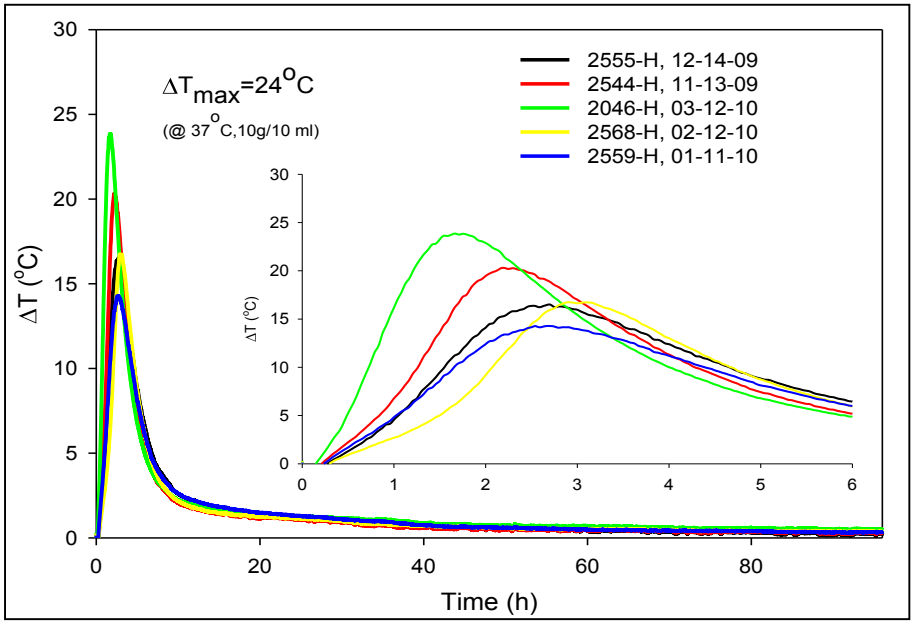



**Figure 10-14: Temperature profile of five salt cakes from Facility H at 37 °C.**

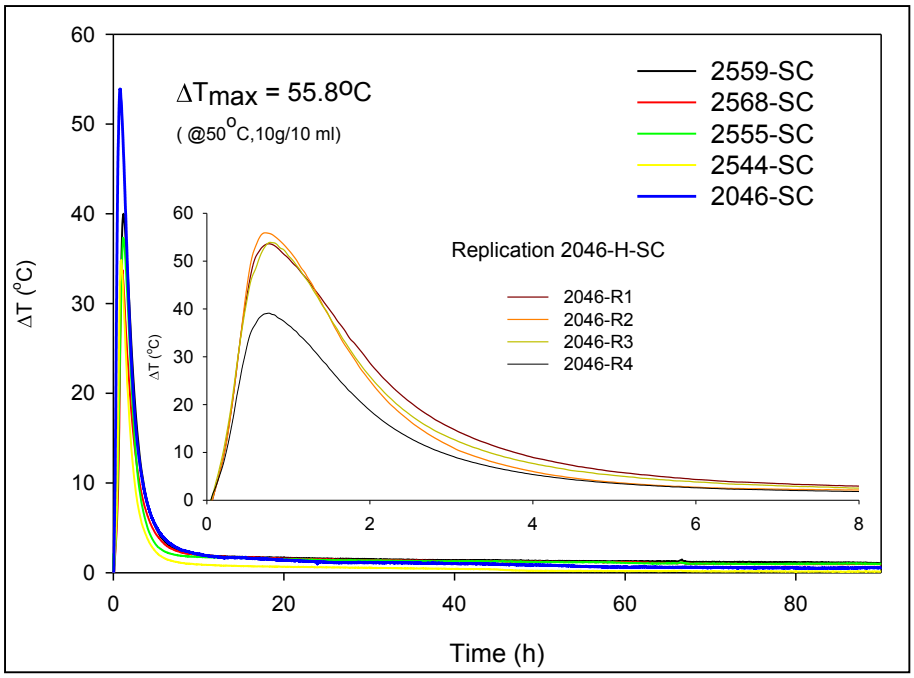



**Figure 10-15: Temperature profile of five salt cakes from Facility H at 50 °C.**

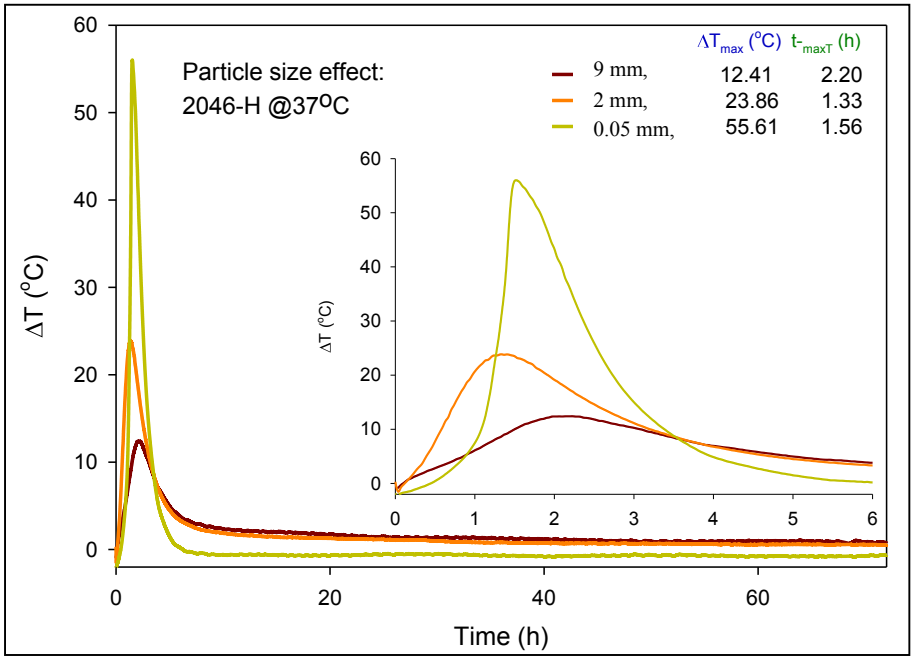



**Figure 10-16: Effect of particle size of salt cake 2046-H on the temperature profile at 37 °C.**

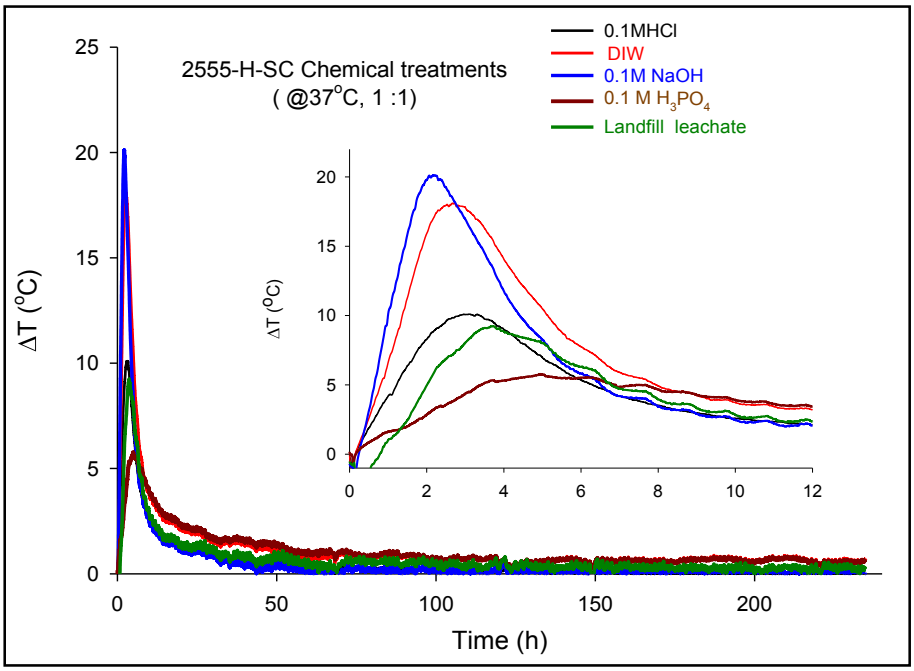



**Figure 10-17: Effect of chemical composition of liquids on the temperature profile of salt cake 2555-H at 37 °C.**

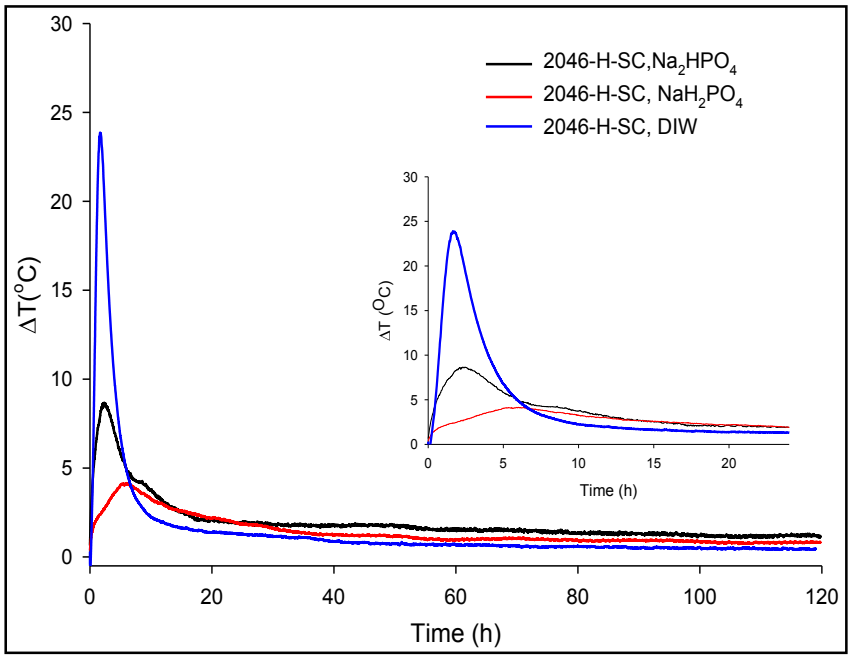



**Figure 10-18: Effect of phosphorus species in liquids on the temperature profile of salt cake 2046-H at 37 °C.**

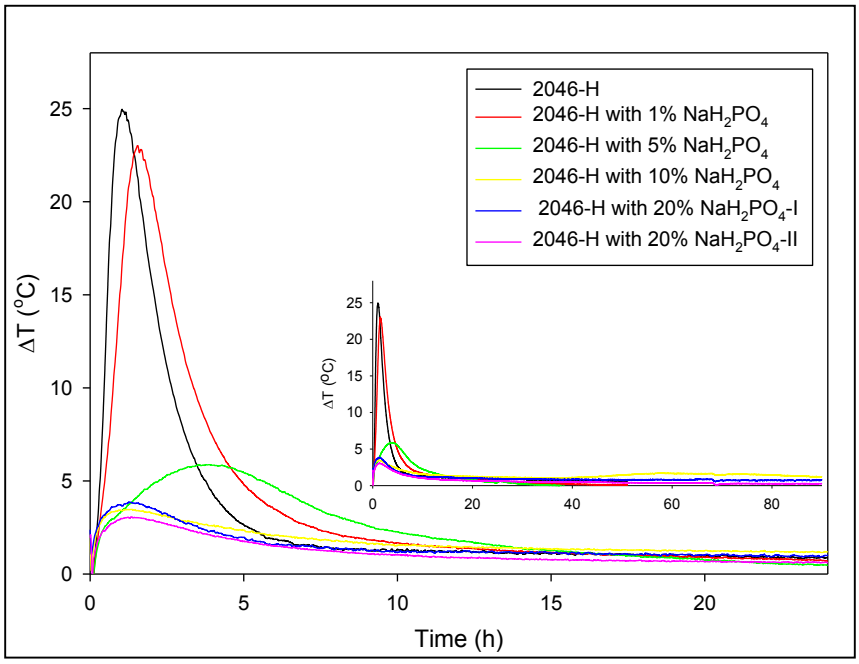



**Figure 10-19: Effect of mix solid phosphorus compounds on the salt cake 2046-H temperature profile at 50 °C.**

The results further confirmed that chemical composition of liquids significantly changed the pattern of temperature change. Phosphate solution or the high amount of DOC leachate (pH6.2, DOC 20,000 mg/L) from the landfill, two different type of ligands for aluminum complex, might have reduced the temperature increase from the reaction of salt cake with water.

## Facility J

Facility J has four samples, which were collected from November 20, 2009 to Feb 5, 2010. Their temperature responses were quite similar at the 37 °C environmental condition, but changed at the higher temperature (e.g. 50 °C) (Figure 7-2). The size effect is also very significant, the response of the small particle is very intensive (Table 6-2, and Figure 7-3). Phosphate in the liquids can also significantly reduce the temperature increase at the short time scale (up to 5 days), but with multiple peaks (Figure 7-4).

**Table 10-9: The Temperature Pattern of Facility J**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2512-J-SC | 9.2 | 0.9 | 22.5 | 1.1 |
| 2515-J-SC | 7.8 | 1.1 | 11.6 | 0.8 |
| 2517-J-SC | 8.4 | 1.5 | 15.6 | 0.9 |
| 2519-J-SC | 6.6 | 1.1 | 9.4 | 0.6 |
| Maximum | 9.2 | 1.5 | 22.5 | 1.1 |
| Minimum | 6.6 | 0.9 | 9.4 | 0.6 |
| Average | 8.0 | 1.1 | 14.8 | 0.8 |

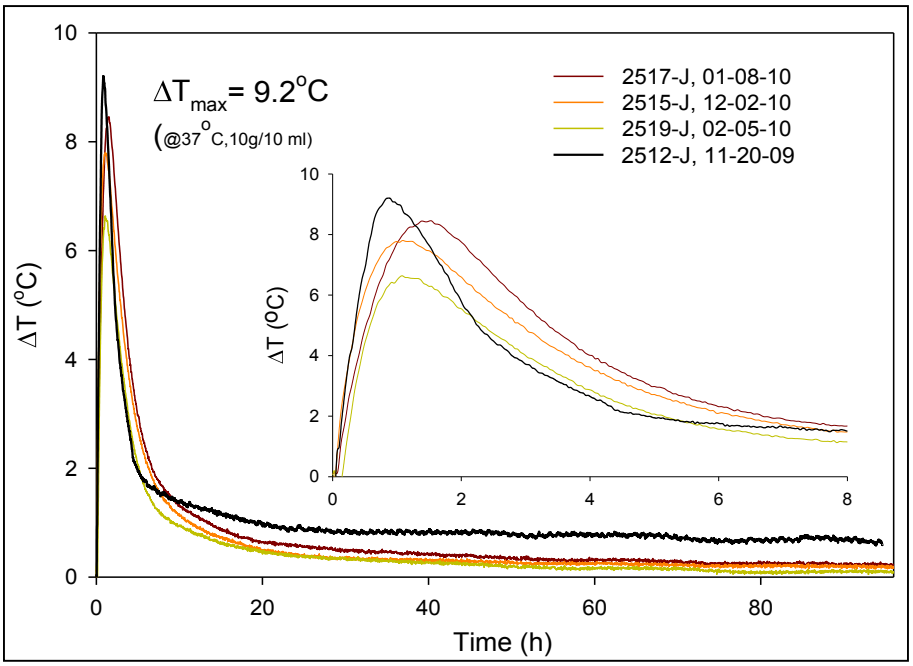



**Figure 10-20: Temperature profile of four salt cakes from Facility J at 37 °C.**

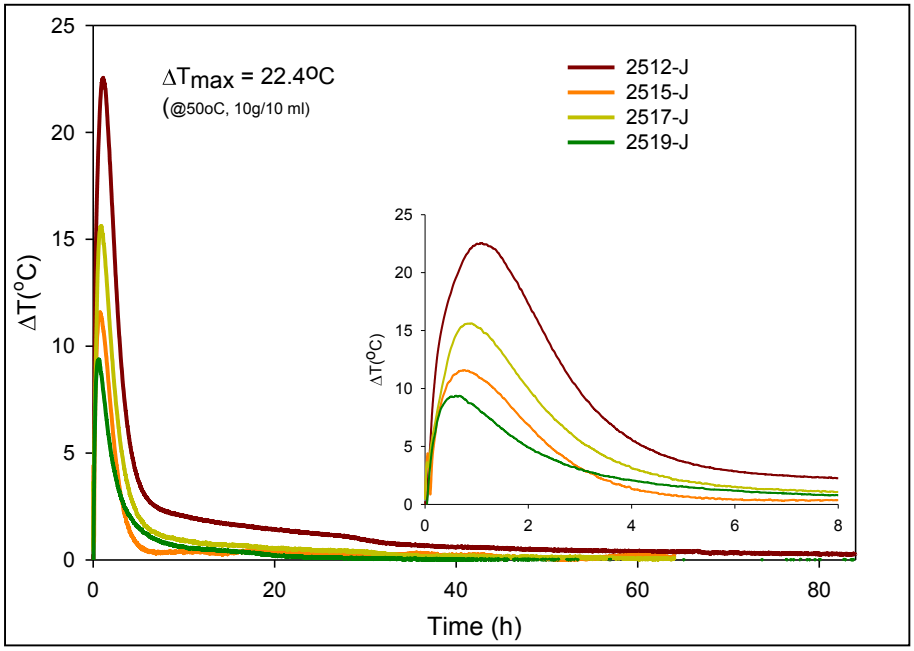



**Figure 10-21: Temperature profile of four salt cakes from Facility J at 50 °C.**

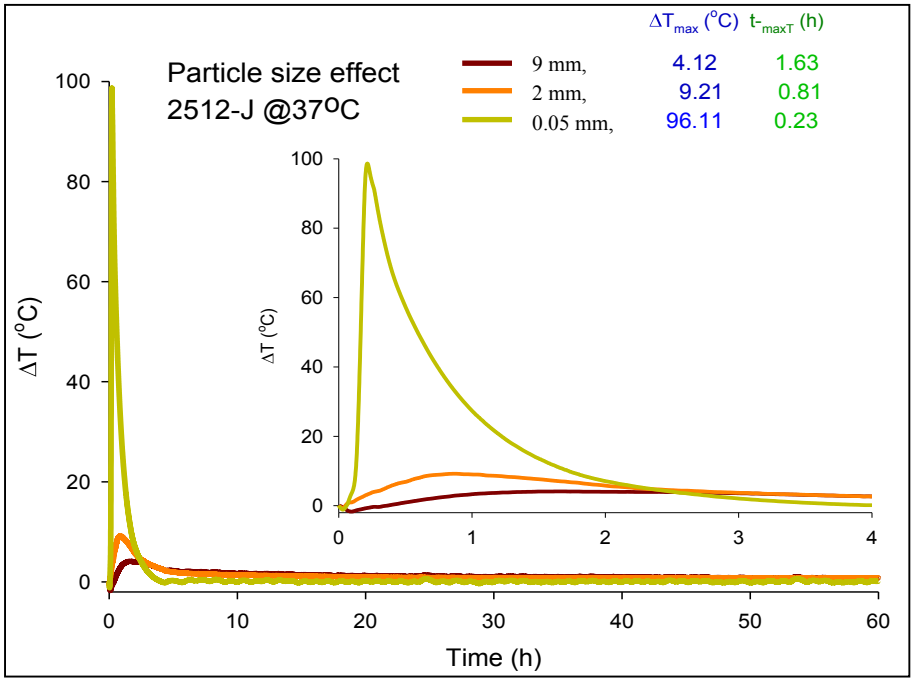



**Figure 10-22: Effect of particle size of salt cake sample 2512-J on the temperature profile at 37 °C.**

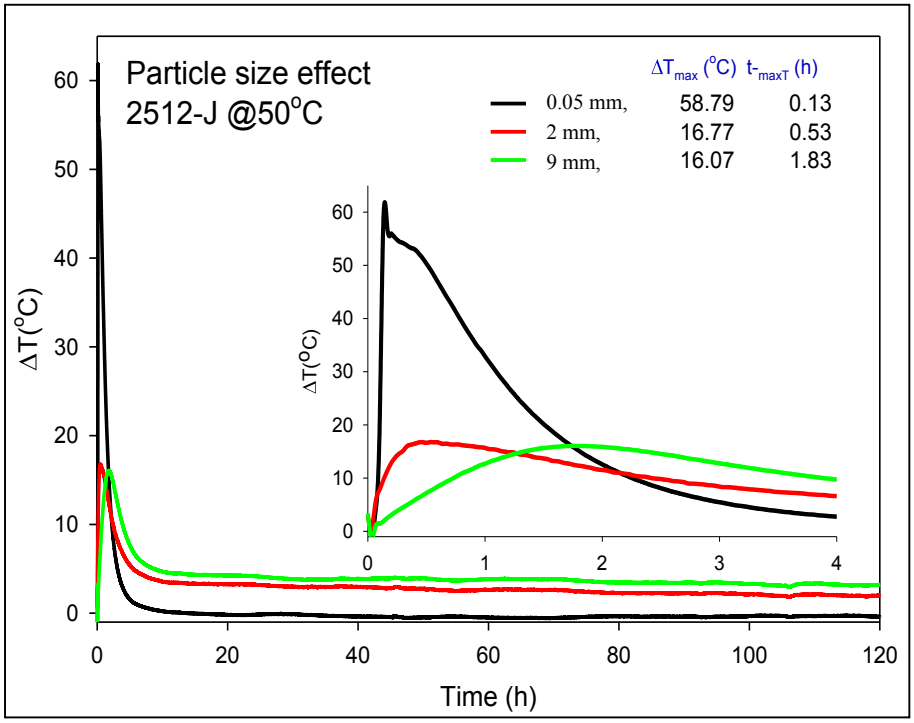



**Figure 10-23: Effect of particle size of salt cake 2512-J on the temperature profile at 50 °C.**

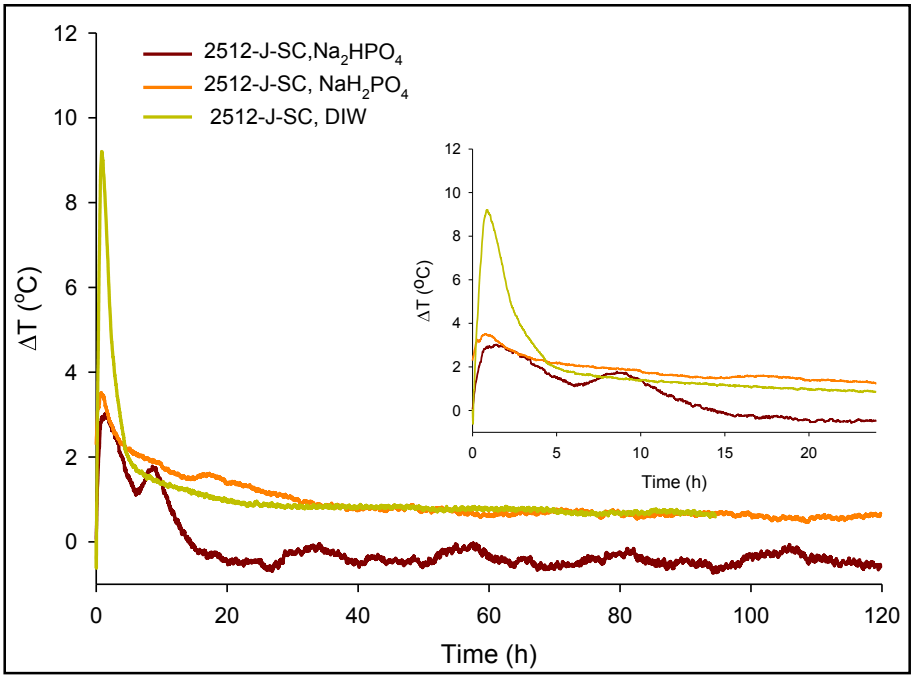



**Figure 10-24: Effect of phosphate solution on the temperature profile of salt cake 2512-J.**

## Facility L

Facility L has five samples, which were collected from December 2, 2009 to April 6, 2010. The highest increased temperature was Sample 2482-L-SC (51.3 °C @ 50 °C, and 44.0 °C @ 37 °C). Note that orthophosphate salt in the solution, a strong inorganic ligands of aluminum, can significantly reduce the absolute temperature increase, but with multiple peaks of temperature

**Table 10-10: The Temperature Pattern of Facility L**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2482-L-SC | 44.0 | 1.0 | 51.3 | 0.4 |
| 2484-L-SC | 36.0 | 1.1 | 40.8 | 0.7 |
| 2486-L-SC | 15.1 | 1.9 | 28.0 | 0.9 |
| 2488-L-SC | 28.7 | 1.9 | 38.1 | 0.7 |
| 2490-L-SC | 22.2 | 1.5 | 28.0 | 0.7 |
| Maximum | 44.0 | 1.9 | 51.3 | 0.9 |
| Minimum | 15.1 | 1.0 | 28.0 | 0.4 |
| Average | 29.2 | 1.5 | 37.2 | 0.7 |

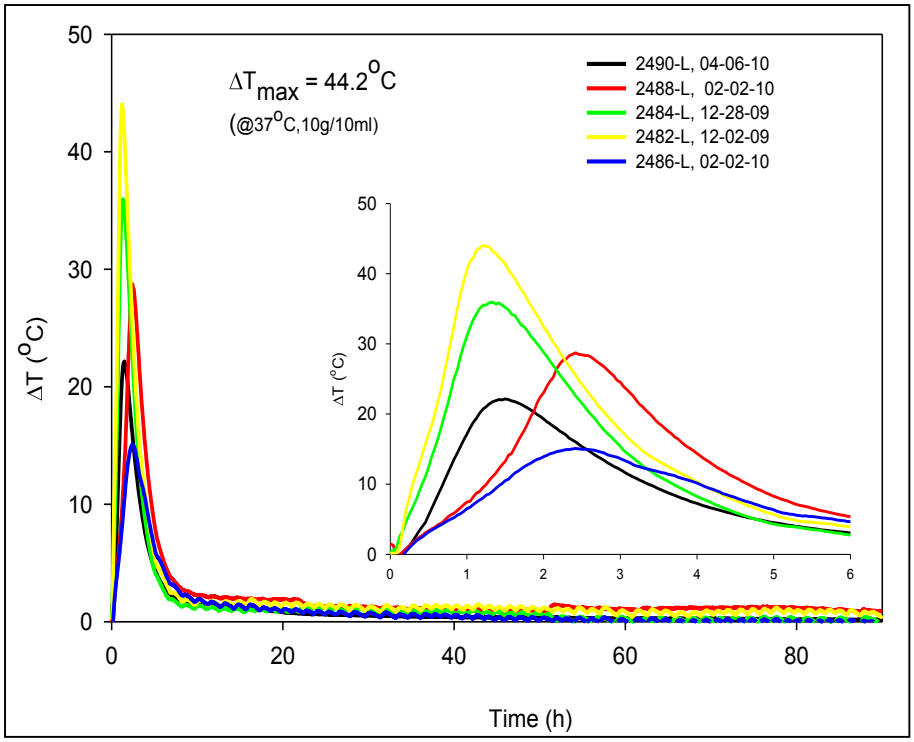



**Figure 10-25: Temperature profile of five salt cakes from Facility L at 37 °C.**

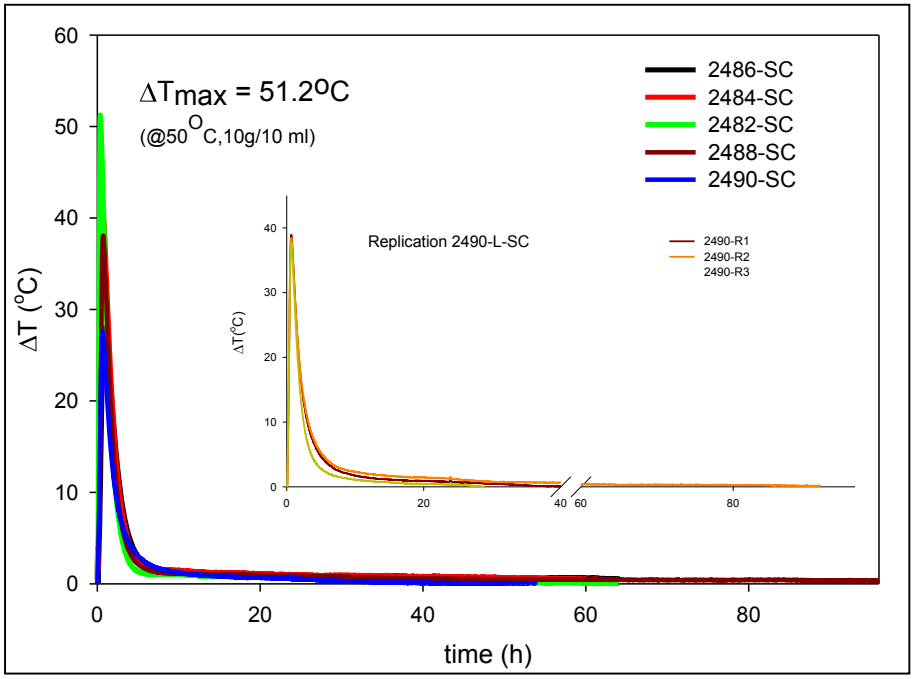



**Figure 10-26: Temperature profile of five salt cakes from Facility L at 50 °C.**

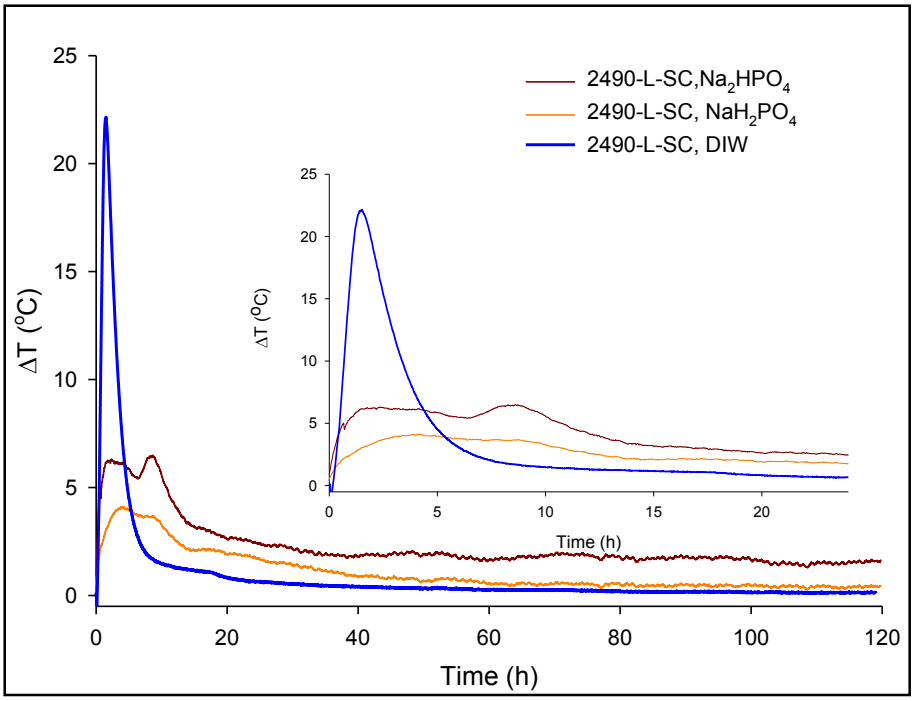



**Figure 10-27: Effect of phosphate solution on the temperature profile of salt cake 2490-L at 37 °C.**

## Facility M

Facility M has four samples, which were collected from February 24 to May 14, 2010. The highest increased temperature was Sample 2601-M (56.3 °C @ 50 °C, and 64.2 °C @ 37 °C). It is confirmed that the chemical composition of liquids significantly change the pattern of temperature, an index of reactivity of salt cake. Inorganic ligands, e.g., phosphate, is an example to reduce the intensity of reaction at the short term (but only short term).

**Table 10-11: The Temperature Pattern of Facility M**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2601-M-SC | 64.1 | 0.5 | 56.3 | 0.3 |
| 2605-M-SC | 19.5 | 1.7 | 22.4 | 0.9 |
| 2609-M-SC | 46.0 | 1.0 | 50.8 | 0.3 |
| 2613-M-SC | 40.0 | 1.0 | 39.9 | 0.5 |
| Maximum | 64.1 | 1.7 | 56.3 | 0.9 |
| Minimum | 19.5 | 0.5 | 22.4 | 0.3 |
| Average | 42.4 | 1.0 | 42.3 | 0.5 |

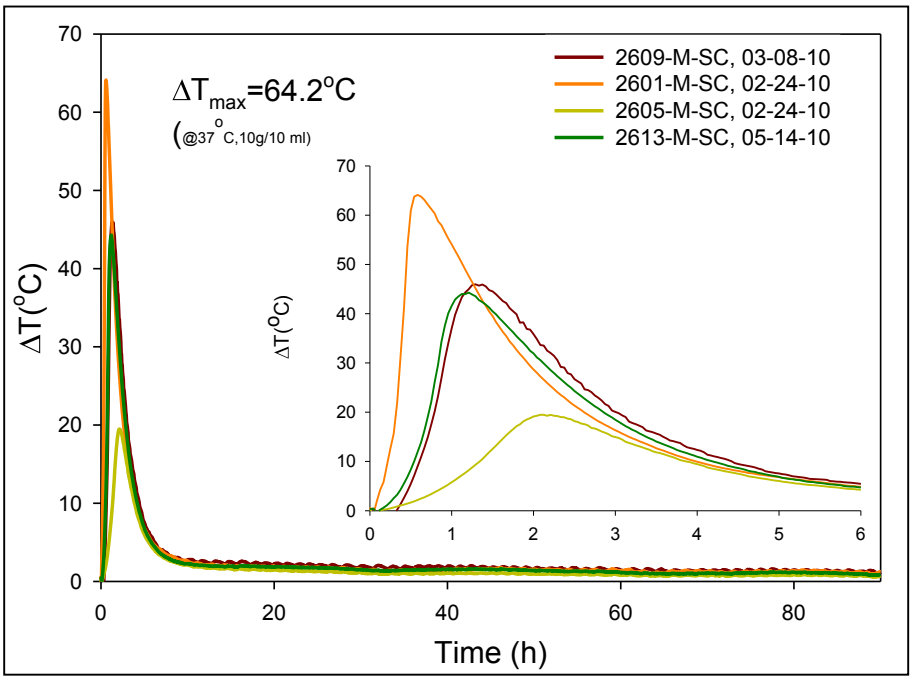



**Figure 10-28: Temperature profile of four salt cakes from Facility M at 37 °C.**

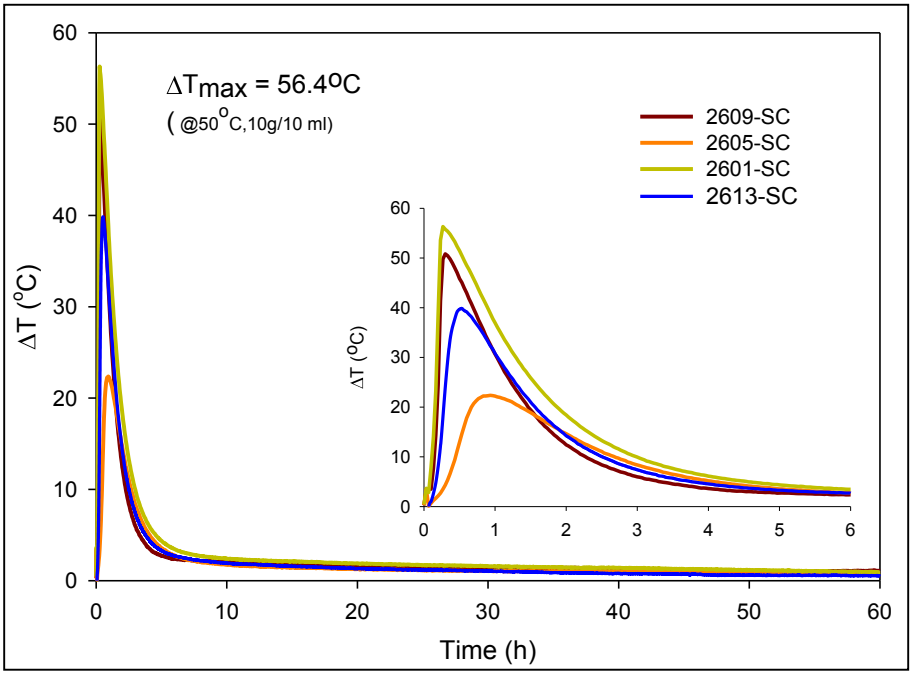



**Figure 10-29: Temperature profile of four salt cakes from Facility M at 50 °C.**

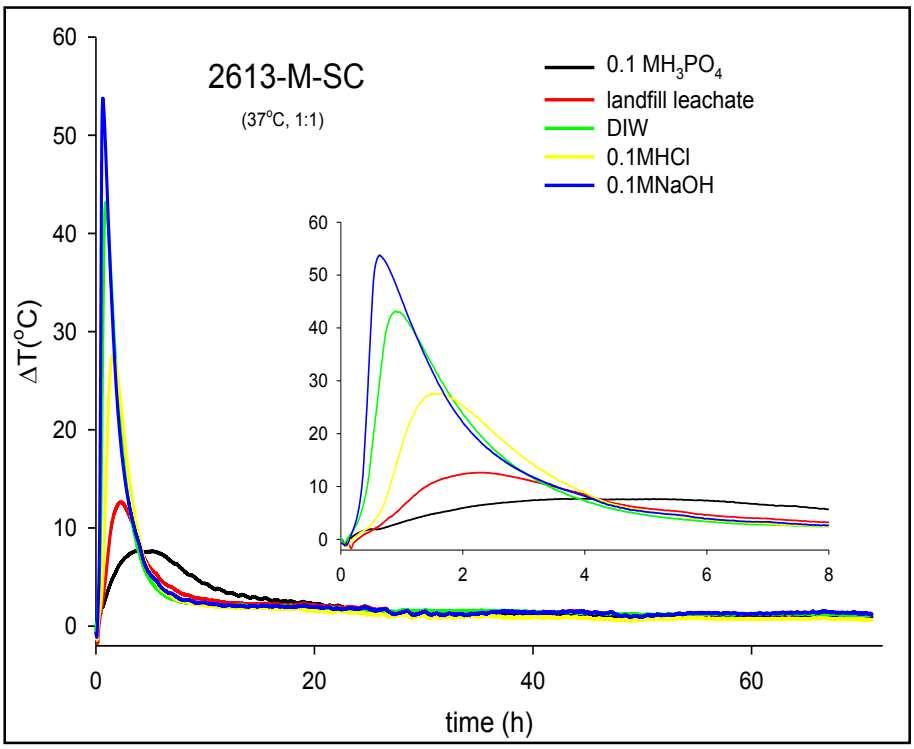



**Figure 10-30: Effect of chemical composition of liquids on the temperature profile of salt cake 2613-M at 37 °C**

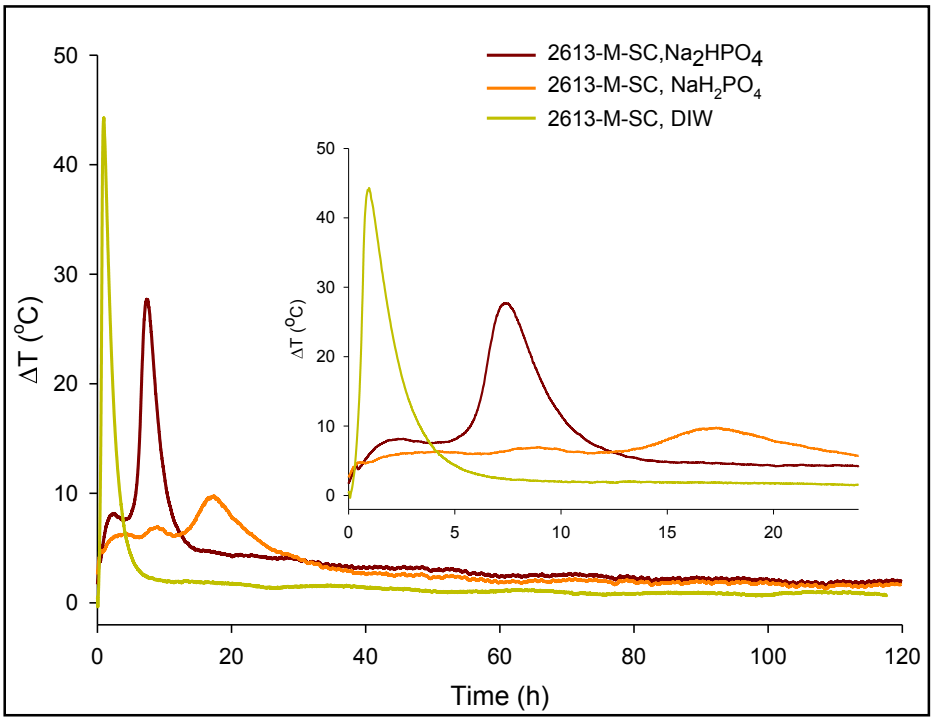



**Figure 10-31: Effect of phosphorus species in liquids on the temperature profile of salt cake 2613-M.**

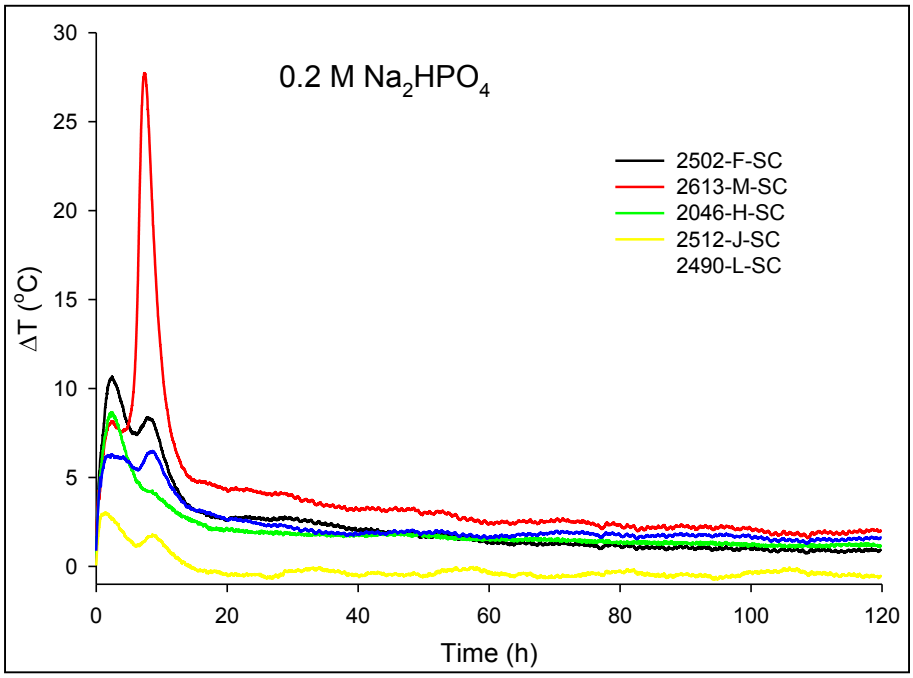



**Figure 10-32: Effect of 0.2 M $Na_2HPO_4$ on the temperature profiles of the selected high reactivity salt cake samples.**

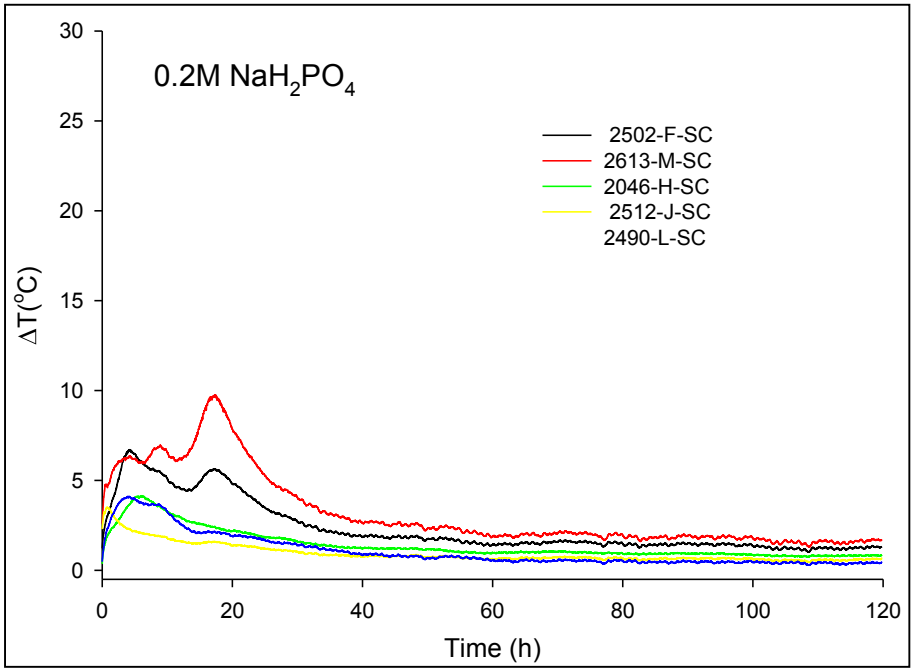



**Figure 10-33: Effect of 0.2 M $NaH_2PO_4$ on the temperature profiles of the selected high reactivity salt cake samples.**

## Facility N

Facility N has four samples, which were collected from December 28, 2009 to April 6, 2010. The highest increased temperature was in Sample 2494-N (12.3 °C @ 37 °C) and 2498-N (18.0 °C @ 50 °C). Note some variations among replications of 2498-N are presented in Figure 10-2, both at 37 °C and 50 °C.

**Table 10-12: The temperature pattern of Facility N**

| Sample ID | 37 °C | | 50 °C | |
|---|---|---|---|---|
| | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) | $\Delta T_{max}$ (°C) | $t_{max-T}$ (h) |
| 2492-N-SC | 7.1 | 1.9 | 7.0 | 1.4 |
| 2494-N-SC | 12.3 | 1.3 | 17.1 | 0.7 |
| 2496-N-SC | 6.7 | 2.9 | 12.1 | 1.2 |
| 2498-N-SC | 7.2 | 2.9 | 18.0 | 1.9 |
| Maximum | 12.3 | 2.9 | 18.0 | 1.9 |
| Minimum | 6.7 | 1.3 | 7.0 | 0.7 |
| Average | 8.4 | 2.3 | 13.6 | 1.3 |

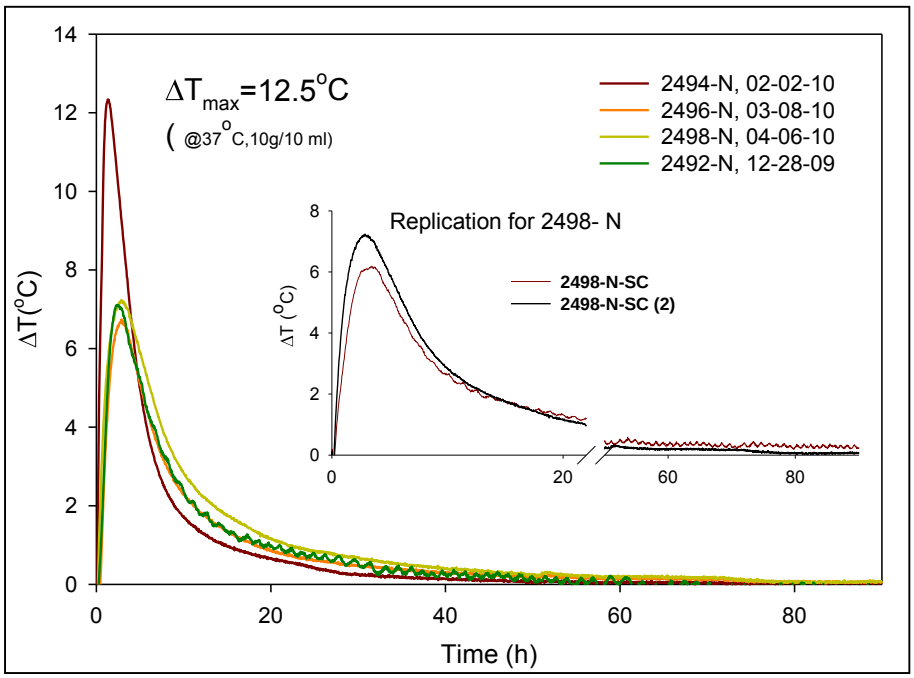



**Figure 10-34: Temperature profile of four salt cakes from Facility N at 37 °C.**

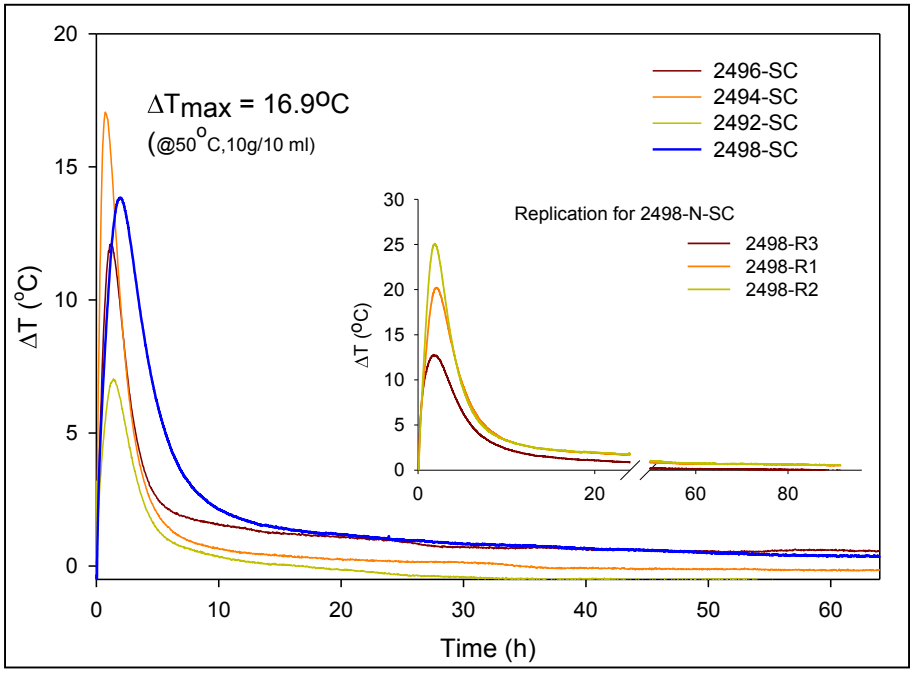



**Figure 10-35: Temperature profile of four salt cakes from Facility N at 50 °C.**

*Secondary Aluminum Processing Waste: Salt Cake Characterization and Reactivity*

# 11. APPENDIX E: Gas Productivity of Salt Cake

**Table E-1 Total generated gas volume and composition from salt cake reactivity***

| Sample ID | Generated Gas Volume (ml $g^{-1}$) | $CH_4$ (%) | $H_2$ (%) | $N_2O$ (ppbv) | $NH_3$-gas (%) | $NH_4^+$-liquid (mg $L^{-1}$) |
|---|---|---|---|---|---|---|
| 2452-B | 12 | 36 | 63 | 3,600 | 0.96 | 1,200 |
| 2454-B | 7.5 | 35 | 63 | 4,900 | 2.2 | 730 |
| 2468-B | 19 | 25 | 75 | 1,100 | 0.66 | 1,000 |
| 2478-B | 30 | 24 | 76 | 400 | 0.83 | 1,100 |
| 2433-C | 31 | 14 | 86 | 230 | 0.30 | 740 |
| 2435-C | 25 | 15 | 83 | 140 | 1.6 | 250 |
| 2532-D | 25 | 17 | 82 | 88 | 0.81 | 30 |
| 2536-D | 1.6 | 90 | 0.0 | 18,000 | 9.9 | 840 |
| 2538-D | 24 | 32 | 67 | 1,600 | 0.21 | 170 |
| 2540-D | 22 | 14 | 84 | 510 | 1.8 | 620 |
| 2437-E | 43 | 14 | 85 | 50 | 0.68 | 210 |
| 2439-E | 28 | 16 | 84 | 78 | 0.42 | 880 |
| 2502-F | 58 | 16 | 83 | 65 | 0.67 | 380 |
| 2504-F | 44 | 8.8 | 91 | 49 | 0.09 | 1,100 |
| 2506-F | 45 | 17 | 83 | 95 | 0.30 | 51 |
| 2508-F | 31 | 12 | 88 | 160 | 0.23 | 730 |
| 2510-F | 60 | 15 | 85 | 520 | 0.08 | 1,100 |
| 2046-H | 15 | 24 | 74 | 210 | 2.6 | 240 |
| 2544-H | 18 | 18 | 81 | 160 | 1.4 | 740 |
| 2555-H | 12 | 22 | 76 | 180 | 1.8 | 540 |
| 2559-H | 22 | 31 | 66 | 150 | 2.2 | 460 |
| 2568-H | 9.1 | 21 | 77 | 2,800 | 2.1 | 1,400 |
| 2512-J | 35 | 7.3 | 92 | 70 | 0.33 | 170 |
| 2515-J | 28 | 16 | 84 | 120 | 0.27 | 110 |
| 2517-J | 23 | 15 | 84 | 130 | 0.64 | 200 |
| 2519-J | 19 | 20 | 79 | 2,700 | 0.39 | 580 |
| 2482-L | 40 | 24 | 75 | 80 | 0.71 | 260 |
| 2484-L | 31 | 22 | 78 | 1,300 | 0.43 | 1,500 |
| 2486-L | 13 | 42 | 57 | 1,800 | 1.4 | 1,500 |
| 2488-L | 1.5 | 49 | 47 | 1,500 | 3.8 | 290 |
| 2490-L | 13 | 12 | 88 | 170 | 0.001 | 95 |
| 2601-M | 78 | 8.7 | 91 | 510 | 0.15 | 660 |
| 2605-M | 47 | 6.8 | 93 | 240 | 0.28 | 450 |
| 2609-M | 210 | 3.1 | 97 | 20 | 0.04 | 350 |
| 2613-M | 170 | 0.5 | 99 | 31 | 0.06 | 500 |
| 2492-N | 12 | 11 | 88 | 180 | 0.50 | 230 |
| 2494-N | 21 | 15 | 83 | 160 | 1.8 | 310 |
| 2496-N | 19 | 14 | 85 | 420 | 0.23 | 490 |
| 2498-N | 19 | 21 | 79 | 190 | 0.07 | 110 |

*L/S =10:1, 10 g SC, < 2 mm, 100 ml DIW, @ 50 ºC, 5 d.

*Secondary Aluminum Processing Waste: Salt Cake Characterization and Reactivity*

## 12. APPENDIX F: Quality Assurance Project Plan (QAPP)

National Risk Management Research Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

**Amendment 2**
**Quality Assurance Project Plan**
**Category IV Measurement Project**

# Management Options for the Safe Deposit of Secondary Aluminum Processing Waste

Original QA ID: L-13928

U.S. Environment Protection Agency Contract No. EP-C-11-006
Work Assignment No. 2-26 (1-26, 0-26)

Prepared for:

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Revision 1
June 6, 2013

**SUMMARY OF WORK ASSIGNMENT 2-26 AMENDMENT 2**
**QUALITY ASSURANCE PROJECT PLAN CHANGES**

The Work Assignment (WA) 1-26 Amendment 2 Quality Assurance Project Plan (QAPP) titled *Management Options for the Safe Deposit of Secondary Aluminum Processing Waste*, dated February 19, 2013, QA ID No. L-13928, is being revised to address the deficiencies identified during the EPA QA Audit that was conducted on April 9, 2013.

The following Sections in this QAPP have been revised:

Quality Assurance Project Plan Approval List
Section 2.1.2 ( page 10, 11)
Section 3.4.1 (page 16)
Section 3.4.3 (page 20, 22)
Section 3.6.4 (page 25)
Section 3.6.5 (page 26)
Section 4.5 (page 27)
Section 6.1 (Table 6.1) ( page 35, 36)

## Quality Assurance Project Plan Approval List

**Pegasus Technical Services, Inc. Concurrences:**

1. Raghu Venkatapathy, PhD, On-Site Technical Manager

   _____ _____
   Signature Date

2. Xiaolan Huang, PhD, WA Leader

   _____ _____
   Signature Date

3. Steven Jones, ASQ CQA/CQE, Contract Quality Assurance Manager

   _____ _____
   Signature Date

**U.S. Environmental Protection Agency Approval for Implementation:**

1. Thabet Tolaymat, PhD, Principal Investigator

   _____ _____
   Signature

2. Robert Ford, PhD, Principal Investigator

   _____ _____
   Signature Date

3. Jim Voit, LRPCD Quality Assurance Manager

   _____ _____
   Signature Date

**Distribution List:**

- Dr. Thabet Tolaymat, Principal Investigator , NRMRL, U.S. EPA.
- Dr. Robert Ford, Co-PI, NRMRL, U.S. EPA.
- Mr. Stephen Wright, Project Officer, NRMRL, U.S. EPA.
- Mr. Jim Voit, QA Manager, LRPCD, U.S. EPA.
- Mr. David Carson, Branch Chief, NRMRL, U.S. EPA*.
- Dr. Raghu Venkatapathy, On-Site Technical Manager, PTSI.
- Mr. Steven Jones, Contract QA Manager, Shaw Environmental & Infrastructure, Inc.
- Dr. Xiaolan Huang, Dr. Mahendranath Arambewela, Ms. Wenwen Yang, Ms. Renata Adkins and Dr. Amro El Badawy, On-Site Contractors, PTSI.
- Mr.Cluxton Phillip, On-Site Contractor
- Mr. Casey Warren, U.S. EPA Student Contractors.

* These individuals are receiving copies of the QAPP and subsequent revisions at their request.

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# SECTION 1.0

# PROJECT DESCRIPTION AND OBJECTIVES

## 1.1 Project Description

Secondary aluminum processing (SAP) wastes result from the smelting of primary aluminum (Al) waste and processing of recycled aluminum. These wastes include 'saltcake,' and air pollution control filter dust (bag house dust) that might contain oxidized aluminum, small particles of aluminum metal, aluminum nitrides, aluminum carbides, and metal oxides. In 1999, it was reported that approximately 2 billion pounds of SAP waste was disposed of in U.S. landfills (U.S. DOE, 1999).

The disposal of vast amounts of SAP wastes containing various Al species can pose a potential threat to the management of landfills. This is because SAP waste can react with water and generate heat as well as flammable gases such as hydrogen and methane. Additionally, when placed in a municipal waste landfill, this reaction may be sufficiently exothermic to initiate the combustion or pyrolysis of other waste materials.

Some municipal waste landfills across the country have experienced significant problems attributed to the SAP wastes in landfills. Some of these events are subsurface heating, excessive subsidence and slope instability, engineering component failure, and dangerous onsite working conditions. Aside from these potential impacts, there may be environmental and community health impacts associated with air emissions, surface water, and groundwater releases.

In light of these problems, the U.S. Environmental Protection Agency (EPA) is initiating a collaborative research effort to examine safe management practices for SAP wastes. The EPA is seeking to better understand SAP waste material, its potential risk, and strategies to prevent, arrest, or exhaust potential peril for safe management of municipal landfills. It is expected that inroads will be made towards the creation of technologies or management practices whereby SAP wastes can be safely deposited in landfills, avoiding future problems.

The EPA has established a coordinating committee to provide technical assistance and project guidance through project advisory meetings, report reviews, and participation in an online research collaboration website.

## 1.2 Project Objectives

The current study aimed at exploring the chemical composition and mineral phase of the waste from SAP industry. Furthermore, temperature change and gas generation after reaction with water or landfill leachate is studying the potential reactivity in laboratory setting. This allows for a better understanding of the behavior of the SAP wastes when disposed in landfills. Additionally, this work provides the methodology to investigate such reactions and thus provides the criteria for determining whether a particular sample of salt cake or baghouse dust can be landfilled.

It is noted that this is a preliminary study. The potential relationship between the chemical composition and reactivity, either gas or heat generation, will be tested and identified in the laboratory setting. Based on the findings, further research will be carried out to investigate and validate the relationships identified in this work.

The specific objectives of the study are:

- To identify and characterize the metal constituents in SAP wastes.
- To identify and semi-quantify the dominant mineral phase present in the SAP wastes using XRD analysis.
- To evaluate temperature change after SAP wastes exposure to water and/or landfill leachate.
- To identify and characterize the generated gas quantity and quality when SAP waste reacts with water and/or landfill leachate.
- To investigate the metal composition of SAP waste leachate after SAP reaction with water and /or landfill leachate.
- To conduct Toxicity Characteristics Leaching Procedure (TCLP) to evaluate the possibility of heavy metal leaching from SAP waste.
- To evaluate heat generation after SAP wastes exposure to water and/or landfill leachate.
- To simulate the thermal changes in Municipal Solid Waste (MSW) landfills as a result of co-disposal of SAP waste with MSW using the TEMP/W model.
- To develop methodology to assess the potential risk of SAP reaction with water and /or landfill leachate.

# SECTION 2.0

# ORGANIZATION AND RESPONSIBILITIES

## 2.1 Project Personnel

### 2.1.1 EPA Staff

The EPA Principal Investigators (PI) for this project are Dr. Thabet Tolaymat and Dr. Robert Ford. Dr. Tolaymat is responsible for overseeing the paperwork associated with the management of the project, and the research procedures associated with total metals analysis. Dr. Ford is responsible for overseeing and conducting research procedures associated with X-ray diffraction, Fourier-transformation infrared spectroscopy, scanning electron microscopy, and thermogravimetric analysis. Dr. Tolaymat and Dr. Ford will also be responsible for coordinating the collection of SAP waste samples through the Aluminum Association and the Environmental Research and Education Foundation. Dr. Tolaymat and Dr. Ford will meet monthly to discuss the progress of the research. Mr. Stephen Wright is the EPA Project Officer for EPA Contract No EP-C-05-056. Mr. Jim Voit is the EPA Land Remediation and Pollution Control Division (LRPCD) Quality Assurance (QA) Manager responsible for approving the Quality Assurance Project Plan (QAPP). Mr. Casey Warren, EPA Student Contractors, and Mr.Cluxton Phillip, EPA On-Site Contractor, are responsible for providing support to the PTSI WA Leader and EPA WA Manager for operation of the experiment, data collection, analysis and reporting.

### 2.1.2 PTSI Staff

- Dr. Karen Koran is the Pegasus Technical Services, Inc. (PTSI) Project Manager. Dr. Raghuraman Venkatapathy, PTSI On-Site Technical Manager, is responsible for supervision of the PTSI staff. Mr. Steven Jones, ASQ CQA/CQE, of Shaw Environmental & Infrastructure, Inc. (Shaw), a subcontractor to Pegasus in this contract, is the PTSI Contract QA Manager and is responsible for oversight PTSI quality program implementation and the review of this QAPP. Dr. Xiaolan Huang ,the Pegasus WA Leader, is responsible for preparation of the QAPP, setup and operation of the experiment, data collection, analysis and reporting. Dr. Mahendranath Arambewela, Ms. Wenwen Yang, Ms. Renata Adkins and Dr. Amro El-Badawy, PTSI On-Site Technical Staff, are responsible for providing support to the PTSI WA Leader for operation of the experiment, data collection, analysis and reporting. Dr. Mahendranath Arambewela is also responsible for ICP and AA analysis, and Ms. Wenwen Yang for IC and GC analysis.

**Table 2.1 _Project Organization and Responsibilities**

| Name | Phone/email | Responsibilities |
|---|---|---|
| Mr. Stephen Wright | (513) 569-7610<br>wright.stephen@epa.gov | EPA Project Officer |
| Dr. Thabet Tolaymat | (513) 487-2860<br>tolaymat.thabet@epa.gov | EPA Principal Investigator |
| Dr. Robert Ford | (513) 569-7501<br>robert. ford@epa.gov | EPA Co-Principal Investigator |
| Mr. Jim Voit | (513) 487-2867<br>voit.jim@epa.gov | EPA LRPCD QA Manager |
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| Mr.Cluxton Phillip | (513)487-2875<br>Cluxton.phillip@epa.gov | On-Site Contractor |
| Mr. Casey Warren | (513)569-7257<br>Warren.Casey@epa.gov | Student Contractor |

## 2.2 Project Schedule

The projected schedule and the main activities are shown in Figure 2.1.

| Activities | Sept.-10 | May-11 | Sept-11 | May-12 | Sept-12 | May-13 | Sept.-13 |
|---|---|---|---|---|---|---|---|
| QAPP/HASP writing | | | | | | | |
| Samples being produced | | | | | | | |
| Sample characterization | | | | | | | |
| Modeling | | | | | | | |
| Report/manuscript writing | | | | | | | |

# SECTION 3.0

# SCIENTIFIC APPROACH

## 3.1 Site Identification and SAP Waste Sampling

Facilities that generate SAP wastes will be identified in collaboration with the Aluminum Association and the Environmental Research and Education Foundation. Each facility will sample both salt cake (SC) and bag house dust (BHD) at different times. The average number of samples to be obtained from each facility is 4. The number of SC samples to be obtained will be at least 35 and the number of BHD samples will be at least 50.

## 3.2 Sample processing

Upon receipt, samples will be logged and then stored in a Hazardous Storage Unit located at the EPA Center Hill Laboratory (CHL) Facility. The storage unit is equipped with a chemical fire suppression system in case any unintended SAP waste reaction occurs. The fire extinguishing system is a chemical system that does not use water since the samples may be water reactive.

Within two weeks of receipt, in order to obtain representative subsamples, the individual samples will be processed as follows:

- The samples will be placed in a stainless steel pan and crushed to pass a 9-mm sieve.
- After thorough mixing, approximately 0.5 kg of the 9-mm sieved materials will be further size reduced down to a size of 2 mm using a grinding machine (Preiser Scientific).
- Approximately 50 gm of the 2 mm size SAP waste will be further reduced to a size of 0.05 mm using a sealed stainless steel jar mill equipped with zirconia grinding beads. The jar mill will be rotated at 20 rpm for 24 hours.
- The 2- mm size will be used for the chemical and physical analysis (pH, conductivity, moisture content and water holding capacity) in the laboratory and the 0.05-mm size will be used for the chemical and XRD analysis.

## 3.3 Sample Characterization

### 3.3.1 Physical Characterization

#### *3.3.1.1 Moisture Content (MC)*

The moisture content of the salt cake samples will be determined. The oven temperature will be set at 105 °C following ASTM Method D-2216 (refer to SOP 4).

#### *3.3.1.2 Water Holding Capacity*

Following the principles of ASTM Method F1815-06 (SOP 5), approximately 10 g of sample will be placed into a cylinder and cold water (Temperature < 12 °C) will be added to make it saturated. The saturated samples will be placed on top of an absorbent membrane until the excess water is drawn away by gravity. Once equilibrium is reached (usually 16 h, overnight), the water holding capacity will be calculated based on the weight of the water held in the sample versus the sample dry weight.

***3.3.1.3 pH and Conductivity (EC)***

The salt cake samples will be reacted with water (1:20, 5 gram/100 ml DIW) and will be incubated for a week at 50 °C. At the end of the incubation period, the samples will be filtered using a 0.45 µm PVDF media and the pH and EC will be determined (SOP 3).

### 3.3.2 Metal Composition

- The SAP waste will be acid digested using EPA SW846 Method 3051A, Microwave assisted acid digestion of sediments, sludges, soils and oils. Following the suggestion of 3051A, the mixed acid (1 HCl and 3 $HNO_3$) will been employed instead of concentrated $HNO_3$ only, a temperature will be set as 185 °C instead of 175 °C, and hold time will be extended from 10 to 30 minutes. The amount of SAP waste to be digested will be 0.1 g instead of 0.5 g because of the high metal content (refer to SOP 8).
- The macro-elements (Al, Ag, Ba, Fe, Mn, Cu, Zn, K, Na, Ca, and S) will be determined by EPA Method 6010B using a Thermo ICP-AES located at the Kirby laboratory at the EPA Center Hill Facility. Trace metals (Pb, Cd, Cr, As and Se) will be analyzed by a Perkin-Elmer graphite furnace AA separately (refer to SOP 9) or Thermo ICP-AES.

### 3.3.3 Mineral Phase

Mineral phase of the 0.05 mm size SAP waste (prepared as described in section 3.2) will be investigated using a Philips X'Pert-MPD system at the EPA CHL Facility (refer to SOP 10). The XRD will be operated under the following conditions:

| | |
|---|---|
| Scan Axis | Gonio |
| Start Position [°2Th.] | 10.0000 |
| End Position [°2Th.] | 106.7800 |
| Step Size [°2Th.] | 0.0100 |
| Scan Step Time [s] | 5.0000 |
| Scan Type | Pre-set time |
| Offset [°2Th.] | 0.0000 |
| Irradiated Length [mm] | 10.00 |
| Specimen Length [mm] | 10.00 |
| Receiving Slit Size [mm] | 0.1000 |
| Anode Material | Cu |
| K-Alpha1 [Å] | 1.54060 |
| K-Alpha2 [Å] | 1.54443 |
| K-Beta [Å] | 1.39225 |
| K-A2 / K-A1 Ratio | 0.50000 |
| Generator Settings | 40 mA, 45 kV |
| Diffractometer Type | XPert MPD |
| Goniometer Radius [mm] | 200.00 |
| Dist. Focus-Diverg. Slit [mm] | 91.00 |
| Incident Beam Monochromator | No |
| Spinning | Yes |

The powder diffraction file (PDF) patterns database from the International Centre for Diffraction Data (ICDD) will be employed for the search, match, and identification. A subset of reference patterns will built for all studied salt cake samples. The semi-quantitative phase analysis will used by the X'Pert HighScore Plus software, based on the CHUNG Normalized RIR Method [66]. The relative intensity of each phase is given by the scale factor, which is determined by least squares fit through all matching reference pattern lines in X'Pert HighScore. The concentration X of phase α is calculated using:

$$\mathbf{X}_{\propto} = \frac{\mathbf{I}_{(\mathbf{hkl})_{\propto}}}{\mathbf{RIR}_{\propto}\mathbf{I}^{\mathbf{rel}}_{(\mathbf{hkl})_{\propto}}}\left[\frac{\mathbf{1}}{\sum_{\mathbf{j=1}}^{\mathbf{n}}\left(\mathbf{I}_{(\mathbf{hkl})_{\mathbf{j}}}\mathbf{RIR}_{\mathbf{j}}\mathbf{I}^{\mathbf{rel}}_{(\mathbf{hkl})_{\mathbf{j}}}\right)}\right] \qquad (2\text{-}1)$$

where $RIR_{\propto}$ is Reference Intensity Ratio, based on the relative net peak height ratio of the strongest line ($I^{rel}$ = 100%) of the phase and of the strongest line of corundum, measured with copper Kα radiation in a mixture of equal weight percentages and $I_{(hkl)_{\propto}}$ is Intensity of reflection of hkl in phase α. hkl is reflection indices.

It is important to note that the normalization used in this method assumes that the sum of all identified phases is 100%. This means that there are no unidentified crystalline phases or an amorphous phase present in the sample. Only under these conditions can meaningful, semi-quantitative results be obtained.

## 3.4 SAP Waste Reactivity Testing

The reactivity of SAP waste with liquids will be evaluated using two approaches. The first one is to quantify the temperature change and the second one is to measure the chemical changes including gas and leachate quality. Different effects of key variables on the temperature change of selected samples will be tested. The samples will be selected based on the mineral phase types (i.e., high metallic aluminum, high aluminum nitride, high aluminum oxide and a combination of the 3 mineral phases) obtained from the XRD analysis. These variables include the solid to liquid ratio, particle size of salt cake, the chemical composition of the solution, and the environmental temperature. Based on these preliminary investigations, a protocol of the temperature profile study for all samples will be prepared. More details in regards to the evaluation of the impact of the previously mentioned key variables on the temperature change upon liquid addition to the SAP waste are described below:

### *A. Effect of Solid to Liquid Ratio and Mass of Aluminum Dross*

The change in temperature will be influenced by the amount of liquid and the mass of SAP waste. Four samples of SAP waste will be selected to investigate the effect of solid to liquid ratio on the temperature change upon reaction with water. Three sets of experiments will be conducted at 50 °C. The samples will be prepared as outlined in section 3.4.1 with the following changes as follows:

- Experiment 1: the solid mass is fixed (10 gm) and the liquid volume will be 3, 5, 10 and 15 ml of preheated DI water.

- Experiment 2: the liquid volume is fixed (3 ml) and the solid weight will be 5, 10 and 15 gm.
- Experiment 3: The ratio of solid to liquid is fixed at 10:3, 10:5 and 1:1. For example in the case of 10:3 ratio the investigated amounts will be 5g:1.5 ml, 10g:3ml, 15g:4.5 ml and 20g:6ml of liquid.

***B. Effect of Particle Size of Aluminum Dross***

Particle size is another major factor that may have an impact on the temperature change upon the reaction of SAP waste with liquids. The smaller the particle size the higher the surface area to volume ratio and consequently the reactivity is expected to be higher. Four samples will be tested as follows:

- Three different particle sizes will be tested (0.05 mm, 2 mm, and 9 mm) for salt cake.
- The environmental temperature will be maintained at 37$^{o}$C and 50$^{o}$C.
- The solid to liquid ratio will be 1 to 1 (10g salt cake with 10 ml DI water).

***C. Effect of Liquid Chemical Composition***

Once SAP waste is disposed in landfills, the chemical composition of the leachate (such as, volatile fatty acids, organic matter, pH) will influence the reactivity of this waste. Therefore, various chemical composition of the liquid will be investigated as follows:

- DI water (pH 6.5), 0.1 M HCl (pH 1.0), 0.1 M NaOH (pH 13), 0.1M $H_3PO_4$ (pH1.65), 0.1M $NaH_2PO_4$ (pH 5.6), 0.1M $Na_2HPO_4$ (pH 8.2), and landfill leachate (pH 6.2, DOC-20,000mg/L).

The ratio of solid to liquid will be fixed at 1 to 1 (10 g salt cake with 10 ml liquid).

***D. Effect of Environmental Temperature***

High temperature may trigger the reaction of metallic aluminum with liquid . Additionally, some components of the SAP waste such as aluminum nitride and aluminum carbide can react at low temperature. Thus, the impact of environmental temperature on the reactivity of SAP waste will be evaluated as follows:

- The environmental temperatures to be investigated will be 20, 37 and 50$^{o}$C.
- The solid to liquid ratio will be fixed at 1 to 1 (10 g aluminum dross with 10 ml liquid). DI water will be used as the liquid.

### 3.4.1. Temperature Generation Potential

Temperature increase is one of the key parameters indicating a reaction between salt cake and, usually, water. The increase in temperature caused by the reaction of salt cake and baghouse dust with liquid will be evaluated as follows:

- A sample of 10 gram of the size reduced salt cake (<2 mm) or baghouse dust will be placed in a 60 ml VOA vial.
- The vial will be sealed and purged with argon for 10 minutes.

- The vial will be then connected to a 1-L Tedlar bag (for pressure relief).
- The VOA vial with the sample will be pre-heated to a certain temperature (37 and 50°C) for 6 hours in order to achieve the required temperature.
- The vial temperature will be monitored and logged using a thermocouple wire placed in the sample.
- A 10 ml of pre-heated DI water (same sample temperature) will be then added to the vial. The temperature change will be recorded over time for at least 72 hours till no temperature change due to the reaction.
- After the reaction completion, the gas in the Tedlar Bag will be released in the hood.

Usually, 6 to 12 vials will be grouped as one set in an insulated box placed in a temperature controlled system. For quality assurance, at least one control sample will be included with each sample group. The control sample is composed of pure quartz sand having the same sample weight and will be processed as the SAP waste samples. The temperature of the control should be within 2 degrees of the set temperature. It was assumed that the average temperature difference between the control and sample was 0 in the last 10 hours of measurement. To isolate the heat transfer among the vials insulation materials will be placed between the vials in the box. Figure 3.1 presents a schematic as well as a photo of the experimental setup. Moreover, reference materials (sodium chloride, potassium chloride, aluminum nitride, aluminum carbide, aluminum oxide and powder metallic aluminum) will be tested to evaluate the interactions of pure aluminum compounds upon liquid addition under the same experimental conditions. The evaluation will follow the same steps outlined earlier for SAP waste samples.

### 3.4.2. Gas Generation and Leachate Chemistry

Gas production potential and leachate chemistry of salt cake as a result of liquid addition will be evaluated under anaerobic conditions in order to mimic most MSW landfills conditions. The experimental procedure for measuring the gas generation and leachate quality is summarized below and schematic of the experimental setup is presented in Figure 3.2:

- A 5 g of size reduced SAP Waste Sample (salt cake sample (<2 mm) or baghouse dust) will be placed into a 500 ml lined media bottle. Reference materials (sodium chloride, potassium chloride, aluminum nitride, aluminum carbide, aluminum oxide and powder metallic aluminum) will also be tested.
- The bottle will be then capped using a lypholization rubber stopper.
- The sealed bottle will be purged with argon for 10 minutes.
- A 100 ml of oxygen-free pre-heated DI water (50°C ) will be added to the bottle.

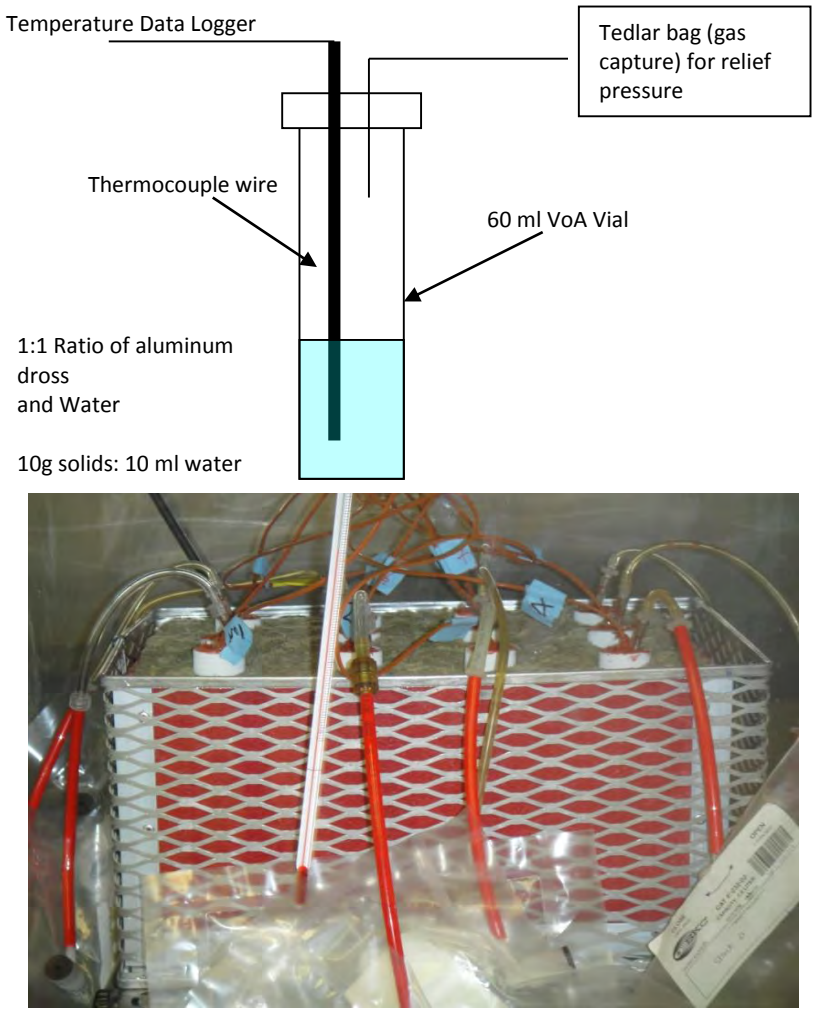


**Figure 3.1: Temperature generation potential apparatus.**

- A 1-L Tedlar bag will be connected to the bottle for the collection of the generated gas.
- The bottle will then be incubated for 7 days at 50°C.
- The Tedlar bag will be disconnected and equilibrated to room temperature and then the gas quantity and quality will be measured. The gas quantity will be measured using a gas syringe and the quality will be measured using Agilent Gas Chromatograph (SOP 1 and 2).
- A 20 ml liquid sample will be taken (using a syringe) from the sealed bottle for liquid ammonia analysis (SOP 7).
- The rest of the liquid will be filtered using a 0.45 µm PVDF filter media and the pH and conductivity of leachate will be measured (SOP 3).
- After measuring the pH and conductivity, the liquid samples will be used for anions and metals analysis. The solution for metal analysis will be preserved by acidification using trace metal grade nitric acid to a pH < 2.0 (SOP 9); the solution for anions analysis will be stored at 4°C (SOP 10).

The experimental setup will be checked for leaks using a gas leak detection fluid ( e.g. LEAK-SEEK®, or soap) before and after the experiment. If any leak is found, the experiment will repeated.

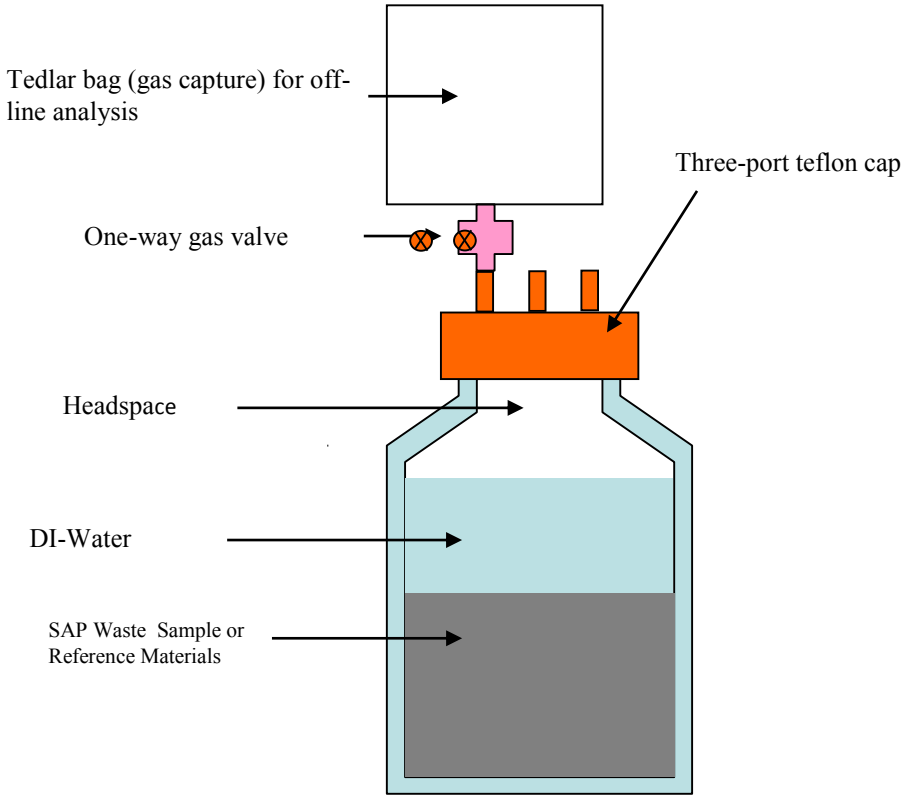


**Figure 3.2: Reactivity test apparatus for gas production.**

### *3.4.2.1 Gas Volume and Composition*

The Gas quantity will be measured using a 60 ml air tight gas syringe per EPA OPPTS method 835.3400. The accuracy of measuring gas volume in a Tedlar bag using a syringe was confirmed in our lab by performing the following test (the experimental setup mimics the test condition and the schematic is presented in Figure 3.3):

- A 5 g of sand sample was placed into a 500 ml lined media bottle and 100 mL of DI water was added to the sand.
- The bottle was capped using a lypholization rubber stopper.
- The sealed bottle was connected to a 1 L Tedlar bag.
- The bottle was then purged with argon at a known flow rate for a certain time (Table 3.1). A flow meter (Cole Parmer) calibrated to argon gas flow was used to measure the actual flow rate from an argon gas cylinder.
- After purging, the gas volume in the Tedlar bag was measured using a syringe and the results were compared to theoretical volume of argon purged.
- The results show that the difference between the actual volume of gas purged and the collected volume using a syringe was < 5% (Table3.1).

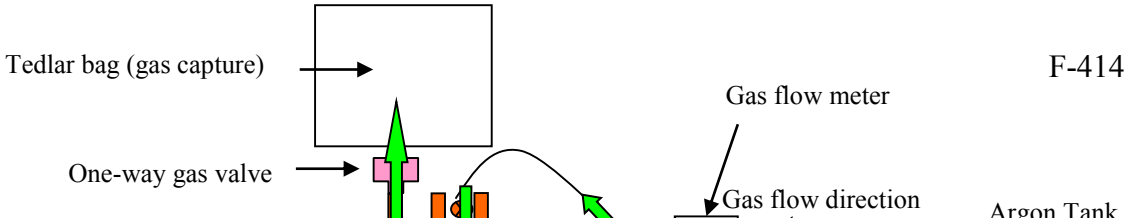

**Figure 3.3: Gas volume verification test apparatus. Green arrows show the gas flow direction**

**Table 3.1 Test results of the gas volume measurement using syringe**

| Gas flow rate (ml per min) | Purging time (min) | Actual gas volume purged (mL) | Gas measured using syringe (mL) | % Difference |
|---|---|---|---|---|
| 120 | 4 | 480 | 495 | 3.0 |
| 120 | 4 | 480 | 505 | 5.0 |
| 117 | 4 | 468 | 480 | 2.5 |
| 117 | 4 | 468 | 465 | 0.6 |
| 115 | 2 | 225 | 230 | 2.2 |
| 115 | 3 | 345 | 345 | 0.0 |

The gas composition will be analyzed for $CH_4$, $CO_2$, $N_2$, $H_2$, $O_2$ and $N_2O$ and $H_2S$. The $CH_4$, $CO_2$, $N_2$, $H_2$ and $O_2$ concentration will be measured using a GC TCD/FID, the $N_2O$ concentration will be measured using GC/ECD (SOP 2) and the $H_2S$ will be measured using a Jerome meter (instrument manual). Additionally, the ammonia concentration in the gas will be measured using an ammonia selective electrode.

#### *3.4.2.2 Leachate Quality Analysis*

The leachate will be analyzed for pH, conductivity (SOP 3), ammonia (SOP 7) and metals (SOP 9). The pH and conductivity will be measured using pH and conductivity probes. The dissolved ammonia in the leachate will be analyzed using Ammonia ion selective electrode. The metal analysis will be performed using a Thermo ICP-AES or Graphite Furnace AA based on the metal concentration. The target metals include Al, As, Cr, Cd, Pb, Se, Fe, Cu, Zn, K, Na, Ca, and Mn.

### 3.4.3. Heat Generation

Heat generation is one of the key parameters to access the safety of aluminum dross disposal in landfills. A custom reaction calorimeter with double vessels has be designed and fabricated to determine the heat generation rate from the aluminum dross reaction (Figure 3.4). In order to obtain the isoperibol condition (constant temperature in the thermal jacket surrounding the reaction vessel), the whole device will be set up in a temperature controlled environment (e.g. temperature room 37.5$^{o}$C or incubation oven, 50$^{o}$C). Ethylene Glycol will be used as a thermal buffer since it has a low specific heat capacity. The experimental procedure for measuring the heat generation is summarized as follows:

- A sample of 5 gram of the size reduced salt cake (<0.05 mm) or baghouse dust or sand (as control) will be placed in a 40 ml inner-vessel (e.g. 40 mL VOA vial with screw cap).
- The inner-vessel with sample will be capped and then put into an outer-vessel (custom made 250 ml vial with a three-port Teflon cap, see Figure 3.4), which contains pre-equilibrium 90 ml Ethylene Glycol. Three thermocouple wires will be placed in the Ethylene Glycol solution. The Ethylene Glycol solution temperature will be allowed to equilibrate overnight (16 h).
- The inner-vessel will then be connected to a 1-L Tedlar bag (for pressure relief).
- A 10 ml of pre-heated liquid (e.g., DI water) (same sample temperature) will be then injected to the inner-vessel by syringe into sample or sand (control). The Ethylene Glycol solution temperature will be recorded over time for at least 72 hours till no temperature change is observed from the reaction.
- .The Ethylene Glycol solution temperature will be monitored every two minutes and logged using date logger.
- The average temperature readings from the three thermocouple wires in the outer vessel from the test sample will be used to calculate the difference with the control (Sand) ($\Delta T$). The sum of temperature change with control ( $\Sigma\Delta T$ ) will be used to access the released heat from sample.
- After the reaction completion, the gas in the Tedlar Bag will be released in the laboratory hood.

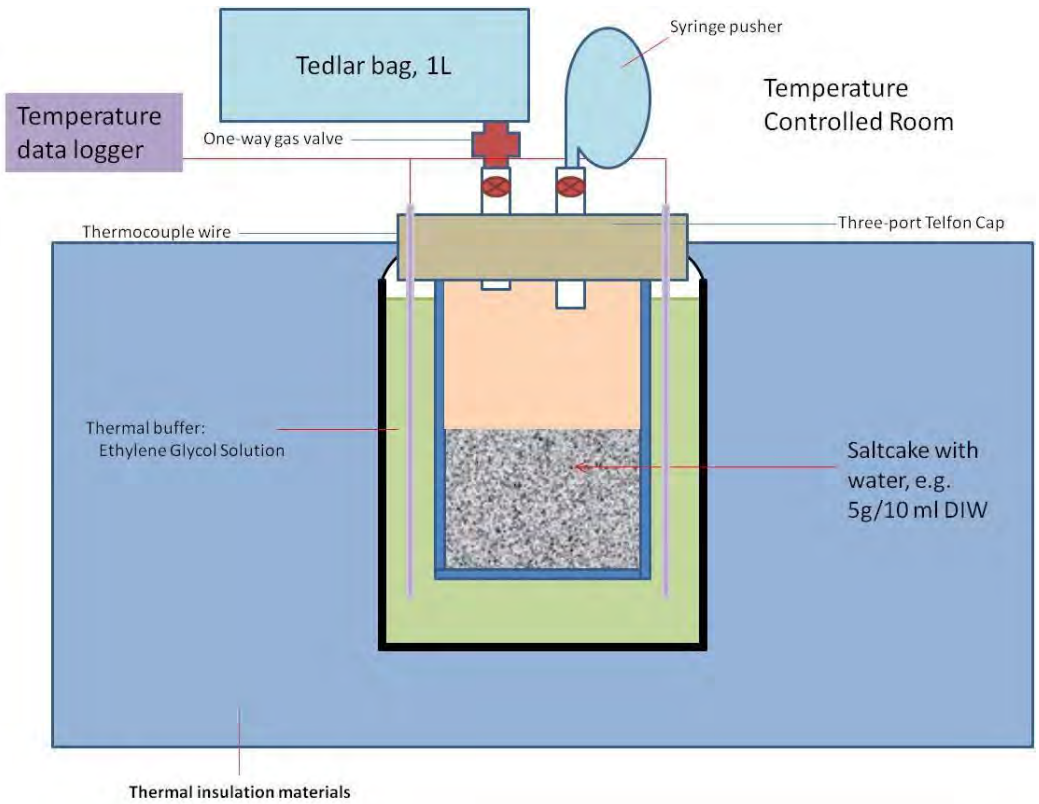


**Figure 3.4: Reactivity Test Apparatus for Measuring Heat Change**

Table 3.2 (below) will be used for calibration as the standard heat generated in the experiments. The heat from the resistance or from the reaction of aluminum dross will be monitored for temperature change. The standard value of heat is based on resistance with different currents and time. Figure 3.5 (below) is the standard curve from the sum of temperature change to the released heat, which is based on the resistance (from Table 3.2). With the Table 3.2 and Figure 3.5, the heat from the reaction of aluminum dross can be estimated.

- A calibration between the above mentioned sum of temperature change ($\Sigma\Delta T$) and the input heat, generated by a fixed resistance (R) with the adjustable electric current (I) with time. The power (P) and voltage (V) was calculated as follows:

$$P = I \times V$$
$$V = I \times R$$

- The generated heat (Q) from resistance is the function of power with time ($Q = P \times t$). The following Table 3.2 is the example of the input energy (released heat) with the adjusted current and time, used for calibration.
- Relationship between the generated heat (Q) and the sum of temperature change ($\Sigma\Delta T$) has been demonstrated as linear, and it was also confirmed that there were no difference of $\Sigma\Delta T$ among the heat time with the same inputted heat (Q). The calibration curve in the current laboratory setting at temperature room and input energy (generated heat Q, kJ) is as following:

$$Q = 0.006\Sigma\Delta T$$

**Table 3.2 The Relation of Heat Generation with Resistance, Current and Time**

| Energy (Joules) | Time Energized (Minutes) | Power Applied (Watts) | Current (Amps) | Potential (Volts) | Resistance (Ohms) |
|---|---|---|---|---|---|
| 3000 | 60 | 0.8333 | 0.1349 | 6.1779 | 45.80 |
| 5000 | 60 | 1.3889 | 0.1741 | 7.9757 | 45.80 |
| 6000 | 60 | 1.6667 | 0.1908 | 8.7369 | 45.80 |
| 10000 | 60 | 2.7778 | 0.2463 | 11.2793 | 45.80 |
| 20000 | 60 | 5.5556 | 0.3483 | 15.9513 | 45.80 |
| 30000 | 60 | 8.3333 | 0.4266 | 19.5363 | 45.80 |
| 10000 | 120 | 1.3889 | 0.1741 | 7.9757 | 45.80 |
| 20000 | 120 | 2.7778 | 0.2463 | 11.2793 | 45.80 |

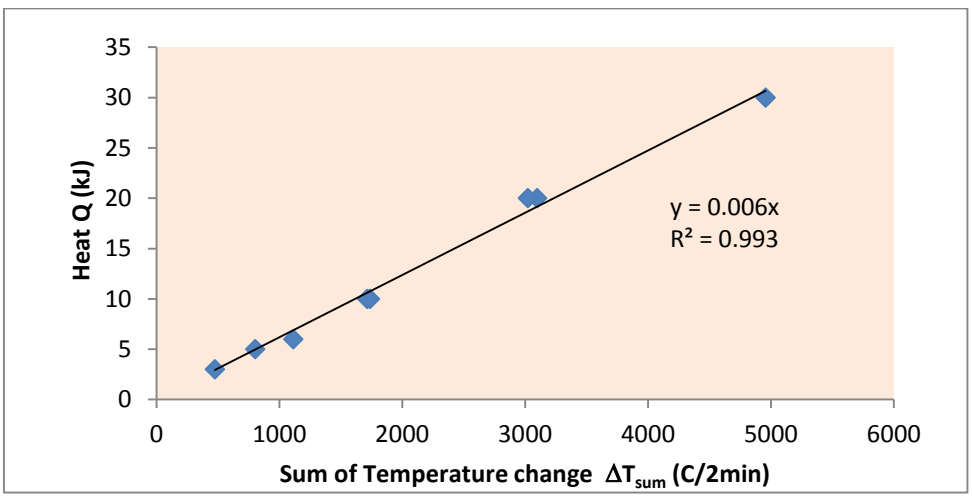


**Figure 3.5: Example of calibration curve of generated heat and the sum of temperature change (37°C temperature controlled room)**

Usually, 4 double-vessels will be grouped as one set in an insulated box placed in a temperature controlled system. For quality assurance, at least one double-vessel will be included as standard or control with each sample group. Two or three thermocouples will be placed in the control vessel. The relative standard deviation of the temperature in the control vessel should be within 5%. If the temperature of control fails, the experiment will be repeated. It was assumed that 0 heat generation in the last 10 hours of measurement and the average temperature difference between the control and sample was 0 in that period. The system will be verified every 6 months with one standard, and the slope of $\sum\Delta T/Q$ will be calculated. The variation limit of $\sum\Delta T/Q$ is ± 15%. If the verification fails, the heat experiment in that period will be repeated. To isolate the heat transfer among the vials, insulation materials will be placed between the vials in the box.

Similar to the temperature profile study, many factors might influence the heat generation process, e.g. the mass of salt cake, particles size, composition of liquids, and environmental temperature. These factors will also be tested.

Moreover, reference materials (sodium chloride, potassium chloride, aluminum nitride, aluminum carbide, aluminum oxide and powder metallic aluminum) will be tested to evaluate the interactions of pure aluminum compounds upon liquid addition under the same experimental conditions. The evaluation will follow the same steps outlined earlier for SAP waste samples.

### *3.4.4 Hydrogen production from metallic aluminum – mechanism study*

A pre-test study of reference materials indicated that the hydrolysis process of aluminum nitride (AlN) can significantly enhance the reaction of metallic aluminum with water, even at room temperature conditions. It was found that the rate of hydrogen generation can be quite high, e.g. 300 ml $H_2$/min/0.5 g Al at 37C. The promotion effect of the product of AlN hydrolysis on the rate of hydrogen generation from the reaction of metallic aluminum as well as the interaction of salt will provide some insight for understanding the mechanisms of the reaction of SAP waste with water. It is known that the product of AlN hydrolysis is related to time, the relation of aging process and its promotion effect will be studied on the generated gas volume, composition and rate.

- Experiment 1 (aging process of AlN hydrolysis): The product of AlN hydrolysis is changeable with the aging process, different species of aluminum hydroxide or oxide might be formed. The pH in the suspension will be monitor. The hydrogen generation from the reaction of metallic aluminum with water will be studied with the different aged byproduct of AlN hydrolysis. The suspension of AlN will be made as 1 g AlN with 20 ml DI water in a closed container (e.g. 40 mL VOA vial with screw cap). The aging time will be from less than 2 h , 24h, 72h, … till 15 week. Adding aged AlN suspension into the reactivity test apparatus (e.g. 60 mL VOA vial with screw cap) with metallic aluminum, monitor the volume of gas generated using a gas syringe, and count the time. The pH of the product generated from the aluminum reaction will be monitor after gas collection. The experiment will be studied at the room temperature (25$^{o}$C).
- Experiment 2 (the effect of mass of product of hydrolysis AlN): The amount of metallic aluminum and the total volume of water will be fixed, the initial mass of AlN will be 0.5, 1 and 2.0 gm (20 ml). Adding aged AlN suspension into the reactivity test apparatus (e.g. 60 mL VOA vial with screw cap) with metallic aluminum, monitor the volume of gas generated using a gas syringe, and count the time. The reaction process will be repeated if the promotion effect is observed.
- Experiment 3 (the effect of mass of metallic aluminum): The amount of suspension of AlN and total volume of solution will be fixed, the initial metallic aluminum mass will be 0.1, 0.5 and 1.0 gm. Adding aged AlN suspension into the reactivity test apparatus (e.g. 60 mL VOA vial with screw cap) with metallic aluminum, monitor the volume of gas generated using a gas syringe, and count the time. The reaction process will be repeated if the promotion effect is observed.

## 3.5. Toxicity Characteristic Leaching Procedure (TCLP) of SAP Waste

The toxicity characteristic leaching procedure (TCLP) will be conducted on SAP waste to determine if the SAP waste is characteristically hazardous. The TCLP will be performed according to the EPA method 1311. The following minor modification to the test will be applied: 1) 10 g SAP waste sample will be used instead of 100 g sample but the liquid to solid ratio will be 20:1 as per the standard procedure, and 2) Whatman 0.45 μm PVDF filters will be used instead of the 0.7 μm glass fiber filters mentioned in the standard procedure. Other than those two modifications, all other steps will be conducted according to the TCLP standard procedure.

## 3.6 Model Thermal Changes in Landfills Accepting SAP waste

### 3.6.1 Model Selection

The selected model will be used to estimate thermal changes in MSW landfills accepting SAP waste. The model will not be used to make definitive conclusions, rather it will be used to provide information about the trends that are likely to occur.

### 3.6.2 Model Attributes/Capabilities

To simulate the thermal changes in MSW landfills as a result of co-disposal of SAP waste with MSW, the selected model must be able to run on an Intel Pentium Dual-Core processor and have the following capabilities:

- Analyze steady-state and transient energy flow, 2-D energy flow in a cross section of in plan view
- Include boundary condition types such as temperature, flux, convective surfaces and imported climate data.

### 3.6.3 Model to be Used

TEMP/W is a finite element CAD software product for analyzing thermal changes in the ground due to environmental factors or the construction of facilities such as buildings or pipelines. The comprehensive formulation makes it possible to analyze both simple and highly complex geothermal problems. TEMP/W can be applied to the geothermal analysis and design of geotechnical, civil, landfill, and mining engineering projects, including facilities subjected to freezing and thawing temperature changes. It was selected as the model to be used for simulating thermal changes in MSW landfills as a result of co-disposal of SAP waste with MSW.

A SOP for the TEMP/W is provided in Appendix A (see SOP 13).

### 3.6.4 Requirements, Parameters, and Assumptions

The requirements needed for application of the selected model include information for input parameters using characterization tests, literature, and other references.

The parameters involved TEMP/W Software include the thermal properties of the waste materials (solid waste and SAP waste), liners and final covers in addition to the boundary conditions (e.g., climate, subgrade temperature, biological activity and chemical reactions of SAP with liquid). These input parameters are classified to three types:

- Type 1: data for parameters that is generated in our laboratory under this approved QAPP which means it is not secondary data.
- Type 2: data for parameters to be collected from peer-reviewed articles, text books and interviews with regulators and facility owners/operators (secondary data).
- Type 3: data for parameters that is being assumed by the research team because there is lack of information in the literature regarding those parameters.

For type 2 (secondary data): since textbooks and peer-reviewed articles have been through a rigorous review process, it may be a valid if we assumed that the data are accurate. However, the research team will take two actions to make sure the secondary data are accurate: 1) Try to identify more than one source for the each input parameter and approve the value of the required parameter if 2 sources or more agree on the value/range for that parameter, 2) contact experts in the field and get their opinion on that obtained values for each parameters.

For type 3 data: the research team will conduct sensitivity analysis on the assumed parameters and if the parameter prove to impact the results significantly then the team will try to find a justifiable value for the parameter by further deep literature search or by contacting experts in the filed to get their recommendations for the more realistic values for the required parameter. In the event there is still no consensus on the value of the parameter, the model will be run based on 2-3 different scenarios for that particular parameter (e.g., a low, medium and high value), and results from all three scenarios will be presented.

- Assumptions and Explanations
    - Only the parameters that have a large impact on the thermal changes over time in the MSW landfills will be included as input into the model.
    - MSW landfill cell that accepts 1000 tons per year of MSW over 2 years period of time.
    - Two scenarios of disposing of SAP waste in MSW landfills will be assumed 1) the SAP waste is being discharged at a dedicated spot within the landfill cell, and 2) the SAP waste is being mixed with the MSW.
    - The proportion of SAP waste to MSW will be varied in order to test different disposal scenarios of SAP waste.
    - The total heat generation from SAP waste (obtained from the experimental work) will be converted to heat generation rates by assuming three possible scenarios: 1) the heat will

be generated over a period of 1 month, 2) heat will be generated over a period of 1 year, and 3) the heat will be generated over a period of 10 year.

### 3.6.5 Quality of the Model Output

The TEMP/W model does not generate any QC outputs. Nonetheless, in order to ensure the quality of the model output, accuracy and precision will be checked as follows:

- The accuracy of the model output will be verified by using the TEMPW/W model to calculate the thermal changes of a standard material. For example, the thermal changes of water as a result of applying different ambient temperatures as boundary conditions will be tested using the TEMP/W model. The output results will be compared to results from the literature.
- The output of model will be verified by another independent research laboratory with the same input parameters and boundary conditions.
- The output of model is a report or paper or presentation related to the managment of secondary aluminum dross.

# SECTION 4.0

# SAMPLING PROCEDURES

## 4.1 Site Preparation

The main facilities that generate SAP wastes will be identified in collaboration with the Aluminum Association and the Environmental Research and Education Foundation. The SAP waste samples will be collected monthly from various secondary aluminum processing facilitie**s.** The selected facilities have been identified by the Aluminum Association to cover a wide range of SAP processes.

## 4.2 Sampling Procedures

In order to achieve a representative sampling of SAP wastes, double samples (BHD and SC) will be taken from the facility directly. The SAP waste will be piled at the generation site per waste type . After cooling (usually 72-120 hrs), each pile would be reduced following ASTM Method C702 – 98 (2003) "standard practice for reducing samples of aggregate to testing size". Two samples will be collected with pre-cleaned sampling equipment.

## 4.3 Sample Containers/Quantities

The first sample will be approximately 400 to 800 g sample collected in a tightly coved plastic container that will be delivered to the EPA CHL Facility within 24 hours in a shipping container. The second sample (10-20 kg) will be stored unpreserved in a tightly covered plastic container at the test site or the sampling company (*not at CHL facility*) for further analysis, based on findings.

## 4.4 Sample Processing/Holding Times

Within two weeks of receipt, in order to obtain representative subsamples, the individual samples will be processed as follows:

- The samples will be placed in a stainless steel pan and crushed to pass a 9-mm sieve.
- After thorough mixing, approximately 0.5 kg of the 9-mm sieved materials will be further size reduced down to a size of 2 mm using a grinding machine (Preiser Scientific).
- Approximately 50 gm of the 2 mm size SAP waste will be further reduced to a size of 0.05 mm using a sealed stainless steel jar mill equipped with zirconia grinding beads. The jar mill will be rotated at 20 rpm for 24 hours.
- The 2- mm size will be used for the chemical and physical analysis (pH, conductivity, moisture content and water holding capacity) in the laboratory and the 0.05-mm size will be used for the chemical and XRD analysis.

## 4.5 Sample Identification

The sample label should include the following:

1. Waste type (salt cake (SC) or baghouse dust (BHD))

2. Sampling location (Each facility will be identified using a letter of the alphabet)
3. Sampling ID (4 digit)

An example label will be BHD-A-2452 meaning that the sample 2452 is a baghouse dust sample collected from facility A.

Each generated sample will have a unique ID that describes and differentiates each sample. All vials will also be labeled following the procedure explained above. Labels for samples to be processed will contain the sample matrix (leachate or solid), sample port number or location, date and time of sampling, the test (e.g., metals, $NH_3$) that will be performed on this sample. A laboratory notebook will be used to record the results of the analysis.

# SECTION 5.0

# MEASUREMENT PROCEDURES

The following laboratory protocols will be carried out to achieve the research objectives as required by the Aluminum Association and the Environmental Research and Education Foundation.

## 5.1 SAP Waste Characterization

The experimental plan involves the determination of the total metal composition and mineral phases for the SAP waste. Additionally, the moisture content, density and water holding capacity will be measured for all SAP waste samples. The quantity of samples to be tested as well as the frequency of sampling are summarized in Table 5.1. The samples containers specifications, the preservation methods and holding times are presented in Table 5.2.

**Table 5.1 Summary of Solid Sampling and Schedule**

| Parameter | Type of measurement | Frequency | Total # of samples |
|---|---|---|---|
| **Moisture** | Critical | Once | 100 |
| **Water capacity** | Non-Critical | Once | 100 |
| **Metals** | Critical | Once | 50 |
| **Mineral** | Non-Critical | Once | 50 |

**Table 5.2 Sample Containers, Preservation and Storage Time**

| Parameter | Amount of sample | Vial type and volume | Preservation | Max. holding time |
|---|---|---|---|---|
| **Matrix: Solid** | | | | |
| Moisture | 10 g | 50 ml plastic vial | NA | NA |
| Metals | 0.1 g | 50 ml plastic vial | $HNO_3$, refrigerate | 6 months |
| Mineral | 2 g | 50 ml plastic vial | NA | NA |
| **Matrix: Aqueous** | | | | |

| | | | | |
|---|---|---|---|---|
| pH | NA | 50 ml plastic vial | NA | <3 h |
| Conductivity | NA | 50 ml plastic vial | NA | <3 h |
| Dissolved Metals | 20 ml | 50 ml plastic vial | $HNO_3$, refrigerate | 6 months |
| Anions ($SO_4^{2-}$, $F_3^-$, and $Cl^-$) | 25 ml | Plastic/Glass | Cool @ 4 $^0$C | 28 days |
| Dissolved $NH_3$ | 5 ml | 50 ml EPA glass vial | $H_2SO_4$, refrigerate | 7 days |
| Matrix: Gas | | | | |
| Composition and concentration | 20 ml | Plastic/Glass | N/A | <3 hours |

## 5.2. Experimental Sampling

The total number of samples and the sampling frequency for the gas and leachate generated from the SAP waste reactivity testing presented in Section 3.4 are summarized in Table 5.3.

**Table 5.3 Summary of Sampling of the Waste Reactivity Testing**

| Parameter | Type of measurement | Frequency | Total # of samples |
|---|---|---|---|
| Temperature | Critical | Continuously | 200 |
| Heat | Critical | Continuously | 80 |
| Gas volume, ml | Critical | Once | 50 |
| Gas Composition, % ($CH_4$, $CO_2$, $H_2$, $O_2$, $N_2$) | Critical | Once | 50 |
| $N_2O$ Concentration, ppm | Non-Critical | Once | 50 |
| NH3 (Gas)concentration, | Critical | Once | 50 |
| Dissolved metal, mg $kg^{-1}$ | Critical | Once | 50 |
| Dissolved ammonia, mg $kg^{-1}$ | Critical | Once | 50 |
| TCLP metals (Ag, As, Ba, Cr, Cd, Se, and Pb),mg $kg^{-1}$ | Critical | Once | 50 |
| pH | Critical | Once | 50 |
| EC | Non-Critical | Once | 50 |

## 5.3 Measurement Methods

The analytical methods for the SAP waste reactivity testing are outlined in Table 5.4. It is noted that the temperature measurements will be recorded automatically and the results will be logged directly to a 34970 Agilent data logger system. The recorded data can then be exported to Microsoft Excel spreadsheets from the Agilent software program.

**Table 5.4 Outline of Analysis Methods**

| Parameter | Measurement | Instrument | Analytical Method/SOP |
|---|---|---|---|
| **Leachate** | | | |
| pH | Critical | Automatic Temperature and Compensation electrode | I.M./ EPA Method 150.1 pH/SOP 3 |
| Conductivity | Non-Critical | Electrodless Toroidal Conductivity Sensor | I.M./ EPA Method 120.1/SOP 3 |
| Ammonia ($NH_4$ in solution) | Non-Critical | Orion 4-Star pH/ISE Benchtop Meter | I.M., EPA method 350.3 Nitrogen-Ammonia / SOP 7 |
| Dissolved Metals | Critical | Thermo Elemental 61 E trace analyzer ICP-AES | SW-846 Method 6010B /SOP 9 |
| TCLP metals (Ag, As, Ba, Cr, Cd, Se, and Pb) | Critical | Thermo Elemental 61 E trace analyzer ICP-AES | SW-846 Method 6010B /SOP 9 |
| Dissolved Anions | Non-Critical | Ion Chromatograph (IC), using AS-18 Dionex chromatography column. | EPA Method 300 / SOP #10 |
| Solids | | | |
| Temperature | Critical | Type T Thermocouple. | N.A./I.M. |
| Heat | Critical | Type T Thermocouple. | N.A./I.M |
| Moisture | Critical | 4 digit balance and 105°C oven | SOP 4 |
| Density | Non-Critical | 2 digit balance and 20.0 ml cylinder | SOP 5 |
| Water Holding Capacity | Non-Critical | 2 digit balance and 50 ml cylinder | SOP 6 |
| Total Metals | Critical | IRIS Intrepid ICP-AES | Method 3051 for acid digestion and Method 6010 B for ICP analysis, SOP 8, and SOP 9 |
| Crystallinity (XRD) | Non-Critical | Philips instruments X'Pert-MPD system | N.A./I.M.<br><br>SOP 10 |

| | | | |
|---|---|---|---|
| | | X-ray Diffraction Spectrometer | |
| **Gas** | | | |
| Gas volume | Critical | Gas Syringe | N.A./I.M./EPA method OPPTS 835.3400 |
| Gas Composition ($CO_2$, $H_2$, $O_2$, $N_2$, $CH_4$) | Critical | Gas Chromatograph (GC/TCD) Agilent 6890 N Series | SOP 1 |
| Gas Composition, $N_2O$ | Non-Critical | Gas Chromatograph (GC/ECD) Agilent 6890 N Series | SOP 2 |
| Gas Composition, $NH_3$ | Critical | Orion 4-Star pH/ISE Benchtop Meter | EPA method 350.3 Nitrogen-Ammonia/SOP 7 |
| $H_2S$ | Non-Critical | Jerome 631X Hydrogen Sulfide Meter | I.M. |

N.A: Not applicable
I.M: Instrument Manual

# SECTION 6.0

# Quality Metrics (QA/QC CHECKS)

## 6.1 Data Quality Objectives and Criteria

The accuracy checks, precision, calibration of instrumentation, and the determination of detection limits are used to ensure the quality control and the confidence level of the obtained results. Precise, documented and valid data are needed for the ultimate decisions to be made. To ensure the quality of the data, all instrumentation will be regularly calibrated. QA/QC checks will be done to ensure the precision and accuracy of the data. Table 6.1 summarizes the QA/QC checks for each monitoring parameter.

### 6.1.1 Definitions

***Accuracy*** is the nearness of a test result to the true value (recovery). Both, standard addition (spiking) and standard checks are common techniques for checking the accuracy. For matrix spikes, the percent recovery could be calculated as follows:

$$\%R = \frac{(Cs - Cu)}{Ca} \times 100$$

Where: $C_s$ = Concentration in spiked aliquot

$C_u$ = Concentration in unspiked aliquot

$C_a$ = Actual concentration of spike added

For standard checks, the percent recovery will be calculated as follows:

$$\%R = \frac{Cm}{Ca} \times 100$$

Where: $C_m$ = measured concentration of the check standard.

$C_a$ = actual concentration of the check standard.

***Precision*** is how repeated measurements closely agree with each other. Laboratory duplicates and triplicates will be used to ensure precision; the relative percent difference (RPD) between duplicates will be calculated as follows:

$$\% RPD = \frac{|C1 - C2|}{0.5(C1 + C2)} \times 100$$

Where: $C_1$ = Concentration of the analyte in the sample

$C_2$ = Concentration of the analyte in the matrix duplicate

The relative standard deviation between replicates will be calculated as follows:

$$\% RSD = (\frac{S}{y'}) \times 100$$

Where: S = Standard deviation

$y'$ = Mean of the replicates

***Method Detection Limit (MDL)*** is the lowest concentration that is different from zero with a 99% level of confidence. To determine the MDL, the lowest standard concentration used for the calibration will be injected number of times and the MDL will be calculated using the following equation:

$$MDL = t(n-1, 1-\alpha = 0.99) \times S$$

Where: n = the number of replicates

S = Standard deviation of the replicates

The MDLs for ICP-AES analysis performed at the EPA CHL Facility is provided in Table 6.2.

**Table 6.1 Summary of QA/QC Checks**

| Parameter | QC Check | Method | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| pH | Initial Calibration* | 2 point calibration | At the beginning | Within ± 0.1 pH unit of the correct value | 1. Re-calibrate |
| Conductivity | Initial Calibration* | 1 point calibration | At the beginning | Within ±2 % of the full range of the probe | 1. Re-calibrate |
| Ammonia ($NH_4$) | Initial calibration | 4 point calibration | As needed | $R^2 \geq 0.995$ | 1. Re-calibrate |
| | Continuing calibration | Run mid point standard | Every 12 samples | ±20 % of the actual concentration | 1. Re-calibrate |
| | Precision | Sample duplicate | Every 6 samples | ≤ 20% RPD | 1. Re do triplicate<br>2. Investigate the problem |
| | Blank | Laboratory blank | Every 6 samples | ≤ Reported detection limit | 1. Investigate problem<br>2. Remove contamination<br>3. Check other blanks |
| | Accuracy | Matrix Spike | Every 6 samples | 100 ± 20 % recovery | 1. Re-run spike<br>2. Check calculations |

| | | | | | |
|---|---|---|---|---|---|
| | | | | | 3. Re-run samples as required |
| Metals | Calibration | 4 point calibration | Before each run | $R^2 \geq 0.995$ | 1. Re-calibrate |
| | Precision | Sample Duplicate | Every 12 samples | ≤ 15 % RPD for macro elements (Al, Fe, Ca, K, Na, S and Mg) and ≤ 25% for trace metals (Ag, Ba, Cu, Mn, Zn ,Cd, As, Se, Cr and Pb). | 1. Re-analyze<br>2. Investigate the problem |
| | Blank | Laboratory blank | Every 12 samples | ≤ Reported detection limit | 1. Investigate problem<br>2. Remove contamination<br>3. Check other blanks |
| | Accuracy | Check Standard | Every 12 samples | 100 ± 15 % recovery for major metals and 100 ± 25 % for trace metals | 1. Re-calibrate<br>2- Re-run samples as required |
| Anions ($SO_4^{2-}$, $F^-$ and $Cl^-$) | Calibration | 4 point calibration | As needed | $R^2 \geq 0.995$ | 1. Re-calibrate |
| | Precision | Sample duplicate | Every 6 samples | ≤ 20% RPD | 1. Re do triplicate<br>2. Investigate the problem |
| | Blank | Laboratory blank | Every 10 samples | ≤ Reported detection limit | 1. Investigate problem<br>2. Remove contamination<br>3. Check other blanks |
| | Accuracy | Check Standard | Following calibration and every 10 samples | 100 ± 15 % recovery | 1. Re-run<br>2. Check calculations<br>3. Re-run samples as required |

| | | | | | |
|---|---|---|---|---|---|
| Temperature | Initial Calibration* | 1 point calibration | At the beginning, and once a year | Within ± 2 % of the full range of the probe | 1. Use new thermocouple ( within 1 year) |
| Heat measurement | Initial Calibration* | 1 point calibration | Every 6 month | Within ± 15 % | 1. Re-do sample<br><br>2. Investigate the problem- Flag data |
| Moisture Content | Initial Calibration* | 1 point calibration | At the beginning | Within ± 5 % of the full range of the probe | 1. Re-calibrate |
| Density | Initial Calibration* | 1 point calibration | At the beginning | Within ± 5 % of the full range of the probe | 1. Re-calibrate |
| Water holding capacity | Initial Calibration* | 1 point calibration | At the beginning | Within ± 5 % of the full range of the probe | 1. Re-calibrate |
| Total metal | Calibration | 5 point calibration | Before each run | $R^2 \geq 0.99$ | 1. Re-calibrate |
| | Precision | Sample duplicate | Every 12 samples | ≤ 25% RPD | 1. Rerun<br><br>2. Prepare new standards<br><br>2. Check calculations |
| | Accuracy | Standard check | With every run | 75-125 % recovery | Re calibration |
| Crystallinity (XRD) | Calibration | Reference standard | Prior to sample analysis | | |
| Gas volume | Initial Calibration* | 1 point calibration | At the beginning | Within ± 5 % of the full range of the probe | 1. Re-calibrate |
| Gas Composition ♣ | Initial Calibration | 6 point calibration | Monthly | $R^2 \geq 0.990$ | 1. Re-calibrate |
| | Precision | Sample Duplicate | Every 12 samples | ≤ 20% RPD | 1. Re-do sample<br><br>2. Investigate the problem- Flag data |
| | Accuracy | Standard Check | Twice a Week | 100 ± 20 % recovery | 1. Re-calibrate |

* Calibration for the probes, electrodes, thermocouples and TDR.
♣ Calibration of the Gerome meter (for $H_2S$) is performed annually by the manufacturer. Calibration verification samples will be analyzed prior to each analysis using a known hydrogen sulfide standard and must be within ± 20 % of the standard value. If the calibration verification sample exceeds the set criteria, the meter will be sent to the manufacturer for NIST-traceable calibration.

## 6.2 Types of QC Samples

***Method Blank*** is a generated sample prepared from a clean matrix (generally deionized water and it could be gas or solid according to the type of matrix), and it is treated exactly as a sample. It is prepared to check for contamination.

***Calibration Blank*** is a volume of reagent water without the analyze. The concentration of the analyze should be less than three times the instrument detection limit

***Matrix Spike*** is a sample with a known concentration of the analyze added to original sample and is used to assure that the recovery of the target compounds is acceptable for the matrix involved.

***Standard Check*** is a sample with a known concentration of the analyze is used to assure that the recovery of the target compounds is acceptable for the matrix involved.

**Table 6.2 MDL and Accuracy for ICP-AES Method 6010B†**

| Metal Name | True Value (µg/ml) | Mean Concentration (µg/ml) | Accuracy (%) | STDEV (µg/ml) | MDL (µg/ml) | MRL (µg/ml) |
|---|---|---|---|---|---|---|
| Al | 0.040 | 0.031 | 79% | 0.001 | 0.004 | 0.013 |
| Ag | 0.010 | 0.011 | 108% | 0.000 | 0.001 | 0.003 |
| Ba | 0.020 | 0.020 | 100% | 0.001 | 0.002 | 0.007 |
| As | 0.050 | 0.055 | 110% | 0.001 | 0.004 | 0.011 |
| Ca | 0.100 | 0.074 | 74% | 0.015 | 0.048 | 0.154 |
| Cd | 0.020 | 0.023 | 113% | 0.0002 | 0.001 | 0.002 |
| Cr | 0.050 | 0.053 | 106% | 0.0004 | 0.001 | 0.004 |
| Cu | 0.020 | 0.020 | 98% | 0.001 | 0.004 | 0.011 |
| Fe | 0.040 | 0.045 | 111% | 0.001 | 0.002 | 0.008 |
| K | 0.100 | 0.134 | 134% | 0.039 | 0.123 | 0.390 |
| Mg | 0.100 | 0.073 | 73% | 0.001 | 0.004 | 0.012 |
| Mn | 0.008 | 0.007 | 93% | 0.000 | 0.001 | 0.002 |
| Na | 0.100 | 0.111 | 111% | 0.009 | 0.027 | 0.087 |
| P | 0.100 | 0.144 | 144% | 0.038 | 0.119 | 0.379 |
| Pb | 0.050 | 0.053 | 106% | 0.002 | 0.005 | 0.015 |
| S | 0.050 | 0.046 | 93% | 0.005 | 0.015 | 0.049 |
| Se | 0.050 | 0.052 | 104% | 0.001 | 0.003 | 0.010 |
| Zn | 0.008 | 0.008 | 94% | 0.000 | 0.000 | 0.001 |

† The results of As, Cd, Cr, Pb and Se are based on the results of July, 2012 and the others are based on the results of Nov , 2011.

## 6.3 Model Calibration and Validation

Many authors have shown TEMP/W to be accurate at simulating thermal changes of various problems as a result of changes in boundary conditions. The software has been successfully used for many applications such as thermal design of roads and airstrips, ground freezing for soil stabilization, insulation design for shallow buried piping, thawing or freezing beneath heated or chilled structures, convective cooling of surfaces and many geotechnical applications (Thermal Modeling with TEMP/W, 2007). Although the parameters and problem setup may vary from any one specific study, the conclusion holds true that TEMP/W is a reliable program. For details of the modeling procedure please see SOP 13.

# SECTION 7.0

# DATA ANALYSIS, INTERPRETATION, AND MANAGEMENT

## 7.1 Data Reporting

Table 7.1 summarizes the reporting units for the various analyses. At the end, each monitoring parameter will be averaged on treatment scale and analysis of variance will be carried out to determine the effect of SAP on temperature and gas emission under different experimental conditions.

Temperature will be recorded by the data logger system and transferred into Microsoft Excel ™ spreadsheets. The remaining analysis results will be recorded in a laboratory notebook and each page will be dated and signed by the person who performs the analysis, then, those data will be fed manually to Excel spreadsheets for statistical analysis. Calculations will be carried out on a computer and will be checked initially by the analyst for gross error and miscalculation. The calculations and data entered into computer spreadsheets will be checked by a second analyst for accuracy by printing out the calculation or data spreadsheet and checking the calculation by hand or checking each entry of data from the original. These copied spreadsheets will be initialed and dated by the second analyst doing the checking, noting the errors.

**Table 7.1 Reporting Units**

| Parameter | Unit |
|---|---|
| pH | pH Units |
| Conductivity | µS/cm |
| Ammonia ($NH_3$, Gas) | ppmv |
| Metals | mg/L |
| Anions | mg/L |
| Crystallinity (XRD analysis) | -- |
| Temperature | $^0$C |
| Moisture Content | % M/M |
| Heat | kJ/g |
| Water holding capacity | g/g |
| Gas volume | L or ml |
| Gas Composition | % V/V or ppm |

Statistical analysis and graphical representation of the data will be performed using Microsoft Excel 2007, Software JMP9.0 and SigmaPlot11.0. The mean, standard error, minimum and maximum values will be used to summarize the elemental composition, mineral phases, and the temperature change as well as the gas composition, and productivity. For the correlations between the temperature change and mineral phases, or relation between heavy metal content and aluminum content, the Pearson product-moment correlation and Spearman's rank correlation will be employed to calculate the p level. The Pearson product-moment correlation coefficient is a

measure of the correlation (linear dependence) between two variables X and Y, giving a value between +1 and −1 inclusive. Spearman's rank correlation coefficient is a non-parametric measure of statistical dependence between two variables. It assesses how well the relationship between two variables can be described using a monotonic function. If there are no repeated data values, a perfect Spearman correlation of +1 or −1 occurs when each of the variables is a perfect monotone function of the other. If $p<0.05$, it is assumed that the relation is significant; and if $p<0.01$, the correlation is very significant. If $p>0.05$, it is believed that there is no correlation.

## 7.2 Data Handling and Disposal

Original project records that are generated by PTSI will be maintained by the WA Leader in accordance with the ORD Policies and Procedures Manual, Section 13.2 Paper Laboratory Records.

## 7.3 Data Reduction and Validation

Data reduction and validation will be done using Microsoft Excel spreadsheets to minimize the calculation errors. A second reviewer will check the reduced data. The reviewed data will include calibration and QC data. The reviewer will also review the laboratory notebooks on a monthly basis. Any detected errors will be corrected before the data is released to the EPA.

## 7.4 Data Storage

Laboratory records will be maintained in accordance with Section 13.2, Paper Laboratory Records, of the Office of Research and Development (ORD) Policies and Procedures Manual. Controlled access facilities that provide a suitable environment to minimize deterioration, tampering, damage, and loss will be used for the storage of records. Whenever possible, electronic records will be maintained on a secure network server that is backed up on a routine basis. Electronic records that are not maintained on a secure network server will be periodically backed up to a secure second source storage media, transferred to an archive media (e.g., compact discs, optical discs, magnetic tape, or equivalent), or printed. Electronic records that are to be transferred for retention will be transferred to an archive media or printed, as directed by EPA.

# SECTION 8.0

# REPORTING

## 8.1 Project Deliverables

Monthly reports will be prepared by the PTSI WA Leader and sent to the PTSI On-site Technical Manager and Project Manager, and submitted to EPA on the 20$^{th}$ of every month. Every ten months an interim report will be prepared to cover all aspects of the project such as the work progress, a summary and discussion of the results, any problems encountered, the adopted solution to the problem and any deviations from the procedures described in the QAPP.

## 8.2 Final Report

The final report will be prepared at the end of the project to summarize all the project aspects, give the final results, the conclusions and the recommendations. The reports will be submitted in both hard and electronic copies. All the reports will be submitted to the EPA Project Officer through the EPA Work Assignment Manager.

## 8.3 Draft Manuscript

The produced final report will be the basis for draft manuscripts that will be produced by Dr. Xiaolan Huang and the project personnel as directed by the EPA WA Manager.

# SECTION 9.0

# REFERENCES

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Yoshimura, H.N., et al. (2008) Evaluation of aluminum dross waste as raw material for refractories. Ceramics International 34, 581-591

**SOP 1**
**SOP for Major Gas Analysis using Gas Chromatography (GC)**

**Refer SOP:** LRPCD SOP M 02.0
NRMRL LRPCD_M 02_0 GC Headspace Analysis for Bioreactor Landfill Gas.pdf

# SOP 2
# SOP for $N_2O$ Gas Analysis using Gas Chromatography (GC-ECD)

**Refer SOP:** LRPCD SOP M 09.0
NRMRL LRPCD_M 09_0 Analysis of Nitrous Oxide Headspace Samples by GC-ECD.pdf

# SOP 3
# PH/Conductivity measurement using the Accumet® Portable AP85 Waterproof Meter

## Scope
This procedure is used to measure the pH and EC for samples generated during the different tests ( EPA methods 150.1 and 120.1).

## Apparatus
- pH and Conductivity meter, Accumet® Portable AP85 Waterproof Meter or equivalent
- pH electrode, Conductivity probe
- Disposable plastic container, 75 ml or larger

## Reagents and materials
- Standard buffer, pH 4.01
- Standard buffer, pH 7.00
- Standard buffer, pH 10.01
- Conductivity Standard Solution
- Deionized water (DI)

## Procedure

### 1. pH Calibration

It is recommend that calibration be done at least two buffers that bracket (one above and one below) the expected sample range.

### 1.1. Preparing for pH calibration

This meter can calibrate up to 5 pH buffer values to ensure accuracy across the entire pH range. Selection is made from the following buffer options:

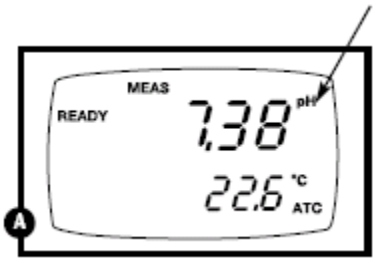
USA: pH 1.68, 4.01, 7.00, 10.00, 12.45
NIST: pH 1.68, 4.01, 6.86, 9.18, 12.45

The meter automatically recognizes these standard buffer values, which makes pH calibration faster and easier.

The protective electrode storage bottle or rubber cap of the probe is removed before calibration or measurement. If the electrode has been stored dry, the probe is re-hydrated in tap water for 10 minutes before use to saturate the pH electrode surface and minimize drift. The probe in rinsed with deionized water after use, and stored in electrode storage solution. If storage solution is not available, pH 4.0 or 7.0 buffer is used.

**1.2. To calibrate for pH**

1. If necessary, the MODE key is pressed to select pH measurement mode. The pH indicator appears in the upper right hand corner of the display.

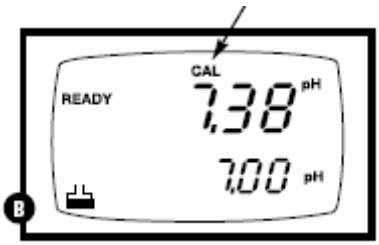



2. The pH electrode is rinsed thoroughly with deionized water or a rinse solution. Wiping the probe is not advisable; this causes a build-up of electrostatic charge on the glass surface.

3. The pH electrode is dipped into the calibration buffer. The end of the probe must be completely immersed into the sample. The probe is gently stirred to create a homogeneous sample.

   **Notes:** The temperature element is in the conductivity cell. For temperature compensated readings, the conductivity cell is dipped into the calibration buffer as well.

4. CAL/MEAS is pressed to enter pH calibration mode. The CAL indicator lights up. The primary display shows the measured reading while the smaller secondary display indicates the pH standard buffer solution.




5. The measured pH value is allowed to stabilize. If the READY indicator has been activated the READY annunciator lights when the reading is stable.

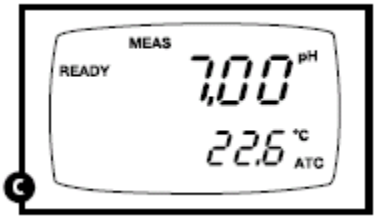
1. Press ENTER to confirm calibration. The meter is now calibrated to the current buffer. The lower display scrolls through the remaining buffer options.

   - White performing a multipoint calibration, step 7 is followed next.
   - While performing a one-point calibration, step 9 is followed next.

2. The electrode is rinsed with deionized water or a rinse solution, and dipped in the next pH buffer.

3. Steps 5 to 8 are followed for additional calibration points.

4. When calibration is complete, CAL/MEAS is pressed to return to pH measurement mode.




Notes:

To exit from pH Calibration mode without confirming calibration, ENTER is not pressed in step 6. Instead CAL/MEAS is pressed.

If the selected buffer value is not within ±1.0 pH from the measured pH value: the electrode and buffer icon blink and the ERR annunciator appears in the lower left corner of the display.

**2. Conductivity calibration**

Up to 5 conductivity points can be calibrated, using a maximum of one point per range (listed below):

| Range | Conductivity: |
|---|---|
| R1 | 0.00-19.99 µS |
| R2 | 0.00-199.90 µS |
| R3 | 0.00-1999.0 µS |
| R4 | 0.00-19.99 mS |
| R5 | 0.00-199.9 mS |

- While measuring in more than one range, it is made sure to calibrate each of the ranges being measured. All new calibration data will over-ride existing stored calibration data for each measuring range calibrated.

- While measuring in ranges near to or greater than 20 mS (10 ppt), or near to or lower than 100 µS (50 ppm), the meter is calibrated at least once a week to get specified ±1% full scale accuracy.

- While measuring in the mid ranges and the probe is rinsed in deionized water and stored dry. The meter is calibrated at least once a month.

- While taking measurements at extreme temperatures, the meter is calibrated at least once a week.

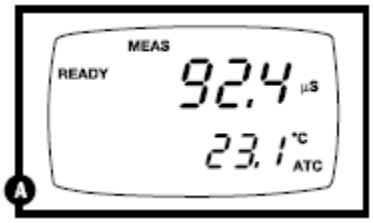
## 2.1. Preparing for conductivity/TDS calibration

For best results, a calibration standard value close to the sample value being measured is selected. Alternatively, a calibration solution value is used that is approximately 2⁄3 the full scale value of the measurement range planned to be used. For example, in the 0 to 1999 µS conductivity range, a 1413 µS solution is used for calibration.

Normalization temperature: The factory default value for normalization temperature is 25°C.

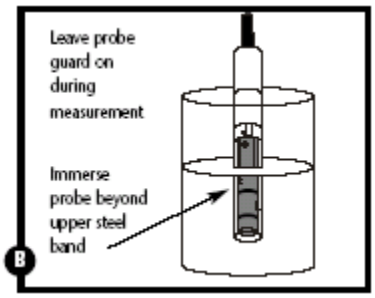
## 2.2. To calibrate conductivity:

1. If necessary, the MODE key is pressed to select conductivity mode.




2. The probe is rinsed thoroughly with deionized water or a rinse solution, then with a small amount of calibration standard.

3. The probe is dipped into the calibration standard. The probe tip is immersed beyond the upper steel band. The probe is stirred gently to create a homogeneous sample.




4. The measured conductivity is allowed value to stabilize. If the READY indicator has been activated the READY annunciator lights up when the reading is stable.

5. CAL/MEAS is pressed to enter conductivity calibration mode. The CAL indicator will appear above the display.

6. ▲ or ▼is pressed to change the value on the primary display to match the value of the calibration standard. The secondary display shows the factory calibrated value.

7. The calibration is confirmed by pressing ENTER. The meter returns to the MEAS (measurement) mode.

8. Steps 1-7 are repeated for other ranges.

   Notes: When entering calibration mode, the meter will display the factory default value. If the meter was previously calibrated, the display may "jump" to the factory default value when switching from measurement to calibration mode.

   To exit from Conductivity Calibration mode without confirming calibration, CAL/MEAS is pressed without pressing ENTER. This will retain the old calibration data in the measuring range of the calibration. The conductivity reading can be offset up to ±40% from the default setting. If the measured value differs by more than ±40%,
   probe is cleaned or replaced as needed.

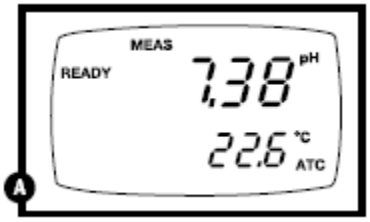
**3. Taking pH Measurements**

To take readings:

1. The pH electrode is rinsed with deionized or distilled water before use to remove any impurities adhering to the probe body. If the pH electrode has dehydrated, it is soak 30 minutes in electrode storage solution, pH buffer, or a 2M–4M KCl solution.

2. Meter is switched on by pressing ON.

3. The MODE key is pressed to select pH measurement mode. The MEAS annunciator appears on the top center of the LCD. The ATC indicator appears in the lower right hand corner to indicate Automatic Temperature Compensation.




**NOTE:** For pH manual temperature compensation, the conductivity cell must be disconnected from the 6-pin connector. The ATC indicator will disappear from the display. A manual temperature compensation value also needs to be set for this (although this function will not be used.)

4. The pH electrode is dipped into the sample. Since the conductivity cell contains the temperature sensor, it is also immersed in the solution. When dipping the probe into the sample, the sensor or the glass bulb of the electrode must be completely immersed into the sample. The probe is stirred gently in the sample to create a homogenous sample.

5. The reading is allowed to stabilize. The reading on the display is noted. If the Ready indicator is selected on, it will appear when the reading is stable.

6. The MODE key is pressed to toggle between pH and conductivity readings.

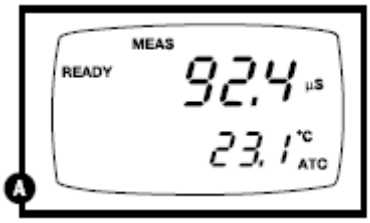
**4. Taking Conductivity Measurements**

To take readings:

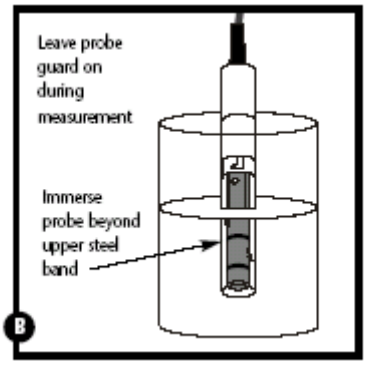
1. The probe is rinsed with deionized or distilled water before use to remove any impurities adhering to the probe body and is dried by shaking in air. To avoid contamination or dilution of the sample, the probe is rinsed with a small volume of the sample liquid.

2. Meter is switched on by pressing ON.

3. The MODE key is pressed to select conductivity (or TDS) measurement mode. The MEAS annunciator appears on the top center of the LCD. The ATC indicator appears in the lower right hand corner to indicate Automatic Temperature Compensation.



4. The probe is dipped into the sample. When dipping the probe into the sample, the tip of the probe must be immersed above the second steel band. The probe is stirred gently in the sample to create a homogenous sample.




5. The reading is allowed to stabilize. The reading on the display is noted. If the Ready indicator is selected on, it will appear when the reading is stable.

## 5. References

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# SOP 4
# Moisture Measurement

**Scope**
This procedure is used to measure the moisture content of the samples during the characterization (ASTM Method D-2216).

**Apparatus**
Mettler Toledo Model AL54 Balance or equivalent
Aluminum weighing dish
Spatula
Blue M (105°C Oven)

**Procedure**
1.0 Balance Calibration
1.1 Calibrate the analytical balance as described in Use of Analytical Balance SOP No.05.SOP.009.00 of LRPCD.
2.0 Sample Drying
2.1 Place the weighing dish on the balance and tare the balance.
2.2 Weigh out about 10g net weight of the sample into a drying dish using a clean spatula. Record it as $W_1$.
2.3 Dry sample at 105ºC for 48 hours.
2.4 Weigh and record dried sample weight in lab book.
2.5 Return sample to oven for 6 hours.
2.6 Reweigh and record dried sample weight in lab book.
2.7 If weight is unchanged < 1%, continue to Step 3.0.
2.8 If weight changed > 1%, return to Step 2.5 and repeat the following steps until a <1% change is observed.
3.0 Weighing
3.1 Record the final weight of the dried sample as $W_2$.

**Analysis**
The moisture content of the sample can be found out using the formula:

$$\% \text{ Moisture Content} = (W_1 - W_2)/W_1 \times 100$$

# SOP 5
# Bulk Density Measurement

## Scope

Bulk density is defined as the weight per unit volume of material. This procedure is used to measure the bulk density of the samples during the characterization (ASTM method for Saturated Hydraulic Conductivity, Water Retention, Porosity and Bulk Density of Putting Green and Sports Turf Root Zones, 2006).

## Apparatus

Mettler Toledo Model AL54 Balance or equivalent
Graduated Cylinder or equivalent

## Procedure

1.0 Balance Calibration

1.1 Calibrate the analytical balance as described in Use of Analytical Balance SOP No.05.SOP.009.00 of LRPCD.

2.0 Fill the sample into a known volume Cylinder

2.1 Measure Volume of cylinder ( V, $cm^3$)

2.2 Place the Cylinder on the balance and tare the balance.

2.2 Fill in the sample through a funnel which is suspended above the cylinder, and the excess material on top of the measuring cylinder is scraped off with a straight edge. (Figure 1)

3.0 Weighing

3.1 Record the net weight of sample in the cylinder as W ( g)

## Analysis

The bulk density of the sample can be found out using the formula:

$$\text{Bulk density (g cm}^{-3}) = W/V$$

Since the variation of bulk density is usually high, five replications are required.

# SOP 6
# Water Holding Capacity Measurement

## Scope

Water holding capacity is defined as the solid that hold generous amounts of water are less subject to leaching. This procedure is used to measure the Water holding capacity of the samples during the characterization (ASTM Method F1815-06).

## Apparatus

Mettler Toledo Model AL54 Balance or equivalent
Graduated Cylinder or equivalent
Absorbent membrane or equivalent

## Procedure

1.0 Balance Calibration

1.1 Calibrate the analytical balance as described in Use of Analytical Balance SOP No.05.SOP.009.00 of LRPCD.

2.0 Fill around 10 g sample into Cylinder and add the water to make it saturated. Put the saturated sample with the cylinder into the absorbent membrane until the excess water is drawn away by gravity. Once equilibrium is reached, the water holding capacity is calculated based on the weight of the water held in the sample vs. the sample dry weight.

2.1 Weight the cylinder with a saturated filtered paper as the bottom, $W_0$

2.2 Fill around 10 gram sample into the known weight cylinder, weight the total weight, $W_1$,

2.3 Make the Cylinder with sample saturated and then placed it on an absorbent membrane until the excess water is drawn away by gravity (see diagram at right).

2.4 Once equilibrium is reached (usually overnight), weight the cylinder with sample again ($W_2$).

## Analysis

The water holding capacity of the sample can be found out using the formula:

$$\text{Water holding capacity } (g\ g^{-1}) = (W_2-W_1)/(W_1-W_0)$$

# SOP 7
# SOP for Ammonia Measurement by ISE

## Scope and Application

This method is applicable to the measurement of 0.03 to 1400 mg $NH_3$-N/L in solution, e.g. surface waters and domestic and industrial wastes (EPA method 350.3). High concentrations of dissolved ions affect the measurement, but color and turbidity do not. Sample distillation is unnecessary.

## Summary of Method

The ammonia-selective electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an electrode internal solution of ammonium chloride. Dissolved ammonia ($NH_{3(aq)}$ and $NH^{4+}$) is converted to $NH_{3(aq)}$ by raising pH to above 11 with a strong base. $NH_{3(aq)}$ diffuses through the membrane and changes the internal solution pH that is sensed by a pH electrode. The fixed level of chloride in the internal solution is sensed by a chloride ion-selective electrode that serves as the reference electrode. Potentiometric measurements are made with the Orion 95-12 gas-sensing electrode on the Orion Star meter or a pH meter with expanded millivolt scale capable of 0.1 mV resolution between −700 mV and +700 mV or a specific ion meter, which will determined ammonia content in solution directly. The method requires no titration and has no turbidity or color interferences.

## Sample Handling and Preservation

Refrigerate at 4°C for samples to be analyzed within 24 h. Preserve samples high in organic and nitrogenous matter, and any other samples for longer storage, by lowering pH to 2 or less with conc $H_2SO_4$.

## Interferences

The problems with ISE measurements are variable stabilization times, changes in the measured potential during repeated analysis, and the effect of variable ionic strength of the solutions. These problems can be minimized by frequent re-calibration.

## Apparatus and Reagents

1. Apparatus
    a. Electrometer: Orion Star meter or a pH meter with expanded millivolt scale capable of 0.1 mV resolution between -700 mV and +700 mV or a specific ion meter.
    b. Ammonia-selective electrode: Ammonia electrode (Orion 9512HPBNWP)
    c. Magnetic stirrer, thermally insulated, with TFE-coated stirring bar (Orion 096019).
2. Reagents
    a. Deionized water: Use for making all reagents.
    b. Sodium hydroxide, 10N ( e.g. Orion 951011).

c. Stock ammonium chloride solution: Ammonia standard, 100 ppm ammonia as N ( e.g. Orion 951207).

**Procedure for Ammonia analysis**

1. *Preparation of standards:* Prepare a series of standard solutions covering the concentrations of 1000, 100, 10, 1, and 0.1 mg $NH_3$-N/L by making decimal dilutions of stock $NH_4Cl$ solution with water.
2. *Electrometer calibration:* Place 100 mL of each standard solution in a 150-mL beaker. Immerse electrode in standard of lowest concentration and mix with a magnetic stirrer. Limit stirring speed to minimize possible loss of ammonia from the solution. Maintain the same stirring rate and a temperature of about 25°C throughout calibration and testing procedures. Add a sufficient volume of 10*N* NaOH solution (1 mL usually is sufficient) to raise pH above 11. If it is necessary to add more than 1 mL of NaOH solution, note volume used, because it is required for subsequent calculations. Keep electrode in solution until a stable millivolt reading is obtained. Do not add NaOH solution before immersing electrode, because ammonia may be lost from a basic solution. Repeat procedure with remaining standards, proceeding from lowest to highest concentration. Wait until the reading has stablized (at least 2 to 3 min) before recording millivolts for standards and samples containing ≤ 1 mg $NH_3$-N/L.
3. *Preparation of standard curve:* Using semilogarithmic graph paper, plot ammonia concentration in milligrams $NH_3$-N per liter on the log axis vs. potential in millivolts on thelinear axis starting with the lowest concentration at the bottom of the scale. If the electrode is functioning properly a tenfold change of $NH_3$-N concentration produces a potential change of about 59 mV.
4. *Measurement of samples:* Dilute if necessary to bring $NH_3$-N concentration to within calibration curve range. Place 100 mL sample in 150-mL beaker and follow procedure in 4*b*above. Record volume of 10*N* NaOH added. Read $NH_3$-N concentration from standard curve.

**References**

1. BANWART, W.L., J.M. BREMNER & M.A. TABATABAI. 1972. Determination of ammonium in soil extracts and water samples by an ammonia electrode. *Comm. Soil Sci. Plant Anal*. 3:449.
2. MIDGLEY, C. & K. TERRANCE. 1972. The determination of ammonia in condensed steam and boiler feed-water with a potentiometric ammonia probe. *Analyst* 97:626.
3. BOOTH, R.L. & R.F. THOMAS. 1973. Selective electrode determination of ammonia in water and wastes. *Environ. Sci. Technol.* 7:523.
4. U.S. ENVIRONMENTAL PROTECTION AGENCY. 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, National Environmental Research Center, Cincinnati, Ohio. AMERICAN SOCIETY FOR TESTING AND MATERIALS. 1979. Method 1426–79. American Soc Testing & Materials, Philadelphia, Pa.
5. Method 4500-NH3 D, E. Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998. APHA, AWWA, & WEF, Washington, D.C. www.standardmethods.org

# SOP 8
# Microwave Acid Digestion

## Scope

This procedure is used to prepare ICP-AES metal analysis sample generated during the different experiments (EPA SW846-3051A).

## Summary of Method

A representative sample of up to 0.1 g is digested in 12 ml of mixed concentrated HCl-$HNO_3$ for 30 min (for solid samples) using microwave heating with a suitable laboratory microwave unit. The sample and acid are placed in a fluorocarbon (PFA or TFM) microwave vessel. The vessel is capped and heated in the microwave unit. After cooling, the vessel contents are filtered, centrifuged, or allowed to settle and then diluted to volume and analyzed by the appropriate SW-846 method. .

## Apparatus

Microwave Accelerated Reaction System (MARS) with Teflon holders

Fluorocarbon (PFA or TFM) digestion vessels (120 mL capacity) capable of withstanding pressures up to 7.5 ± 0.7 atm (110 ± 10 psig) and capable of controlled pressure relief at pressures exceeding 7.5 ± 0.7 atm (110 ± 10 psig).

## Reagents and Materials

Conc. $HNO_3$

Conc. HCl

Deionized (DI) water

Pipetter, 10 ml capacity

## Interferences

Very reactive or volatile materials that may create high pressures when heated may cause venting of the vessels with potential loss of sample and analytes. The complete decomposition of either carbonates, or carbon based samples, may cause enough pressure to vent the vessel if the sample size is greater than 0.25 g when used in the 120 mL vessels with a pressure relief device that has an upper limit of 7.5 ± 0.7 atm (110 ± 10 psi). This is required to retain the heat characteristics of the

calibration procedure. Limits of quantitation will change with sample quantity (dilution) as with instrumentation."

**Sample Collection, Preservation, and Handling**

All sample containers must be prewashed with detergents, acids, and water. Plastic containers are preferable. See SOP 10, Step 3.1.3 of this manual, for further information. Aqueous waste waters must be acidified to a pH of < 2 with $HNO_3$.

**Procedure**

1. Weigh a well-mixed sample to the nearest 0.001 g into the fluorocarbon sample vessel equipped with a single-ported cap and a pressure relief valve. For SAP sample, no more than 0.10 g sample will be used. This sample is added into the digestion vessel with the number of the vessel recorded on the preparation sheet.

2. A reference standard and a master control without sample are treated in the same manner along with spikes and duplicates.

3. Add 3 ± 0.1 mL concentrated HCl firstly, then 9± 0.1 mL concentrated $HNO_3$ to the vessel in a fume hood. Check to make sure the pressure relief disks are in the caps with the smooth side toward the sample and start the caps a few turns on the vessels.

4. Evenly distributed the vessels in the carousel according to the manufacturer's recommended specifications. Blanks are treated as samples for the purpose of balancing the power input. When fewer than the recommended numbers of samples are digested, the remaining vessels should be filled with 0.1 g of reference standards and12 mL of mixed HCl-$HNO_3$ to achieve the full complement of vessels.

5. Program the microwave unit according to the manufacturer's recommended specifications and, if used, connect the pressure vessels to the central overflow vessel with PFA-fluorocarbon tubes. The chosen sequence will bring the samples to 185 deg C ± 5 deg C in 30 minutes and will hold at 185 deg C during the second 30 minutes. Start the turntable motor and be sure the vent fan is running on high and the turntable is turning. Start the microwave generator.

6. At the end of the microwave program, allow the vessels to cool for at least 5 minutes in the unit before removal to avoid possible injury if a vessel vents immediately after microwave heating. The samples may be cooled outside the unit by removing the carousel and allowing the samples to cool on the bench or in a water bath. When the vessels have cooled to room temperature, weigh and record the weight of each vessel assembly. If the weight of the sample plus acid has decreased by more than 10% discard the sample.

7. Complete the preparation of the sample by carefully uncapping and venting each vessel in a fume hood. Transfer the sample to an acid-cleaned bottle. If the digested sample contains

particulates which may clog nebulizers or interfere with injection of the sample into the instrument, the sample may be filtered.

8. Filtering: The filtering apparatus must be thoroughly cleaned and pre-rinsed with dilute (approximately 10% V/V) nitric acid. Filter the sample through qualitative filter paper into a second acid-cleaned container.

9. The concentration values obtained from analysis must be corrected for the dilution factor from the acid addition. If the sample will be analyzed by ICP-MS additional dilution will generally be necessary. The dilutions used should be recorded and the measured concentrations adjusted accordingly.

**Quality Control**

1. For each analytical batch of samples processed, analytical reagent blanks (also field blanks if they were taken) should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

2. Duplicate samples should be processed on a routine basis. A duplicate sample is a real sample brought through the whole sample preparation and analytical process. A duplicate sample should be processed with each analytical batch or every 20 samples, whichever is the greater number.

3. Spiked samples or standard reference materials should be employed to determine accuracy. A spiked sample should be included with each group of samples processed and whenever a new sample matrix is being analyzed.

**SOP 9**

# ICP-AES Total Metals Measurement

**Refer SOP:** LRPCD SOP 08.0
NRMRL LRPCD_M 08_0 ICP-OES Analytical Procedure for IRIS Intrepid.pdf

**SOP 10**
**SOP for Ion Chromatography (IC) Anions Measurement**

**Refer SOP:** LRPCD SOP 07.0
NRMRL LRPCD_M 07_0 Ion Chromatographic Analysis of Inorganic Anions in Aqueous Samplesx.pdf

# SOP 11
# SOP for X-ray Diffraction Spectroscopy Measurements

## 1.0 Scope and Application

X-ray Diffraction (XRD) Spectroscopy is used to determine the crystalline structure of non-amorphous materials.

## 2.0 Summary of Method

The three-dimensional structure of nonamorphous materials, such as minerals, is defined by regular, repeating planes of atoms that form a crystal lattice. When a focused X-ray beam interacts with these planes of atoms, part of the beam is transmitted, part is absorbed by the sample, part is refracted and scattered, and part is diffracted. Diffraction of an X-ray beam by a crystalline solid is analogous to diffraction of light by droplets of water, producing the familiar rainbow. X-rays are diffracted by each mineral differently, depending on what atoms make up the crystal lattice and how these atoms are arranged.

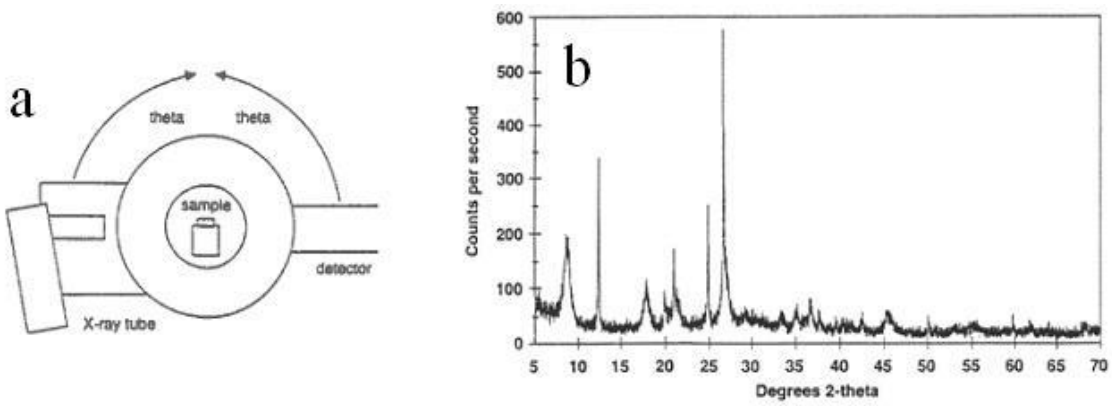


**Figure X2.** (a) Simplified configuration sketch of the X-ray source (X-ray tube), the X-ray detector, and the sample during an X-ray scan. In this configuration, the X-ray tube and the detector both move through the angel theta (θ), and the sample remains stationary. (b) Example of an X-ray powder diffractogram produced during an X-ray scan. The peaks represent positions where the X-ray beam has been diffracted by the crystal lattice. The set of *d*-spacings (the distance between adjacent planes of atoms), which represent the unique "fingerprint" of the mineral, can easily be calculated by the 2-theta (2θ) values shown. The use of degrees 2-theta in depicting X-ray powder diffraction scans is a matter of convention, and can easily be related back to the geometry of the instrument, shown in Figure 5a. The angle and the *d*-spacings are related to Bragg's Law, as described in the text.

## 3.0 Definitions

*X-ray*: A form of electromagnetic radiation with a wavelength in the range of 0.01 to 10 nanometers. X-rays are primarily used for diagnostic radiography and crystallography.

## 4.0 Sample Handling and Preservation

Samples will be collected in glass or plastic vials. Analysis of the collected sample will occur within 48 hour of collection. No preservation techniques will be required after collection and prior to analysis. Samples will be disposed of after analysis negating the need for further preservation.

## 5.0 Apparatus

Philips Instruments X'Pert® MPD System X-ray Diffraction Spectrometer

## 6.0 Procedure for Crystal Structure Determination

In X-ray powder diffractometry, X-rays are generated within a sealed tube that is under vacuum as in the Phillips X'Pert X-ray diffractometer employed in these studies. A current is applied that heats a filament within the tube; the higher the current the greater the number of electrons emitted from the filament. This generation of electrons is analogous to the production of electrons in a television picture tube. A high voltage, typically 15-60 kilovolts, is applied within the tube. This high voltage accelerates the electrons, which then hit a target, commonly made of copper. When these electrons hit the target, X-rays are produced. The wavelength of these X-rays is characteristic of that target. These X-rays are collimated and directed onto the sample, which has been ground to a fine powder or deposited as a wet paste onto filter paper. A detector detects the X-ray signal; the signal is then processed either by a microprocessor or electronically, converting the signal to a count rate. Changing the angle between the X-ray source, the sample, and the detector at a controlled rate between preset limits is an X-ray scan (Figures X2a and X2b).

When an X-ray beam hits a sample and is diffracted, we can measure the distances between the planes of the atoms that constitute the sample by applying Bragg's Law.

Bragg's Law is: $n = 2d\sin$

where the integer $n$ is the order of the diffracted beam, is the wavelength of the incident X-ray beam, d is the distance between adjacent planes of atoms (the d-spacings), and is the angle of incidence of the X-ray beam. Since we know $\lambda$ and we can measure $\Theta$, we can calculate the d-spacings. The geometry of an XRD unit is designed to accommodate this measurement (Figure 5b). The characteristic set of d-spacings generated in a typical X-ray scan provides a unique diffraction pattern for specific crystal structures. When properly interpreted, by comparison with standard reference patterns and measurements, this "fingerprint" allows for identification of the crystal structure and mineral phases present.

## 7.0 Method

1. Mount sample in XRD sample cell and load in it into the instrument
2. Open the instrument software control package on the adjacent computer (X'Pert)
3. Select data acquisition
4. Input the parameters for the current measurement.
    a. For the current project samples will be analyzed from 5 to 110 degrees 2 Θ
    b. Data points will be collected every 0.1 degrees for 2 seconds
5. The resulting scan will be compared to a reference diffraction pattern data base provided by Philips Instruments

## 8.0 Quality Control

- Prior to analyzing any samples the instrument will be calibrated using a reference material (Si 111 crystal) to ensure the angle measurements are correct.

## References

Philips Instruments X'Pert® MPD System X-ray Diffraction Spectrometer Users Manual

Kittel, C. Introduction to Solid State Physics, Seventh Edition. J Wiley & Sons, Inc. New York. NY. 1996.

# SOP 12
# Laboratory Glassware Cleaning

**Scope**
This procedure is used to clean laboratory glassware for use in general laboratory activities that include: acid digestions, bench-scale experiments, etc. Additional cleaning steps might be needed for some particularly contaminated glassware or analytical procedures.

**Summary**
All the laboratory equipment and glassware that come in contact with the soil, extraction fluid or leachate must be rinsed off and soaked in soap water for two hours, then soaked in 5% nitric acid followed by rinsing with deionized (DI) water to remove any residual deposits. This equipment includes supplies, utensils and containers or any surface that will come into direct contact with the above materials.

**Reagents and Materials**

Cleaning Brush – Soft, non-damaging brush (e.g. Fisher Scientific or equivalent)
Detergent – viz: Sparkleen, Fisher Scientific or equivalent.
Reagent Grade Water – Deionized (DI) water with a resistivity of 18.2 mohm can be provided by commercially available deionization systems (e.g., Milli-Q Plus, Millipore, or equivalent grade).
5% Nitric Acid – Made by dilution of ACS grade nitric acid (e.g. Fisher Scientific or equivalent grade) with DI water.

**Procedure**

1. Rinse loose debris from the surface of the object using tap water.
2. Wash the object thoroughly using a brush, soap and water. Triple rinse with tap water.
3. Soak the object in a soap bath for two hours.
4. Triple rinse with tap water.
5. Soak the object in a 5% nitric acid bath overnight
6. Remove the object from the water bath and triple rinse all surfaces with DI water.
7. Dry the object using drying racks. Take care to limit the exposure of the objects to air-borne particulates or any source of contamination.
8. Objects that are not of immediate use should be stored in an area where the exposure to air-borne particulates is a minimum.

**Acid Bath Neutralization**

The acid bath needs to be periodically prepared in order to keep it clean. To assure the safe and environmental disposal of the 5% nitric acid solution, it will be neutralized to obtain a pH in the range of 6-9. The neutralization will be done with sodium carbonate and a pH-meter or pH-paper will be used to verify the final pH.

# SOP 13

## SOP for TEMP/W Modeling of Thermal Changes

### 1.0 Scope and Application

TEMP/W will be used to simulate thermal changes in MSW after disposal of SAP waste.

### 2.0 Background

TEMP/W is a finite element CAD software product for analyzing thermal changes in the ground due to environmental changes or due to the construction of facilities such as buildings or pipelines. The comprehensive formulation makes it possible to analyze both simple and highly complex geothermal problems. TEMP/W can be applied to the geothermal analysis and design of geotechnical, civil, mining and solid waste engineering projects, including facilities subjected to freezing and thawing temperature changes.

#### *a. Hardware Requirements*

To run the program the following hardware requirements are necessary:

- Intel Pentium processor or higher
- 16 Mb RAM
- Hard disk with at least 20 Mb free disk space
- VGA graphics (High Color recommended)
- MS Windows 95, 98, NT, 2000, XP, Vista (32/64 bit) or Window 7 (32/64 bit)

## 3.0 Procedure and Parameters

*For this work, the following parameters should be placed in the correct fields within the TEMP/W program:*

*Geometry Information:*

- *Landfill cell shape*

*Materials Information:*

- *Thermal function*
- *Unfrozen and frozen water content*
- *Thermal conductivity*
- *Volumetric heat capacity*

*Boundary Conditions Information:*

- *Specify the boundary conditions that describe the various landfilling scenarios of SAP waste*
- *Assign boundary conditions to the appropriate regions of the landfill cell*

### *Running TEMP/W*

- *Select the analysis type (steady state or transient)*
- *Select the run duration and the number of runs*
- *From the Tools Menu select Verify/Optimize and then select Solve Analyses*

### *Results*

- *View the results through Contours from the Draw Menu and Graph from the Draw Menu*
- *View results as needed*

# SOP 14

## Test Method for Toxicity Characteristics Leaching Procedure

### Scope

The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes. This is a standardized test that determines the potential mobility of contaminants in a liquid or solid under simulated landfill conditions. The TCLP uses an acidic leachant (acetic acid) to depict a 'worst case scenario' of a hazardous waste co-disposed into a municipal solid waste landfill.

### Summary of Test Method

The TCLP procedure can be found in SW 846, Method 1311. In order to perform the TCLP analysis, an initial pH measurement of the sample must be made in order to determine the appropriate extraction fluid. Sample (< 9.5 mm) will be used in this test. The type of extraction fluid (pH=4.93 or pH=2.88) is decided based on the characteristics of the sample. The sample will be mixed with the extraction fluid at a liquid-solid ratio (L/S) of 20, rotated in a tumbler at 30±2 rpm for 18±2 hours. The leachate will be filtered, acid digested and analyzed for the metal content.

### Reagents

1. "Unless otherwise noted, it is assumed that ACS reagent grade chemicals or their equivalent are used. Also, unless otherwise noted, references to water are understood to mean reagent grade water as described in 05.SOP.011.00, Reagent Water Quality and Monitoring."
2. D.I. water (ASTM Type 2)
3. Hydrochloric acid (1N), HCl
4. Nitric acid (1N), $HNO_3$
5. Sodium hydroxide (1N), NaOH
6. Glacial acetic acid, $CH_3CH_2OOH$

### Equipment

1. Analytical Balance
2. Bottle Extraction Vessel, 2000 mL HDPE bottles
3. pH meter and buffers
4. Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.
5. Filter Holder
6. Borosilicate glass fiber filter.
7. Extraction Fluid Transfer Devices such as positive displacement unit.
8. Beaker or Erlenmeyer flask, glass, 500 mL.
9. Magnetic stirrer

### Procedure

1.0 Particle size reduction

1.1 Particle size reduction to less than 9.5 mm will be performed as follows:

- The samples will be placed in a stainless steel pan and crushed to pass a 9-mm sieve.
- After thorough mixing, approximately 0.5 kg of the 9-mm sieved materials will be further size reduced down to a size of 2 mm using a grinding machine (Preiser Scientific).
- The 2 mm size samples will be used for the TCLP test.

2.0 Extraction fluid preparation

2.1 Extraction Fluid # 1: Add 5.7 mL glacial $CH_3CH_2OOH$ to 500 mL of reagent water, add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

2.2 Extraction Fluid # 2: Add 5.7 mL glacial $CH_3CH_2OOH$ to 1L of reagent water. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

3.0 Preliminary TCLP preparation

3.1 pH measurement

3.1.1 Calibrate pH meter according to SOP 3.

3.1.2 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

3.1.3 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1.

3.1.4 If the pH is > 5.0, add 3.5 ml N HCl, slurry briefly, cover with a watchglass, heat to 50ºC, and hold at 50ºC for 10 minutes.

3.1.5. Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

4.0 TCLP test

4.1 Weigh out 10 g of the sample in a 500 mL plastic bottle.

54.2 Add 200 mL of the extraction fluid to the sample bottle.

4.3 Rotate the bottle rotary agitator at 30 ± 2 rpm for 18 ± 2 hours.

4.4 Following the agitation, filter the extract using Whatman 0.45 µm PVDF filters..

4.5 Following filtration, acidify the sample to pH less than < 2 using concentrated nitric acid and store at 4°C.

4.6 Analyze sample for total metals after acid digestion following method 3015A.

5.0 Precision and Controls

5.1 A duplicate sample will be prepared and processed every 15 samples.

5.2 Controls are provided by running the bottles with the extraction fluid but no sample in them.

**Expression of Results**

Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations to determine if sample is a hazardous waste or not.

**Quality Control**

The procedures given in EPA Method 1311 will be followed.

1. A minimum of one blank (using the same extraction fluid as used for the samples) must be digested and analyzed for every 15 samples.
2. A sample duplicate will be conducted every 15 samples.
3. A standard check will be analyzed every 10 samples for the ICP analysis.
4. All analyses that fall outside the range for precision and accuracy will be repeated.