Aqueous and Solid Phase Characterization of Potential Tank Fill Materials

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EXECUTIVE SUMMARY

SREL conducted a series of batch and column studies to address uncertainty in the realistic pH and E_h ranges associated with the grouted waste tank systems, including an evaluation of three candidate Tank Closure Grout (TCG) formulations. The paste component formulations for the three TCG test materials included conventional reducing TCG made with Ordinary Portland Cement (OPC; 18%), blast furnace slag (BFS; 30%) and fly ash (FA; 52%); TCG with no BFS (TCG-NBFS) made with OPC (18%), and FA (82%); and a Consolidated Low Strength Material (CLSM) made with OPC (7.7 %), and FA (92.3 %). The three paste materials were cured for 90 days, size-reduced, and for column testing mixed with quartz sand to requisite grout proportions. The granular paste (batch) or paste + sand equivalents of cast grout (column) were then subjected to different atmospheric conditions, including open to the laboratory atmosphere (i.e., oxidizing), under a constant Ultra-High Purity (UHP) N_2 purge, and within an anaerobic Coy Chamber with anoxic conditions maintained by addition of a 95% N_2/5% H_2 gas mixture to establish a consistent 2% H_2 atmosphere. Batch tests consisted of size-reduced grout materials equilibrated with pore water simulant for 150+ days, with pH and E_h monitored weekly and small aliquots of the pore waters collected for chemical analysis. Column tests were also performed in which the size-reduced TCG formulations were constantly leached under saturated conditions with the pore-water simulant that was either in equilibrium with the lab atmosphere or purged with UHP N_2 to reduce dissolved O_2 levels.

The observed results generally agreed with previous laboratory tests aimed at defining achievable E_h and pH conditions in tank waste grouted systems (SRNL-STI-2015-00446; SRNL-STI-2016-00432; SRNL-STI-2018-00484). The pH results were predominantly consistent with both the values derived from geochemical modeling and more recent laboratory testing. The TCG displayed the highest pH followed by the TCG-NBFS and finally the CLSM, with both the TCG and TCG-NBFS appearing to better maintain a higher pH (i.e., buffer the system) than the CLSM under all three test atmospheres. The observed E_h values, however, were less extreme than values used in waste release models (WRMs) to represent various stages in the aging of reducing tank1 closure grout. The lowest E_h values were observed for all samples equilibrated in the anaerobic Coy Chamber under a H_2/N_2 atmosphere, with the N_2 glovebag yielding the next lowest values and the batch samples open to the lab atmosphere yielding the highest E_h values. Even though there was a great deal of scatter in the data, the TCG materials containing the BFS generally provided the lowest E_h values (i.e., most reducing) under all batch and column test conditions, followed by TCG-NBFS and then CLSM.

The original dry feed materials, the three initially cured TCG formulations, and the TCG materials that were subjected to various batch treatments were extensively characterized by XRF and XRD analysis. Initial XRF analysis evaluated the two most common sample preparation methods, i.e., pressed pellets and borate fused beads. Although results were generally consistent for both preparation methods, analysis of pressed pellets overestimated the level of CaO in FA materials while underestimating the SiO_2 content. Thus, all grout and dry feed materials were analyzed as fused beads. Minor changes to Na_2O and K_2O contents were observed for batch samples that had been subjected to

extended leaching, but such changes were insufficient to correlate with any significant changes to the mineralogy of the batch tested materials.

X-ray diffraction patterns of both pre-leached and leached samples were dominated by amorphous phases presumed to be associated with unreacted silicate glasses in the BFS and FA components, and amorphous calcium silicate hydrate (C-S-H) and calcium aluminosilicate hydrate (C-A-S-H) gels formed via the hydration/pozzolanic reactions of the OPC, BFS, and FA. As anticipated, the poorly reacting crystalline phases of FA (i.e., quartz, mullite, hematite, and magnetite) were also detected in all pre-leached and leached samples. In addition to the aforementioned phase constituents, all pre-leached samples indicated the presence of strätlingite, calcite, ettringite, and varied (alumina-ferric oxide-mono) AFm carbonates and/or sulfates. Ettringite was not detected in any leached samples, and the AFm phases either typically disappeared in the leached samples or their proportions were significantly reduced. Strätlingite persisted to some degree in almost all of the leached samples irrespective of the leaching environment; however, it was barely detectable (and therefore not quantifiable) for the CLSM, TCG-NBFS, or TCG subjected to a reducing environment. In addition, for all TCG samples (containing BFS) strätlingite was barely apparent above background irrespective of the leaching environment. Calcite persisted in all samples and the highest calcite proportions were detected for samples subjected to the oxic environment during leaching. These results were anticipated and presumed due to sample carbonation in the CO_2-containing oxic environment. Hydrotalcite and a phase tentatively identified as kuzelite (or monosulfoaluminate) were also observed but only in the BFS-containing TCG samples; both minerals persisted in all the leached TCG samples irrespective of leaching environment. Hydrotalcite was anticipated due to the magnesia (MgO) content of the BFS (approximately 6 wt% measured via XRF). Monosulfoaluminate is a reaction product of tricalcium aluminate (a primary cement phase) and ettringite (a cement hydration product).

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List of Acronyms and Abbreviations

ACI	American Concrete Institute
AFm	alumina-ferric oxide-mono
AFt	alumina-ferric oxide-tri
ASTM	American Society for Testing and Materials
BFS	Blast Furnace Slag
C-A-S-H	Calcium Aluminosilicate Hydrate
CLSM	Controlled Low-Strength Material
CRMs	Certified Reference Materials
C-S-H	Calcium Silicate Hydrate
CTF	Cognizant Technical Function
DIW	Deionized Water
DOE	Department of Energy
EPA	Environmental Protection Agency
FA	Fly Ash
FTF	F-Area Tank Farm
FY	Fiscal Year
GOF	Goodness of Fit
HTF	H-Area Tank Farm
ICDD	International Centre for Diffraction Data
ICP-MS	Inductively Coupled Plasma-Mass Spectrometer
OPC	Ordinary Portland Cement
ORP	Oxidation Reduction Potential
MCR	Manufacturer Certification Report
NIST	National Institute of Standards & Technology
PA	Performance Assessment
PDF	Powder Diffraction File
QA	Quality Assurance
QC	Quality Control
REF	Reference Sample
SD	Standard Deviation
SREL	Savannah River Ecology Laboratory
SRM	Standard Reference Material
SRR	Savannah River Remediation LLC
SRS	Savannah River Site
TCG	Conventional Tank Closure Grout Formulation containing BFS
TCG-NBFS	Tank Closure Grout without Blast Furnace Slag
UHP	Ultra-High Purity
USEPA	United States Environmental Protection Agency
w/cm	Water-to-Cementitious Material Ratio
WRM	Waste Release Model
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

INTRODUCTION

The Performance Assessments (PA) for the Savannah River Site (SRS) F-Area Tank Farm (FTF) and H-Area Tank Farm (HTF) assume that solubility controls the behavior of several key radionuclides, including neptunium (Np), plutonium (Pu), uranium (U) and technetium (Tc). Solubility limits for these radionuclides are controlled by the pH and E_h conditions attributed to the interaction of grouted tank waste with soil moisture and groundwater. Infiltration passes through the tank materials and drives changes in the composition and mineralogy of the grout materials. Over time, the pH, E_h and pore solution composition will vary as the tank grout ages and degrades, with the system becoming more oxidized and the pH decreasing. This aging process has been described as a series of degradation stages that can be related to the number of pore volumes in contact with the tank waste materials, which can then be converted to reaction time within a flow and transport model (Denham and Millings, SRNL-STI-2012-00404).

Denham et al. (SRNL-STI-2012-00404) developed a sequential batch reaction model describing radionuclide release from residual tank waste based on component solubility as controlled by tank grout degradation in contact with water that has passed through the closure cap. Infiltration passes through the tanks and drives changes in grout mineralogy/composition, with the emerging fluid reflecting such interactions that vary over time. Three stages of grout degradation summarized in **Table 1** can be defined by the amount of infiltrating water that contacts the grout materials (i.e., pore volumes), which can then be converted to time. However, major uncertainties to this approach include defining the initial infiltrate composition and the mineralogy/composition of the Tank Closure Grout (TCG) materials.

Table 1. Tank waste porewater conditions reflecting three stages of reducing TCG aging (Denham and Millings, SRNL-STI-2012-00404).

Parameter	Red. Region II	Ox. Region II	Ox. Region III
pH	11.1	11.1	9.2
E_h (volts)	-0.47	0.56	0.68
Ca^{2+} (molar)	4.0E-03	4.0E-03	6.6E-05
Na^{+} (molar)	1.0E-03	1.0E-03	1.0E-03

More recently, a series of laboratory tests were conducted to determine the solubility values of Pu, Np, U, and Tc under various grouted waste tank conditions (King and Hobbs, SRNL-STI-2015-00446; King and Hobbs, SRNL-STI-2016-00432; Layton, SRR-CWDA-2016-00086; King, SRNL-STI-2018-00484). These tests have focused on measuring contaminant solubility under pH and E_h conditions that reflect the three stages of reducing grout degradation (i.e., RRII, ORII, and ORIII). These tests have used residual tank waste samples and waste simulants in contact with grout-representative phases to evaluate contaminant solubility. The laboratory tests have generally been successful in achieving the target pH conditions associated with the three degradation stages; however, it has been far more difficult to achieve the target E_h values using standard grout materials. For

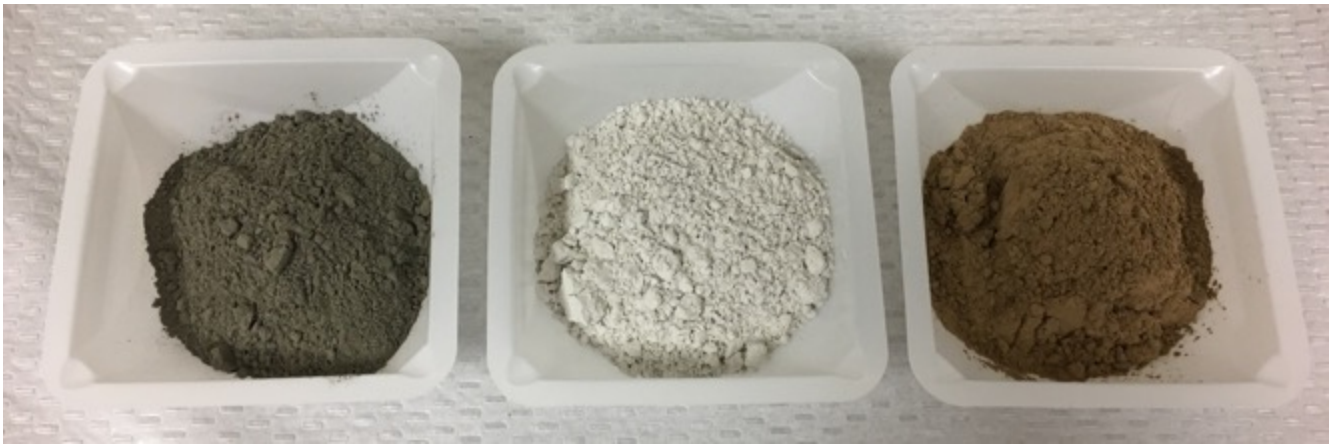
instance, E_h values for the RRII chemical conditions with representative grout solids have been considerably less negative than the target value (-0.47 V), with the addition of ferrous sulfide (FeS) used to produce more reducing conditions. Such discrepancies in E_h can impact contaminant solubility. While Np was more insoluble than the Waste Release Model (WRM) value, the observed solubilities of Pu, Tc, and U were actually greater because of the more oxidizing conditions. Even so, the higher solubilities were shown to have a negligible impact on peak doses in 1,000 or 10,000 years (Layton, SRR-CWDA-2016-00086). In general, the highest and lowest E_h values observed in previous testing were approximately +0.5 V and -0.2 V, respectively, even with the addition of non-representative oxidants and reductants (King and Hobbs, SRNL-STI-2015-00446; King and Hobbs, SRNL-STI-2016-00432, Layton, SRR-CWDA-2016-00086; King, SRNL-STI-2018-00484). This uncertainty with respect to E_h condition remains a key question concerning radionuclide solubility based on inspection of E_h-pH solubility diagrams contained in SRNL-STI-2012-00404.

In an effort to address the uncertainties related to the chemical conditions associated with the aging of grouted tank waste the University of Georgia's Savannah River Ecology Laboratory (SREL) conducted a series of experimental tests evaluating the dynamic interactions of pore-water solutions in contact with TCG. Experiments focused on monitoring changes in aqueous chemistry (i.e., E_h, pH, DO, alkalinity, solution components, etc.) that occur in grouted tank closure systems with extended leaching. Dynamic column experiments and extended batch equilibrations evaluated the interactions of three TCG formulations under simulated leaching conditions designed to predict potential leachate chemistries that will impact the residual waste layers in various SRS waste tanks. In addition to monitoring eluate chemistry, changes in the properties and solid phase composition of TCG materials were extensively characterized by x-ray diffraction (XRD) spectroscopy and x-ray fluorescence (XRF) spectroscopy.

MATERIALS AND METHODS

Three TCG formulations were created for testing using the following dry-feed materials (**Figure 1**): (1) Type I/II Ordinary Portland Cement (OPC; Holcim US, Inc. Birmingham, AL 35221), (2) Class F fly ash (FA; The SEFA Group, Inc. Lexington, SC 29073), and (3) Grade 100/120 blast furnace slag (BFS; Lehigh Cement, Cape Canaveral, FL 32920). The manufacturer certification reports for the dry feeds used in this study are provided in **Appendix A**. An image of the three dry feed materials is provided in **Figure 1**. The final test paste formulations are outlined in **Table 2**, consisting of three dry feed mixtures: conventional TCG made with OPC (18%), BFS (30%) and FA (52%); TCG without BFS (TCG-NBFS) made with OPC (18%), and FA (82%); and a Consolidated Low Strength Material (CLSM) made with OPC (7.7 %), and FA (92.3 %).

Figure 1. Photograph of the three dry feed materials used to make the TCG formulations outlined in Table 2 (L to R: Type I/II Ordinary Portland Cement, Lehigh Grade 100/120 Blast Furnace Slag, and SEFA Class F Fly Ash).



The TCG paste materials were prepared and cured for the required duration following the applicable requirements of ASTM C192, *Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory.* The water to cementitious materials ratio for the first two TCG formulations was 0.579 (w/cm), i.e., 579 gm of water (579 mL) added to 1 kg of mixed dry cement, slag, and fly ash. The w/cm ratio for the CLSM was 0.847. The paste materials were poured into labeled 2" ID x 4" plastic concrete cylinder molds for curing (Test Mark Industries, Inc.). A limited set of monoliths were also created using transparent 50 mL polypropylene containers. Laboratory tap water was used in the production of the TCG formulations. Two separate batches of each paste formulation were created. One batch was wrapped in moist towels, sealed in plastic bags and then cured in a sealed cooler in the laboratory. This batch was used for all "open" system atmosphere tests. The second batch of curing cylinders was wrapped in moist towels, sealed in Lock & Lock USA, Inc. (Cerritos CA 90703) plastic containers and cured in an anoxic Coy Chamber (95% N_2/5% H_2 atmosphere). This batch was used for the "closed" system under a reducing atmosphere.

After 90 days, selected monoliths representing each formulation and atmosphere treatment were sliced open to produce the required "size reduced" (granular) paste materials for batch and column testing. **Figure 2** provides an example of the three test formulations after curing in the transparent cylinders, with the TCG formulation displaying the darker color generally attributed to the presence of BFS (Chaouche et al., 2017), and the TCG-NBFS and CLSM materials having the browner color. The greater amount of bleed water is also clearly apparent for the CLSM materials.

Table 2. Tank Fill Grout Paste Formulations: A. Provided in SRR-CWDA-2019-00038; B. Masses required for each kg of dry feed materials.

A.

Materials	Tank Fill Grout Paste Dry Ingredients			Sand	Water
	Cement Type I/II	Slag Grade 100/120	Fly Ash Class F		
	lbs/yd^3	lbs/yd^3	lbs/yd^3	lbs/yd^3	gal/yd^3
TCG Paste	125	210	363	2590	48.5
TCG Paste w/o Slag (TCG-NBFS)	125	N/A	573	2590	≤ 48.5
CLSM	50	N/A	600	2515	66.00

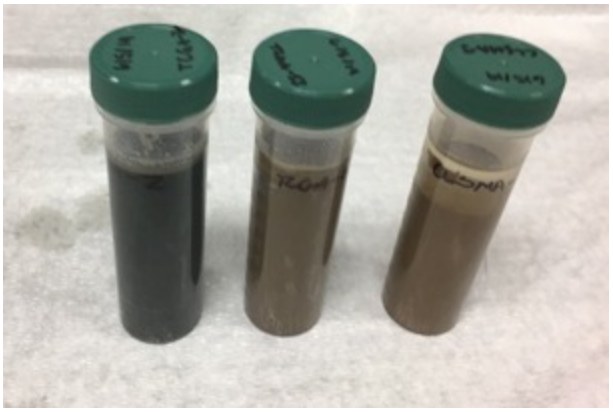
B.

Materials	Tank Fill Grout Paste Dry Ingredients			Sand*	Water**
	Cement Type I/II	Slag Grade 100/120	Fly Ash Class F		
	gm/kg	gm/kg	gm/kg	gm/kg grout	w/m
TCG Paste	180	300	520	3,711	0.579
TCG Paste w/o Slag (TCG-NBFS)	180	N/A	820	3,711	0.579
CLSM	77	N/A	923	3,869	0.847

*gm of sand required for each kg of TCG paste

**water to dry feed material ratio

Figure 2. Examples of the three TCG past formulations cured for 90 days in clear transparent containers: TCG, TCG-NBFS, and CLSM.

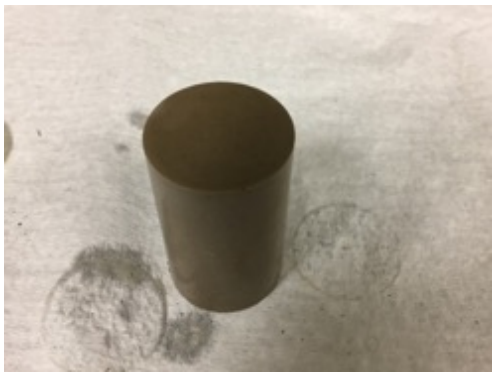


The size reduction process is illustrated in **Figure 3**. The "open" system materials cured in the lab cooler were size-reduced in the lab while the "closed" system materials were size reduced inside the Coy Chamber to reduce exposure to O_2. After removal from the cylinder (Fig. 3A), the monoliths were placed in 6 mil, clear polyethylene plastic bags and broken up by hand using a rock hammer. The resulting materials were then screened through a 2 mm sieve. An example of the TCG formulation

displaying the darker color associated with BFS is provided in **Figure 3B**. There was no observed difference in appearance between the TCG materials cured in the lab and the TCG materials cured in the Coy Chamber. All materials retained on the 2 mm sieve were then placed back in the plastic bags (**Fig. 3C**), and repeatedly crushed by hand until the entire sample passed through the sieve. This was done to avoid any selective sampling of the cured materials based on hardness. An example of the TCG formulation without BFS is provided in **Figure 3D** displaying the brown color. The resulting < 2 mm fraction was further crushed to yield a smaller, < 0.5 mm fraction. Initially efforts to create a smaller < 0.15 mm fraction were deemed impractical, especially when braking up materials in the Coy Chamber. Even so, this procedure generated two size fractions for testing, i.e., (1) the "size-reduced" 0.5 to 2 mm fraction and (2) the "crushed" < 0.5 mm fraction. The two size fractions were then sealed in plastic containers and stored under an inert atmosphere until their use in batch or column experiments. The sand was only added to the size-reduced materials when the dynamic columns were being packed.

Figure 3. Size Reduction of TCG and TCG-NBFS samples

A. TCG Monolith



B. >2 mm TCG Retained

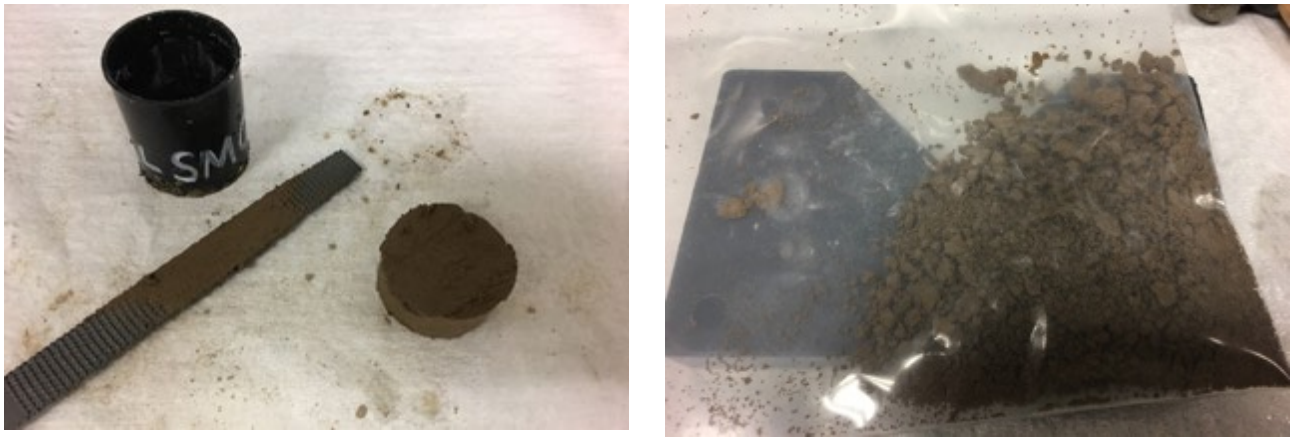
C. Size Reducing TCG-NBFS

D. > 2mm TCG-NBFS Retained

This size reduction procedure generally worked well for the TCG and TCG-NBFS formulations, but not for the CLSM. Even after curing 90 days, the CLSM monoliths were still quite friable and difficult to break up without creating additional clumps that were further retained on the sieve (**Figure 4**). Therefore, only the "size-reduced" 0.5 to 2 mm fraction was used in testing the CLSM.

Figure 4. CLSM monolith removed from curing mold and sticking to rasp during attempts to size reduce for batch and column testing (A). Material clumping in bag during manual size reduction (B).

A. CLSM B. CLSM



Batch Test Methods

The test matrix for batch and column experiments is provided in **Table 3**. Batch testing was initially only planned for the "open" lab atmosphere and the "closed" Coy Chamber atmosphere treatments. The batch experiment was expanded to include a third intermediate atmosphere in which the batch samples were equilibrated in a glove bag under constant UHP N_2 purge to reduce exposure to O_2 without creating the extremely low E_h values observed in the Coy Chamber. Oxygen levels in the chamber were routinely maintained at less than 1%, which correlates to a dissolved O_2 levels of < 0.4 ppm.

Twenty gram samples (20 ± 0.1) of the size-reduced grout paste formulations (both "size reduced" and "crushed") were weighed into labeled 50-mL centrifuge tubes with vented caps for gas exchange (Bio-Reaction Tubes, polypropylene tubes with 0.22 µm hydrophobic membrane in the vent cap; VWR, Radnor, PA 19087-8660). The weight of each test tube was recorded at each step in the experiment to facilitate mass balance calculations and account for moisture losses due to evaporation with extended equilibration times. Examples of the test tubes containing either the size-reduced and crushed materials, and a full treatment set out in the lab are shown in **Figure 5**. Three replicates were used for each batch treatment.

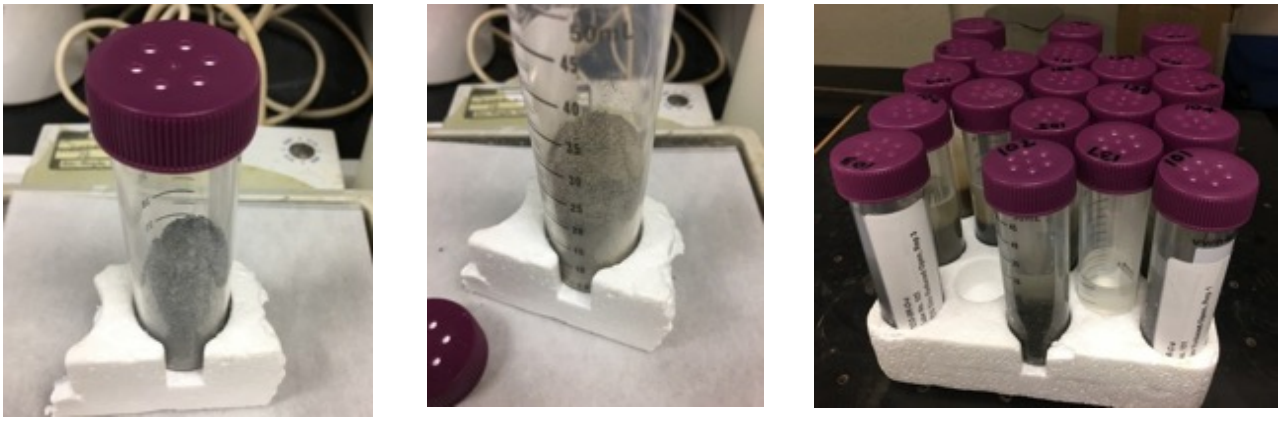
Table 3. TCG treatment configuration for column and batch testing.

Batch Test ID#	Column Test ID#	System	Material	Form
Open Atmosphere Treatments				
O1SR(a,b,c)	O1(a,b)	Open/oxic	TCG paste	Size-Reduced (SR)
O1Cr(a,b,c)		Open/oxic	TCG paste	Crushed (Cr)
	OI1(a)	Open/oxic	TCG with Sand-Intact Monolith	Intact
O2SR(a,b,c)	O2(a,b)	Open/oxic	TCG paste w/o blast furnace slag	Size-Reduced (SR)
O2Cr(a,b,c)		Open/oxic	TCG paste w/o blast furnace slag	Crushed (Cr)
O3SR(a,b,c)	O3(a,b)	Open/oxic	CLSM paste	Size-Reduced (SR)
Closed Atmosphere Coy Chamber				
C1SR(a,b,c)		Closed/anoxic	Tank Closure Grout paste	Size-Reduced (SR)
C1Cr(a,b,c)		Closed/anoxic	Tank Closure Grout paste	Crushed (Cr)
C2SR(a,b,c)		Closed/anoxic	Tank Closure Grout paste w/o	Size-Reduced (SR)
C2Cr(a,b,c)		Closed/anoxic	Tank Closure Grout paste w/o	Crushed (Cr)
C3SR(a,b,c)		Closed/anoxic	CLSM paste	Size-Reduced (SR)
N_2 Anoxic Atmosphere				
N1SR(a,b,c)	N1(a,b)	N_2 Atm	Tank Closure Grout paste	Size-Reduced (SR)
N1Cr(a,b,c)		N_2 Atm	Tank Closure Grout paste	Crushed (Cr)
NI1(a)	NI1(a)	N_2 Atm	TCG with Sand-Intact Monolith	Intact
N2SR(a,b,c)	N2(a,b)	N_2 Atm	Tank Closure Grout paste w/o blast furnace slag	Size-Reduced (SR)
N2Cr(a,b,c)		N_2 Atm	Tank Closure Grout paste w/o blast furnace slag	Crushed (Cr)
N3SR(a,b,c)	N3(a,b)	N_2 Atm	CLSM paste	Size-Reduced (SR)

Notes: “SR” and “Cr” after number in Test ID refers to “size-reduced” and “crushed” samples that are included in batch testing with “a,b,c” in Test ID# denoting three replicates (a, b, and c). For column tests only two SR replicates (a and b) were conducted. For intact testing, only one sample type (TCG with Sand) was exposed to the open air leachate, and one was exposed to the N_2 purged leachate.

Figure 5. Batch equilibration tubes open to the treatment atmosphere.

A. **Size-Reduced TCG** **B. Crushed TCG** **C. Batch Treatments**



A surrogate soil pore-water solution (**Table 4**) based on the predicted composition of SRS rainwater (Strom and Kaback; WSRC-RP-92-450) that has equilibrated in contact with kaolinite and amorphous silica was used as the target test leachate (Denham and Millings; SRNL-STI-2012-00404). **Table 5** provides the recipe to the concentrated TCG leachate stock solution along with the final concentration of the simulant. The stock and final test solutions were made from research grade chemicals using deionized water, 18.2 MΩ cm at 25 °C (Milli-Q Element, Millipore, Inc., Billerica, MA 01821). The pH of the test solution was typically between 6 and 7. There was no attempt to mimic the dissolved levels of Si and Al in the leaching solution due to concerns over the stability of such a solution. The levels of chloride (Cl^-) were higher in the test solution due to charge balance constraints with the major cations. This discrepancy is unlikely to impact batch or column results.

Table 4. Leaching solution prescribed in RFQ No. 190129 (from SRNL-STI-2012-00404).

Parameter	Value	FW (g/*M*)	g/L	mg/L
pH	4.68			
Dissolved Gases	M			
O_2(aq)	2.19E-04	32	7.0E-03	7.01
CO_2(aq)	1.07E-05	44	4.7E-04	0.47
Solutes	*M*			
Cl^-	2.74E-05	35.45	9.7E-04	0.97
Na^+	8.69E-06	22.99	2.0E-04	0.20
Ca^{+2}	2.06E-06	40.08	8.3E-05	0.08
Mg^{+2}	1.34E-06	24.31	3.3E-05	0.03
Al^{3+}	8.43E-07	26.98	2.3E-05	0.02
H_4SiO_4(aq)	1.90E-03	64.1	1.2E-01	121.8
SO_4^{-2}	1.35E-05	96.1	1.3E-03	1.30

Table 5. Stock solution and final dilute background solution for batch equilibrations and column leaching tests.

TCG Stock Solution (1 mL for each L of leachate solution)			
Stock Solution	M	FW	g/L
NaCl	8.69E-03	58.44	0.508
$CaCl_2.2H_2O$	2.06E-03	147.01	0.303
$MgCl_2.6H_2O$	1.34E-03	203.3	0.272
Na_2SO_4	1.35E-02	142.08	1.918
Final Dilute Treatment Solution			
Solutes			mg/L
Cl^-			0.24
Na^+			0.82
Ca^{+2}			0.08
Mg^{+2}			0.03
SO_4^{-2}			1.30

Twenty mL of the dilute test solution were added to each batch tube, and the tubes were all weighed before placement on an orbital shaker at ≈100 rpm. Each day the batch slurries were vigorously shaken by hand to ensure effective mixing. The test leachate was pre-equilibrated in the Coy Chamber for the "closed" system treatments to reduce dissolved O_2 levels prior to its use in batch tests. At least once a week, the tubes were shaken by hand and then allowed to settle. After settling, the pH and oxidation reduction potential (ORP) values were taken in the clear supernatant solution above the slurry. A 1 mL aliquot of the solution was then taken for chemical analysis. The sample was acidified (2% HNO_3) for preservation, and then analyzed for major elements (i.e., Na, K, Ca, Mg, Al, and Fe) by inductively coupled plasma mass spectrometry (ICP-MS) in accordance with the quality assurance (QA) protocols of EPA Method 6020B. One mL of fresh test leachate was then added to each tube to maintain a fixed solid to liquid ratio (L/S = 1). As the batch tests continued, it became obvious that solution was being lost from the samples due to evaporation. To account for this discrepancy, each sample tube was weighed and the observed loss of mass was corrected for by addition of deionized water.

The initial batch experiments were started near the end of September 2019. It soon became obvious that the ORP values observed for the "closed" system (i.e., Coy Chamber) were exceptionally low because of the H_2/N_2 atmosphere used in keeping the system anoxic. To address this issue, a third batch treatment began in January 2020 using the same TCG paste formulation treatments as before. The samples were equilibrated in a glove bag under a constant purge of UHP N_2 controlled by a Neutronics Model 1100 O_2 Analyzer set to maintain an O_2 level less than 1%. The pH and ORP values of the batch treatments were measured on a weekly basis in a manner consistent with the other batch treatments.

After 130 days of equilibration, Replicate A for each batch treatment was removed for analysis of the remaining solid materials. Any supernatant water on the surface of the grout slurry was drained and the samples were then quick frozen with liquid N_2. The residual materials were then freeze dried in preparation for subsequent XRF and XRD analysis. Additional sample preparation details will be discussed below. At the same time that Replicate A was removed, an additional 10 mL of test solution was added to each Replicate C. As before, the remaining batch test replicates B and C were equilibrated continuously on an orbital shaker. Each day all remaining batch samples were vigorously shaken by hand as before, with the pH and ORP measured at least once a week. Each workday, 10 mL of solution was removed from the replicate C samples and then replaced with 10 mL fresh test solution (**Table 5**). As before, the 10 mL sample was acidified (2% HNO_3) for preservation, and then analyzed for major elements (i.e., Na, K, Ca, Mg, Al, and Fe) by ICP-MS. This was done to simulate the removal of soluble grout components due to pore-water exchange in a dynamic system.

The week of March 9th, 2020 the SREL was alerted that UGA would likely close all facilities in response to the Covid-19 pandemic. In preparation for closure, most of the ongoing experiments were terminated the week of March 16th due to limited access to SREL and the SRS. A summary of the duration of each experiment is provided in **Table 6**. All of the column tests were terminated because they required daily maintenance to continue. Batch tests in the "open" lab and the Coy Chamber (i.e., "closed" test) were suspended and the residual treatments (Reps B and C) were quick frozen with liquid N_2 and freeze dried for subsequent XRF and XRD analysis, as done previously with the Replicate A samples. However, the ongoing batch test in the N_2 glovebag was continued because it could be reasonably maintained with limited access to the lab. After closure, essential lab personnel were granted limited access to labs for the purposes of monitoring facilities and equipment, and maintaining critical experiments that could not be suspended. Thus, the batch study under the N_2 atmosphere was allowed to continue for similar duration as the other two experiments.

Table 6. Summary of final batch and column test durations.

Treatment	Size Fraction**	Batch Equilibration Duration (days)			Column Duration (days)	
		A	B	C*	A	B
Open	Intact	-	-	-	68	-
	SR	130	172	130 + 42	130	67
	Cr	130	172	130 + 42		-
N_2	Intact	-	-	-	68	-
	SR	141	141	141	130	67
	Cr	141	141	141	-	-
N_2/H_2	SR	130	172	130 + 42	-	-
	Cr	130	172	130 + 42	-	-

*Batch Rep C for open and N_2/H_2 treatments subject to 42 days of enhanced leaching after first 130 days of equilibration. This consisted of removing 10 mL of solution from the batch sample and replacing it with 10 mL of fresh test solution (Table 5) every work day.

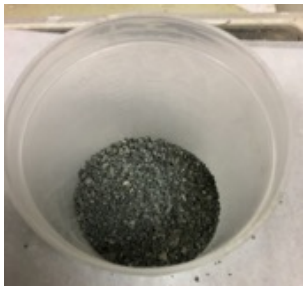
**Batch experiments with SR and Cr samples were performed for TCG and TCG-NBFS formulations. Only SR samples were used in CLSM batch experiments (Table 3).

Column Test Methods

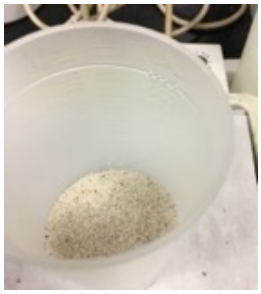
The column leaching tests were restricted to one of the size reduced treatments (i.e., 0.5 to 2 mm fraction) plus intact monoliths. The size reduced materials for the three cured paste formulations (i.e., TCG, TCG-NBFS, and CLSM) were mixed with clean quartz sand (Ottawa sand) according to the ratios defined in **Table 2** (i.e., 3.71 g sand/g TCG paste or TCG-NBFS paste, and 3.87 g sand/g CLSM paste). The sand was added to help maintain constant flow through the size-reduced TCG materials and to achieve equivalent proportions of cementitious materials and sand as in monolithic production grouts, i.e., equivalent reaction capacity. The size reduced materials plus sand (TCG + sand ≈ 100 g) were packed to a uniform bulk density. The system was then saturated from the bottom in an upflow manner to facilitate saturation. Two saturated column replicates were conducted with each of the three grout formulations under two different inlet solution treatments (open to the atmosphere and purged with UHP N_2), i.e., 3 grouts x 2 leachates x 2 replicates = 12 columns. A multi-channel peristaltic pump (Fisherbrand FH100M Multichannel Pump) was used to maintain a constant flow rate in all 12 columns. One set of columns was leached with the dilute (**Table 5**) treatment solution in equilibrium with the lab atmosphere, and the second set was leached with the same solution that was continuously purged with UHP N_2. Oxygen (O_2) levels in the headspace above the purged solution were typically less than 1%, consistent with the N_2 batch treatments. For one set of column replicates, the effluent pH and ORP were manually monitored on a weekly basis. To reduce leachate exposure to the atmosphere that could result in anomalous changes to pH and ORP values, a section of tubing containing effluent exiting the columns was collected for immediate measurement of pH and ORP before exposure. The pH and ORP values for the second column replicate were monitored continuously using flow-through pH and ORP cells.

Figure 6. Mixing TCG materials with sand for column leaching tests.

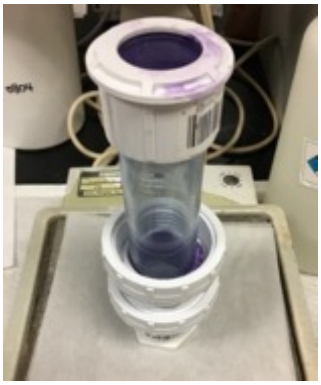
A. **Size Reduced TCG**



B. Ottawa Sand



C. Empty Flow-Thru Cell



D. Cell packed with TCG/Sand Mixture

After saturation, the pressure head was continuously monitored at the inlet of each column as an indicator of hydraulic conductivity and column plugging. A 0-30 PSI pressure transducer was attached to each column inlet and the output voltage was calibrated to a water column that was open to the atmosphere at the column inlet. Column leachate samples were collected each week and weighed to verify flow rates, and samples were retained for subsequent analysis of major elements (i.e., Na, K, Ca, Mg, Al, and Fe) by ICP-MS.

TCG Analysis

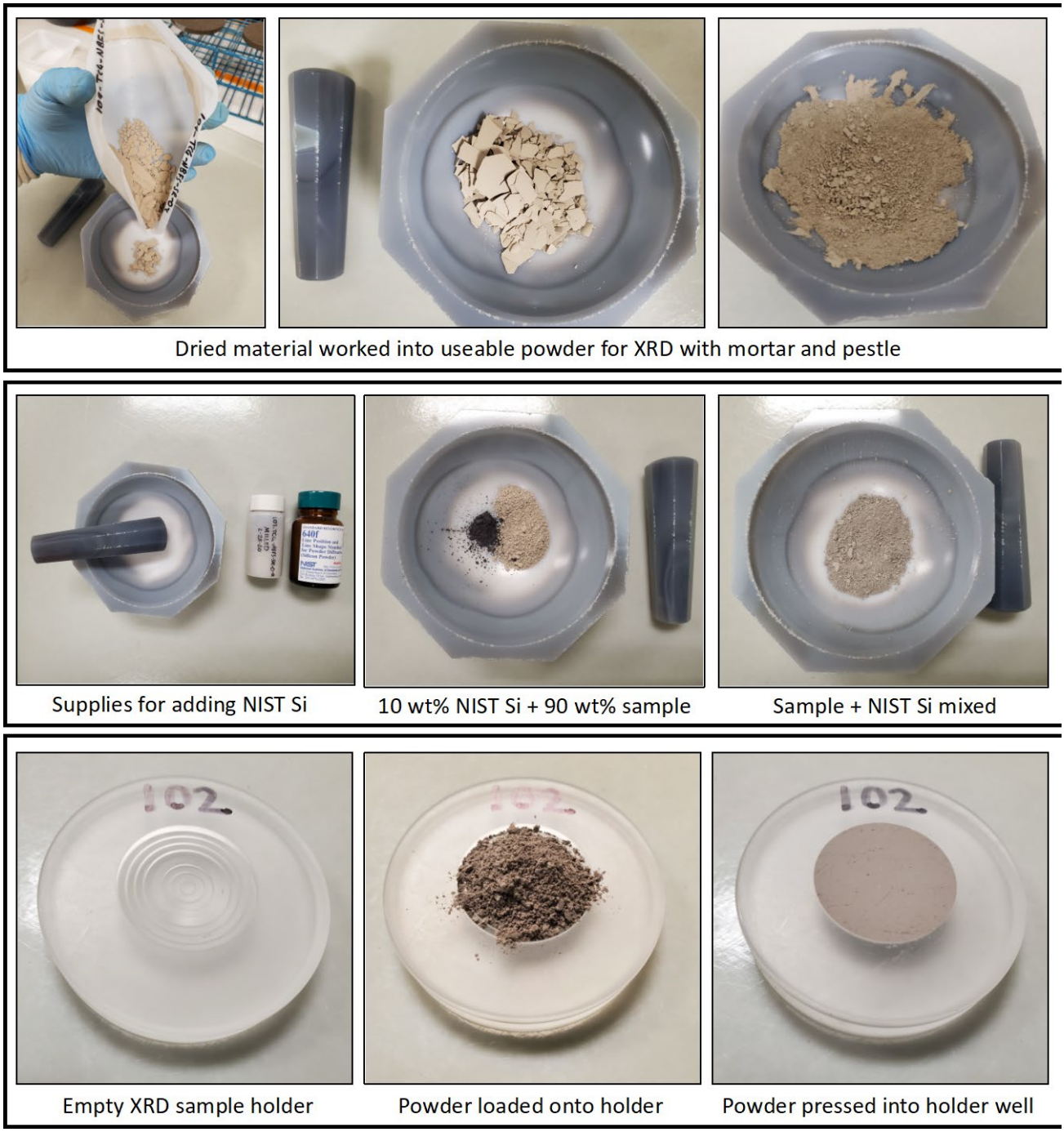
The initial dry cementitious materials, resulting paste formulations, and size-reduced materials exposed to the batch leaching scenarios were analyzed for mineralogy by x-ray diffraction (XRD; ASTM C1365-06) and composition by x-ray fluorescence (XRF; ASTM C114-15). Note that XRD was performed on Replicates A and C though no XRD pattern differences were detected between the replicates; **hence, only XRD data for Replicate C is presented in this report.** Prior to XRF and XRD analysis the dry feeds and test grout pastes were wet milled using isopropyl alcohol (3 g sample/7 mL isopropyl alcohol) for 10 minutes in a XRD-McCrone Mill (Retsch Inc.) at the level 4 power setting. The McCrone Mill was designed to rapidly grind materials for XRD analysis while preserving the materials crystalline structure, and has been widely used in the preparation of cementitious materials for XRD analysis (Schreiner et al., 2018; Snellings et al., 2014). As noted in the discussion of the batch methods, the TCG materials were all freeze dried prior to milling for XRD and XRF analysis. After grinding, the slurry was washed into a recovery pan using additional alcohol and then placed in a drying oven at 40 ºC.

The dry feed materials and several certified reference materials (CRMs) were initially prepared as pressed pellets for XRF analysis. However, discrepancies were noted in the results when compared to previous grout data. Homogeneous glass beads were created using a 1 gm of milled sample mixed with 10 gm of lithium borate flux (49.75% Lithium Tetra-borate/49.75% lithium metaborate with 0.5% lithium bromide wetting agent) and fused using a Katanax X-300 Fusion Fluxer (SPEX SamplePrep. Inc., Metuchen, NJ 08840). A photograph of some of the resulting flux beads is shown in **Figure 7**.

Figure 7. Photograph of 40 mm diameter, homogeneous lithium borate flux beads made from (Bottom: Left to Right) flux blank with no sample added, TCG, TCG-NBFS, and an iron steel slag material.

For XRD, the dried size-reduced samples were worked into a powder and mixed with 10 wt% National Institute of Standards & Technology (NIST) Standard Reference Material (SRM) Silicon. The NIST silicon is used both as a means to correct peak displacement within the XRD patterns but also as an internal standard (of known wt%) that aids in the quantification of amorphous and crystalline phases in the samples. **Figure 8** indicates the steps involved in preparation of the sample for XRD analysis.

Figure 8. Preparation of samples for XRD analysis.



Samples were analyzed using a Bruker D2 Phaser XRD (Bruker AXS LLC, Madison, WI) set to the following scan parameters:

2θ Range	Step Size (Δ2θ)	Time per Step	Sample Spin Speed
5-70°	0.02°	4 sec.	30 rpm

Phase identification was achieved using Bruker DIFFRAC.EVA (https://www.bruker.com/products/x-ray-diffraction-and-elemental-analysis/x-ray-diffraction/xrd-software/eva.html) by comparing each pattern to reference patterns (referred to as powder diffraction (PDFs)) compiled by the International Center for Diffraction Data (ICDD). Specifically, the authors utilized the ICDD PDF-4+ (2019) database (refer to the ICDD website at http://www.icdd.com).

Phase proportions were determined via Rietveld analysis using Bruker DIFFRAC-TOPAS (https://www.bruker.com/products/x-ray-diffraction-and-elemental-analysis/x-ray-diffraction/xrd-software/topas.html). The software uses a curve-fitting algorithm to match the peaks of reference samples to those identified in the XRD scan. Curve-fitting each phase ultimately allows the proportions (wt%) of the crystalline phases to be calculated. The use of the internal Si standard also allows the proportion of amorphous phase in each sample to be determined. Additional information regarding the fundamentals of Rietveld refinement is provided at http://profex.doebelin.org/wp-content/uploads/2015/02/Lesson-1-XRD-and-Rietveld-Refinement.pdf

RESULTS

Batch Tests – pH and E_h

Table 17 in the Conclusions section provides a summary of all the batch and column data. Both the "crushed" and "size-reduced" fractions for the test materials performed in a similar fashion, so all of the presented data reflects the average of the two size fractions combined. In addition, the pH and E_h does not include sample replicates that were used for the enhanced leaching treatments discussed below. The pH and E_h values for the batch equilibration experiments are provided in **Figure 9** for all three atmospheric treatments, i.e., open lab atmosphere (**9A and 9D**), N_2 purged atmosphere (**9B and 9E**), and Coy Chamber reducing atmosphere (**9C and 9F**). A clear difference was observed in the pH values under all three atmospheric treatments, with the TCG displaying the highest pH followed by the TCG-NBFS and finally the CLSM, with both the TCG and TCG-NBFS appearing to better maintain a higher pH (i.e., buffer the system) than the CLSM under all three test atmospheres. The observed pH values were consistent with geochemical modeling and laboratory tests evaluating contaminant solubility in tank waste grouted systems, with both the TCG and TCG-NBFS displaying pH values that are 11.1 (see **Table 1**) or higher under all test environments (SRNL-STI-2015-00446; SRNL-STI-2016-00432; SRNL-STI-2018-00484).

Trends with respect to E_h for the three different TCG paste formulations were more difficult to discern given the level of scatter in the data (**Fig. 9D** thru **9F**). As expected, the lowest E_h values were

observed for all samples equilibrated in the anaerobic Coy Chamber under a H_2/N_2 atmosphere (**9F**), with the N_2 glovebag (**9E**) yielding the next lowest values and the batch samples open to the lab atmosphere yielding the highest E_h values (**9D**). In general, the TCG materials containing the BFS provided the lowest E_h values (i.e., most reducing) under most test conditions, followed by the TCG-NBFS and then CLSM. For TCG, the E_h ranged from -0.42 to 0.16 V in the Coy Chamber, -0.12 to 0.18 V under the N_2 atmosphere and 0.12 to 0.26 V under the oxidizing open system. For the TCG-NBFS, the E_h ranged from -0.36 to 0.23 V in the Coy Chamber, 0.03 to 0.22 V under the N_2 atmosphere and 0.16 to 0.28 V under the oxidizing open system. And finally, for the CLSM the E_h ranged from -0.45 to 0.30 V in the Coy Chamber, 0.03 to 0.27 V under the N_2 atmosphere and 0.20 to 0.35 V under the oxidizing open system. The similar performance of the three TCG formulations in the Coy Chamber suggests that the results were more controlled by the atmospheric condition than the TCG formulation.

These results are generally consistent with previous reports concerning the inability to observe the low E_h values assumed in the TF PA geochemical modelling for RRII (-0.47 V) in the absence of added amendments (SRNL-STI-2015-00446; SRNL-STI-2016-00432; SRNL-STI-2018-00484). Even though the E_h values were generally higher than the RRII target (-0.47 V), they still remained lower than two oxidized grout stages, ORII (0.56 V) and ORIII (0.68 V), for both the open and N_2 purged systems.

Batch Tests – Major Cations (Na, K, Ca, Mg, Al, and Fe)

Figures 10 and 11 provide the pore water composition data for the three TCG formulations in the open atmosphere treatment and the Coy Chamber. Similar data for the N_2 purge treatment was not collected due to logistical limitations incurred from lab closure. It is important to note that very limited solution was removed from the batch equilibrations during initial sampling for major cation analysis (1 mL for each sampling event), and that solution was replaced with 1 mL of fresh simulant. Thus, changes in pore water composition during this period reflect reactions occurring within the batch tube. After 130 days, labeled “Enhanced Leaching” on **Figures 10 and 11**, an additional 10 mL was added to Replicate C for each treatment and then removed the following day to mimic pore water turnover. This was continued until the batch studies were suspended, yielding 420 mL of leachate solution. Just prior to starting the Enhanced Leaching, Replicate A from each batch treatment was removed from testing for XRF and XRD analysis. Replicate B was maintained and monitored in the same manner as before the “Enhanced Leaching” began for Replicate C.

In **Figures 10 and 11**, it is apparent that most of the soluble components are derived from the TCG materials, as demonstrated by comparison with the control batch tubes that contained no grout materials, only the dilute pore water simulant (**Table 5**). When the enhanced leaching started, the concentration of all measured cations present in the pore water simulant decreased dramatically. The final concentrations for Ca, Na, Mg and K after enhanced leaching are also reported in **Table 17** for comparison with the concentrations used in geochemical modeling efforts for predicting contaminant solubility in grouted tank systems.

Figure 9. Batch pH and redox potential values for batch grout samples equilibrated under three treatment environments: Oxic – the open laboratory; Anoxic – inside an N_2 bag (< 1% O_2); and Reducing – inside a Coy Chamber (H_2/N_2 atmosphere).

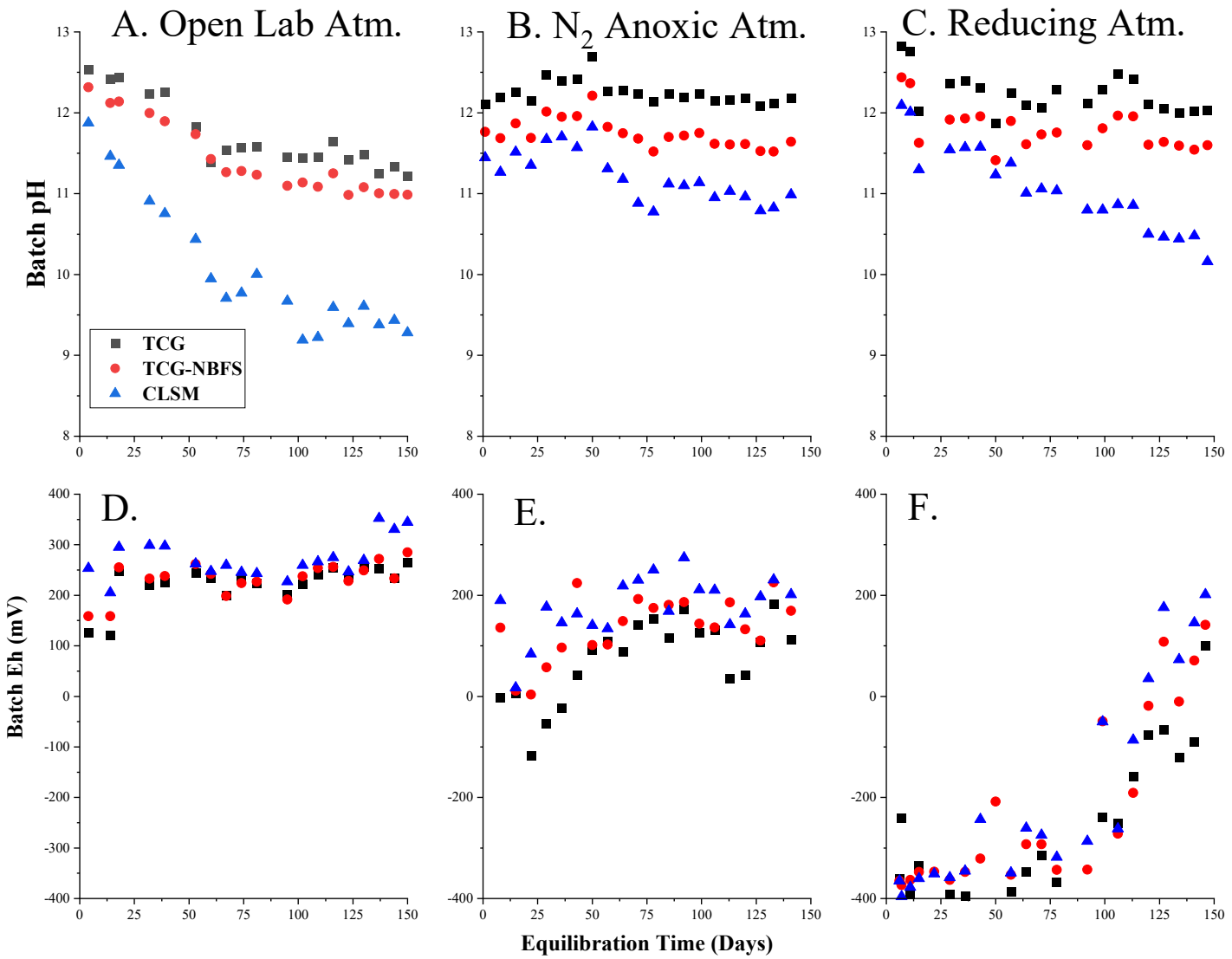


Figure 10. Major cations (Na, K, Ca) under two batch treatment environments: Oxic – the open laboratory and Reducing – inside a Coy Chamber (H_2/N_2 atmosphere).

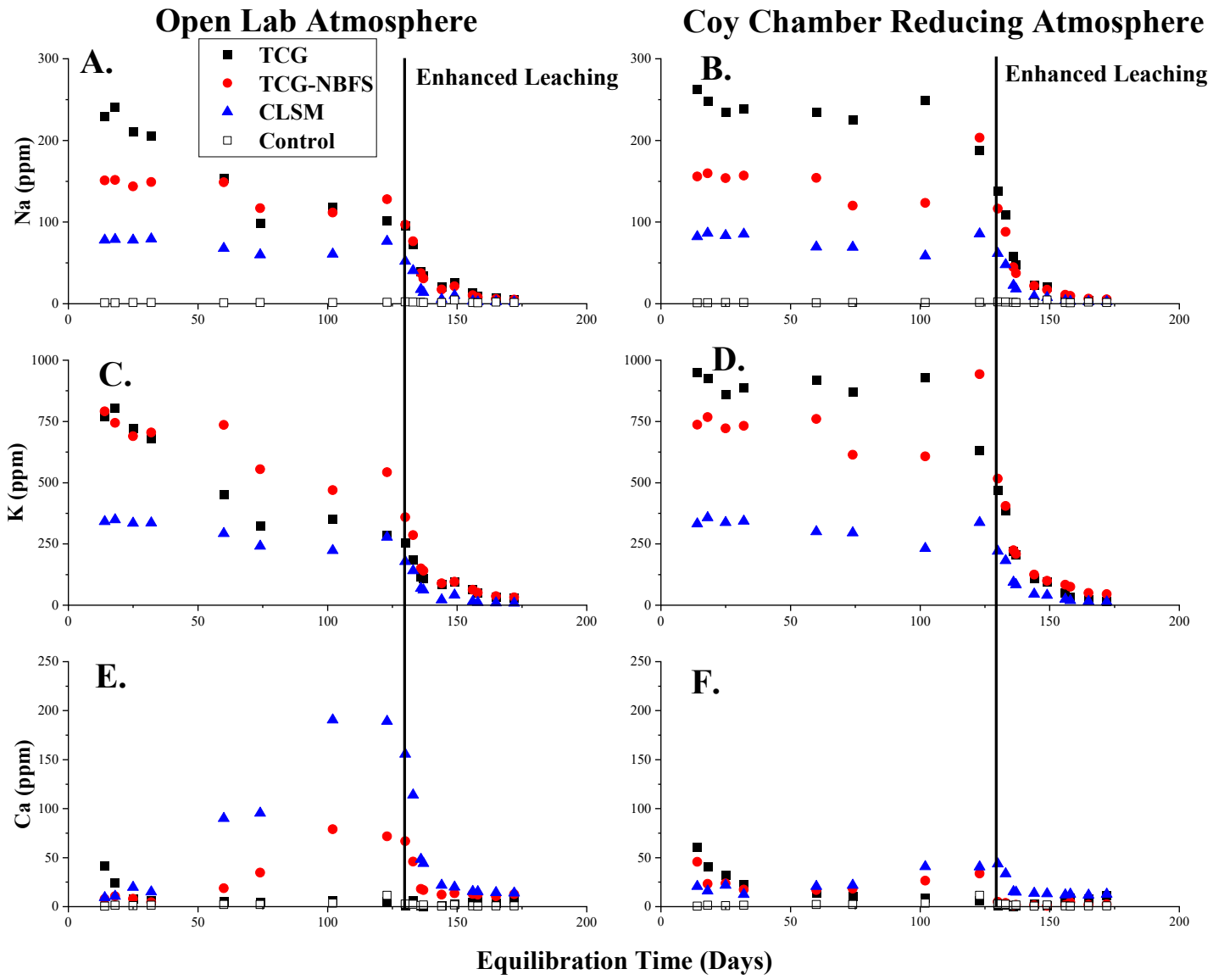
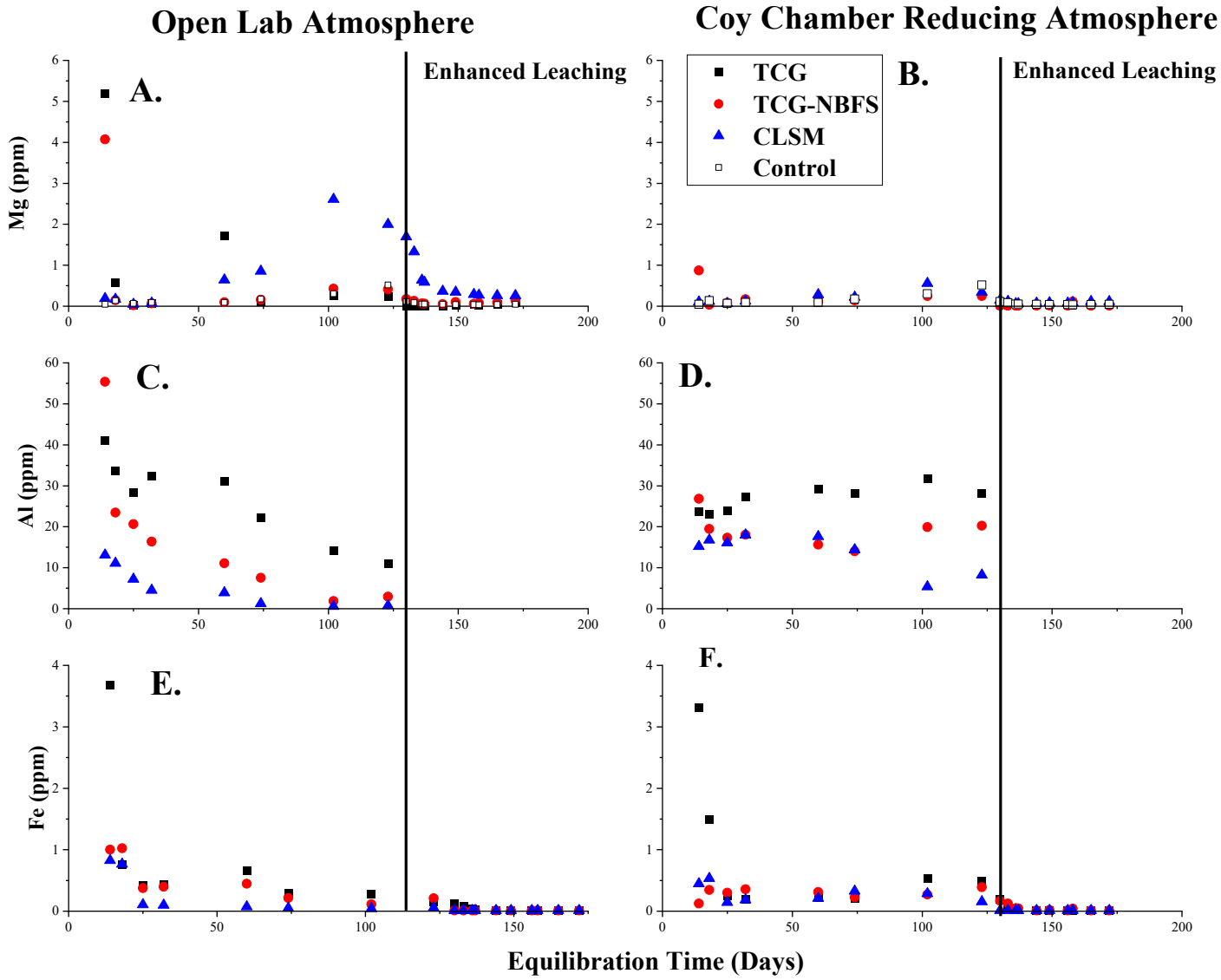


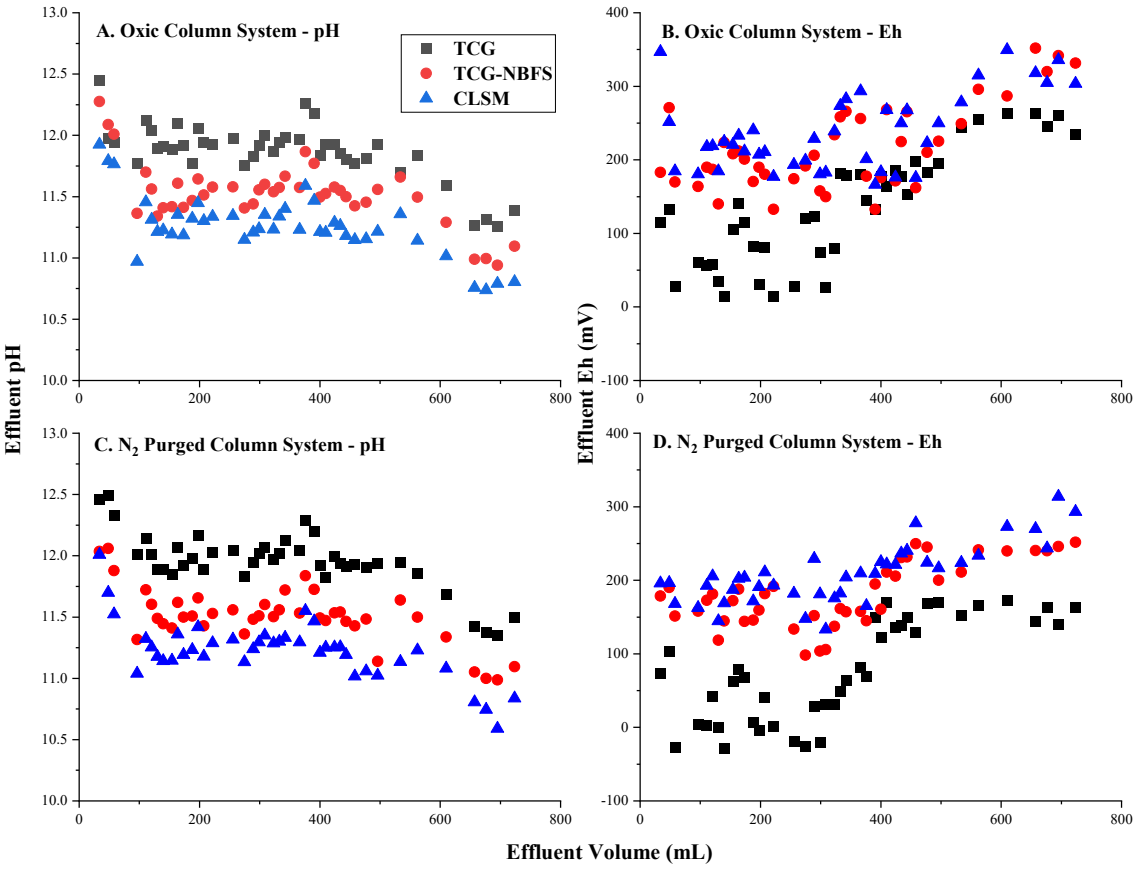
Figure 11. Major cations (Mg, Al and Fe) under two batch treatment environments: Oxic – the open laboratory and Reducing – inside a Coy Chamber (H_2/N_2 atmosphere).



Column Results – pH and E_h

The pH and E_h results for the manual column systems are presented in **Figure 12**. The total leached effluent at the time the column tests were suspended reflects an L/S ratio (paste solids only, not sand) of ≈ 38, with an initial flow rate of ≈ 5.0 mL d^{-1}. No decrease in saturated hydraulic conductivity was observed over the course of leaching for any of the column treatments based on the inlet pressure readings. It is important to note that the two column treatments consist of the dilute pore water solution in equilibrium with the lab atmosphere and the same solution constantly purged with UHP N_2. Oxygen levels in the head space above the N_2 purged leachate were typically less than 1%. Consistent with the batch studies, the TCG consistently showed the highest effluent pH in both test atmospheres, followed by the TCG-NBFS formulation and then the CLSM. The effluent pH for both systems is quite similar and appears to be decreasing with increasing leaching. Data collected from the automated column system presented in **Figure 13** suggests that the decrease in pH and the increases in E_h may reflect limited changes associated with sample exposure to the atmosphere while taking the measurements manually. Even so, there was not a great deal of difference in the pH values for the two atmosphere treatments, with the TCG pH remaining above the 11.1 value used for geochemical modeling.

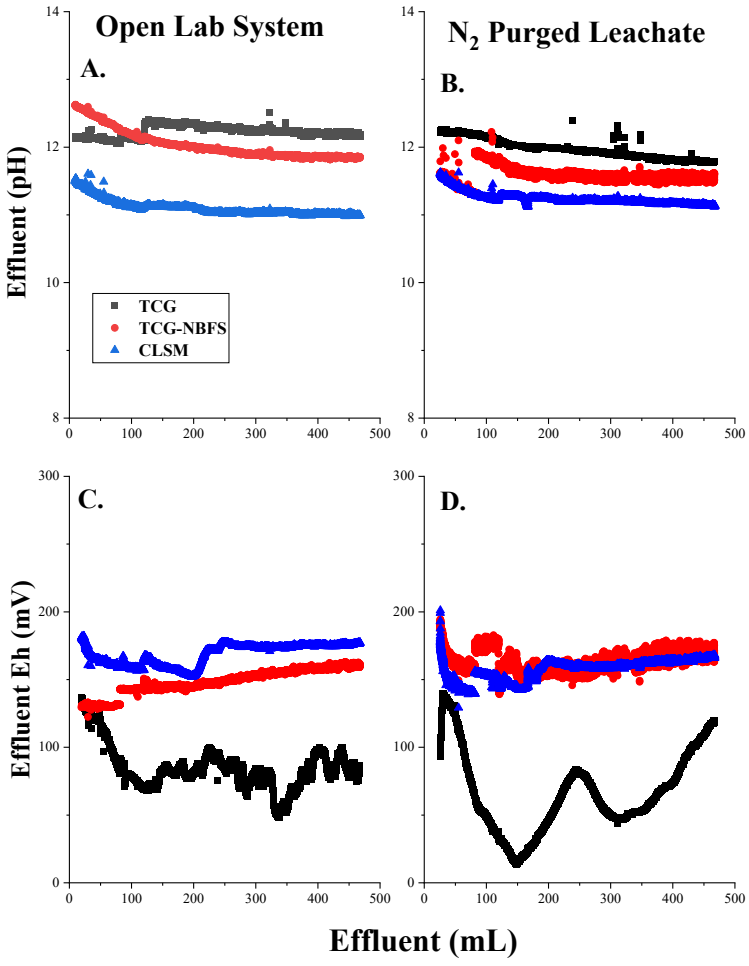
Figure 12. Effluent pH and E_h values monitored manually for column leaching tests using various TCG formulations subject to two different inlet solution treatment scenarios.



Observed trends with respect to the E_h results for the three materials are somewhat clearer than observed for the batch experiments, although there is still considerable data fluctuation and overlap, with initial values E_h for TCG starting out around 0.0 V and increasing to around 0.3 and 0.2 V for the open and N_2 purged systems, respectively. Although this is higher than the value used in PA TF geochemical models for the RRII stage of weathering, it is much lower than the ORII or ORIII E_h values as well (**Table 1**).

The pH and E_h results for the column systems monitored continuously with flow-through cells are presented in **Figure 13**. As observed for the manual columns the effluent pH was generally higher for TCG followed by TCG-NBFS and then the CLSM for both atmospheric treatments and remain fairly constant over the course of leaching. However, the manual columns were subject to greater leaching because the manual columns were started at an earlier date.

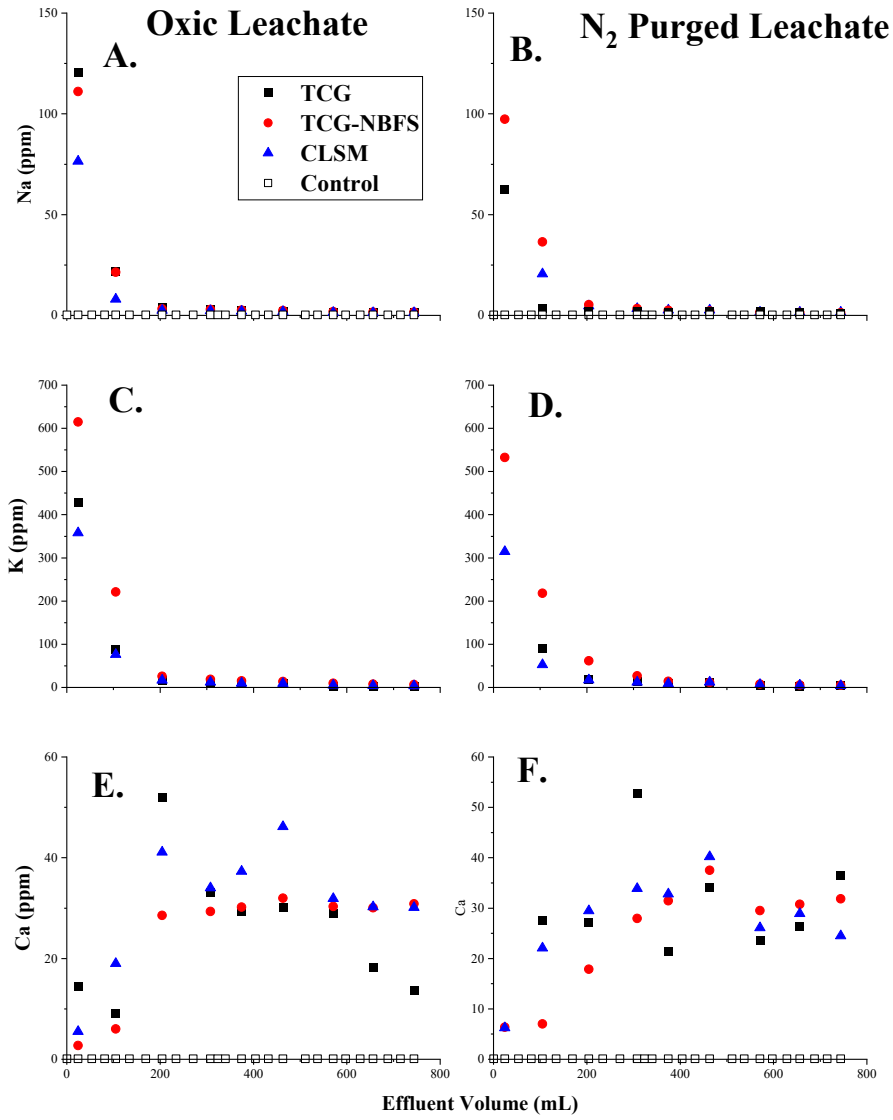
Figure 13. Effluent pH and E_h values monitored with flow through cells for column leaching tests using various TCG formulations subject to two different inlet solution treatment scenarios.



The major cations present in the column leachates are provided in **Figures 14** and **15**. As with the batch data, the column results are summarized in **Table 17**. Such information can be used to

constrain future modeling efforts aimed at evaluating the aging of waste tank grout. The inlet levels of cations (i.e., Na, Ca, and Mg in **Table 5**) present in the test solution (i.e., Control) are provided in the graphs for comparison. In general, the leaching trends are quite similar under the two atmospheric treatments. A significant amount of Na and K are initially leached from all the TCG materials, with Na levels approaching those of the inlet test solution, 0.8 ppm. In contrast, the Ca levels display a more complicated leaching history, at first increasing and then decreasing with continued leaching, but still much higher than the inlet solution, i.e., control.

Figure 14. Major cations (Na, K and Ca) present in effluent from the column experiments. Data reflects effluent from manual flow through columns.



The leaching behavior for the other major cations is a bit more complicated (**Figure 15**). Magnesium levels in the column effluent are initially quite low for all TCG formulations, lower than the inlet solution, 0.03 ppm (**Table 5**). Over time they appear to increase for the CLSM and TCG-NBFS treatments, with levels that are a bit higher than the inlet solution. In contrast the Mg levels for

TCG show little change, remaining around 0.01 ppm. For effluent Al, there are initially higher levels in the TCG-NBFS and CLSM leachates that decrease over time to a value < 5.0 ppm. This is still higher than the level predicted by geochemical modeling to mimic the test solution (**Table 4**), despite the fact that no Al was added to the inlet solution for these studies. Even though the effluents were filtered (0.45 µm pore size) before analysis, these levels may reflect colloidal materials. The levels of dissolved Fe were generally quite low, which is indicative of the moderate reducing conditions for both test atmospheres.

Figure 15. Major cations (Mg, Al and Fe) present in effluent from the column experiments. Data reflects effluent from manual flow through columns.

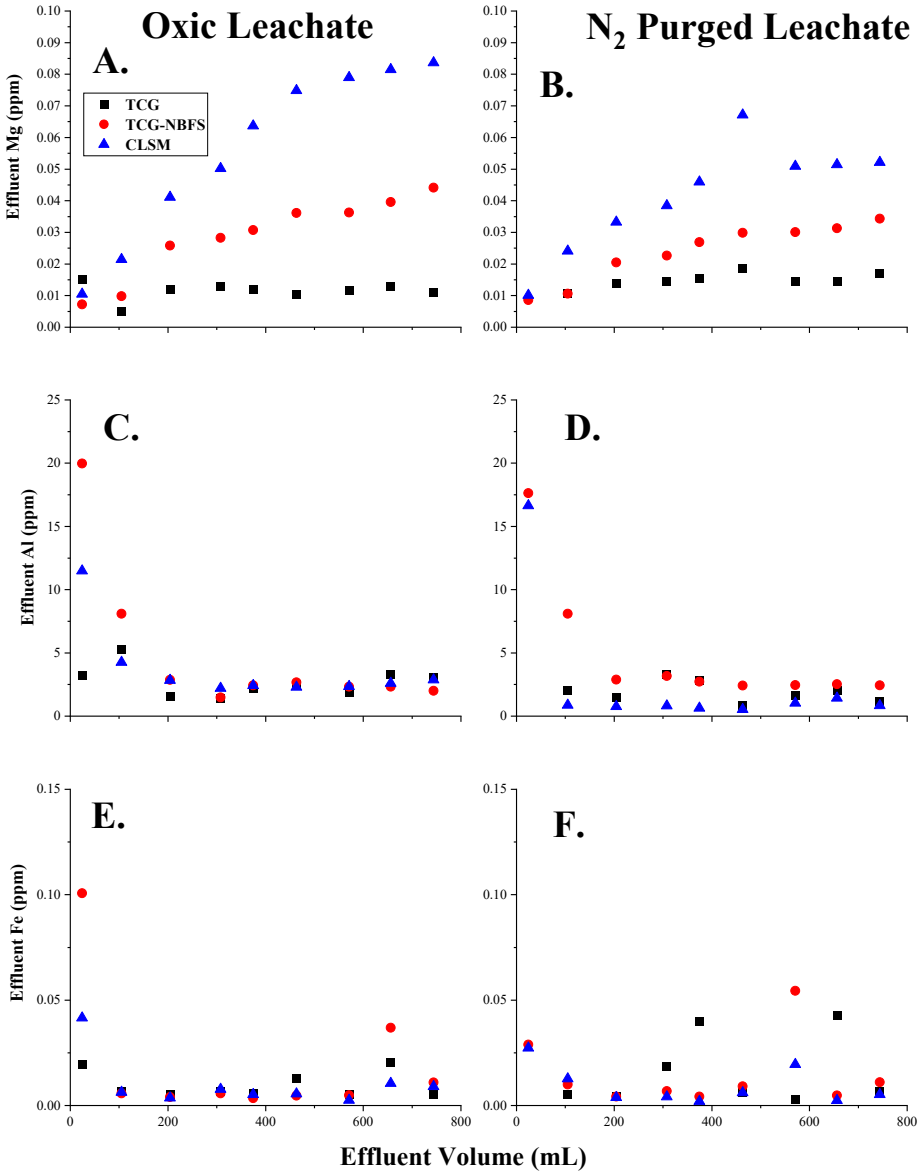
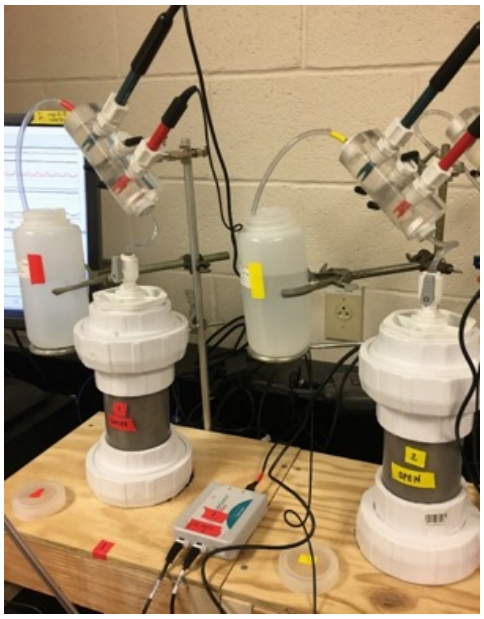


Figure 16A shows the automated column system with two sand-packed columns surrounding intact TCG + Sand monoliths in a manner such that pore water flows in an upward direction contacting the outer monolith surface, maintaining saturation, and then exiting the column and passing through a flow-thru cell containing both a pH and ORP electrode. The solution then flows into a 1L plastic bottle for sample collection. The effluent pH and ORP values for both columns were constantly monitored. **Figure 16B** provides the effluent pH and E_h values for the two columns, the only difference being the inlet treatment solution, with one equilibrated with the lab atmosphere and the second leached with the same test solution that was constantly purged with UHP N_2.

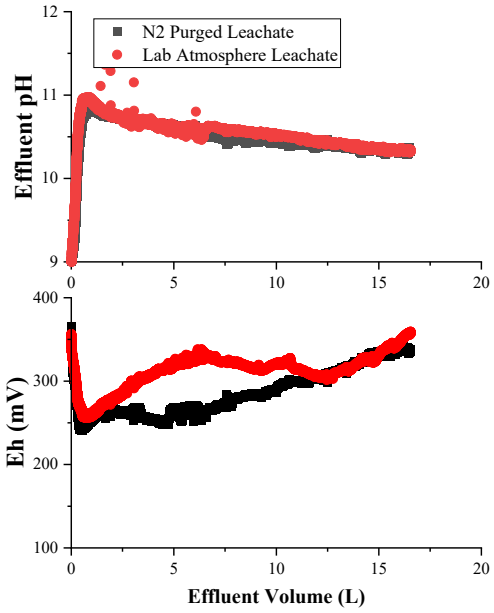
Despite the different inlet solutions, the effluent pH and E_h values were quite similar for the two columns. In both cases it initially took a couple pore volumes for the effluent pH to reach a maximum of 11 before slowly decreasing to about 10.5 with extensive leaching. Aside from occasional scatter in the data, it is difficult to distinguish between the pH results of the two inlet solution treatments. Even so, the significant rise in pH when compared to the inlet solutions (pH ≈ 6 to 7) demonstrates the impact of the TCG monoliths on the effluent pH. The effluent E_h values were also quite similar for the two treatments except for a time during leaching when the column leached with the inlet solution in equilibrium with the atmosphere was somewhat higher (≈ 25 to 50 mV). Although the lower E_h is consistent with less dissolved O_2 present in the N_2-purged inlet solution treatment, it may reflect drift in the ORP electrode, as the data become quite similar with continued leaching. However, the E_h values for the intact columns were considerably higher than values observed for columns packed with size-reduced TCG paste under the same inlet solution treatments. This suggests that the impact of the two TCG + sand monoliths may be limited by slow diffusion (mass transfer) of pore solutions within the intact monoliths when compared to the much faster movement of pore solutions contained within the surrounding sand matrices (i.e., mobile vs immobile pore water). It is important to note that the pH and ORP electrodes were routinely calibrated on a weekly basis.

Figure 16. Large columns containing intact TCG + Sand monoliths packed in sand.

A.



B.



XRF Analysis of TCG Paste Formulations

The composition of the dry feed materials and two CRMs are reported in **Table 7**. The data shows a comparison between of the two sample preparation methods for XRF analysis, i.e., pressed pellets and fused lithium-borate beads. In general, the two sample preparation method yielded quite similar results for the two CRMs, with major elements generally displaying recoveries within ± 5% of the certified value. The dry feed compositions are generally consistent with reported compositions used by Denham and Millings (SRNL-STI-2012-00404, from Langton, 2009, SRNL-STI-2009-00477) for estimating the solubility of residual contaminants present in grouted tank waste, and by Dyer (2018, SRNL-STI-2018-00586) in modeling SDU pore fluids resulting from Saltstone and SDU grout aging. However, two clear differences are evident with respect to the CaO and SiO_2 contents for the FA (2019-IR-05-0195), with the pressed pellets providing an overestimate of the CaO content and an underestimate of the SiO_2 content. Thus, the flux bead method was used for the preparation of all samples for XRF analysis. The flux bead method is also preferable in that it yields a stable test sample that can be stored indefinitely for reanalysis later.

Table 8 provides the XRF results for the initial grout formulations, plus grout samples that were subjected to various batch leaching scenarios. Three sets of data reflect samples that were initially cured 90 days, size reduced, and then equilibrated approximately 130+ days (i.e., Replicate A) under the three test atmospheres, the open lab, the N_2 purged glovebag and the H_2/N_2 Coy Chamber. As expected, results for these samples are consistent with the initial cured formulations because very little mass was ever removed from the batch vessels. The remaining two sets of samples reflect the enhanced leaching treatments from the open lab and Coy Chamber batch tests.

A mass balance calculation was performed on the major cation extraction data for the batch tests presented in **Figures 10 and 11**, starting at the point when the "enhanced leaching" began, with a total extraction volume of approximately 420 mL for each sample. The sum of component mass (i.e., Na, K, Ca, Mg, Al, and Fe) extracted was then converted to an oxide basis for comparison with the XRF results. These values were then subtracted from the initial material composition based on XRF, assuming an initial sample mass for the batch tests of ≈ 20 g. The results for these two sets do show evidence of loss of Na and K in the XRF results. In **Table 9**, the Na_2O and K_2O results for the initial three TCG formulations and those formulations subject to enhanced extraction are provided for comparison. The total mass loss for the other measured components was too small to be detected in the XRF data.

Table 7. Chemical composition of standard reference cements and the initial dry feed materials analyzed by XRF using the pressed pellet and the lithium borate fused bead sample preparation method.

		Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	SiO_2	SO_3	TiO_2	ZnO	SrO
SRM FLX110	Certified Values	4.70	68.13	0.18	0.94	0.65	0.05	22.01	2.88	0.17	0.003	0.04
Pressed Pellet Samples		4.97	67.80	0.18	0.89	0.68	0.11	22.17	2.99	0.16	0.00	0.04
	Recovery (%)	106%	100%	100%	95%	105%	220%	101%	104%	94%	33%	98%
Fused Bead Samples		4.80	65.70	0.26	1.00	0.79	0.05	22.10	2.80	0.18	0.001	0.04
	Recovery (%)	102%	96%	143%	107%	122%	100%	100%	97%	106%	40%	106%
SRM FLX131	Certified Values	23.10	42.89	3.24	0.29	1.62	0.47	8.73	18.19	1.15	0.006	0.07
Pressed Pellet Samples		22.86	43.27	3.13	0.27	1.57	0.53	8.53	18.19	1.13	0.01	0.07
	Recovery (%)	99%	101%	97%	94%	97%	114%	98%	100%	98%	150%	104%
Fused Bead Samples		22.20	43.66	3.22	0.32	1.66	0.44	8.30	16.90	1.24	0.01	0.07
	Recovery (%)	96%	102%	99%	111%	102%	94%	95%	93%	108%	132%	110%
Dry Feed Materials Pressed Pellet Results												
	Unique Identifier	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	SiO_2	SO_3	TiO_2	ZnO	SrO
Class F Fly Ash	2019-IR-05-0195	28.77	11.91	9.58	2.35	1.05	0.24	44.39		1.96	0.02	0.12
Type II Portland Cement	2019-IR-05-0201	4.62	68.28	3.23	0.56	1.58	0.17	20.57	2.30	0.20	0.09	0.09
Grade 100/120 BFS (LeHigh)	2019-IR-05-0261	13.57	42.76	0.79	0.23	6.23	0.35	31.75	2.27	0.52	0.00	0.10
Dry Feed Materials Fused Beads Results												
Class F Fly Ash	2019-IR-05-0195	25.30	1.40	9.67	2.50	0.95	0.29	56.70	0.09	1.52	0.04	0.11
Type II Portland Cement	2019-IR-05-0201	5.10	63.76	3.48	0.51	1.44	0.16	19.70	2.27	0.28	0.10	0.10
Grade 100/120 BFS (LeHigh)	2019-IR-05-0261	12.10	42.42	0.95	0.33	6.22	0.15	32.40	2.76	0.59	0.03	0.12

Table 8. Chemical composition of different initial TCG formulations, and batch-weathered TCG materials.

		Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	SiO_2	SO_3	TiO_2	ZnO	SrO
Initial Cured Materials												
TCG		**17.30**	**26.75**	**5.73**	**1.51**	**2.62**	**0.26**	**41.20**	**1.46**	**1.03**	**0.03**	**0.12**
TCG-NBFS		**21.10**	**14.23**	**8.59**	**2.24**	**1.06**	**0.25**	**48.60**	**0.56**	**1.35**	**0.04**	**0.13**
CLSM		**23.90**	**6.99**	**9.16**	**2.39**	**1.04**	**0.26**	**52.80**	**0.29**	**1.47**	**0.03**	**0.12**
		Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	SiO_2	SO_3	TiO_2	ZnO	SrO
Equilibrated in Open Lab - Removed After Batch Equilibration 130 to 140 days (Batch Rep A)												
TCG-O		**17.40**	**26.09**	**5.78**	**1.39**	**2.62**	**0.18**	**42.00**	**1.37**	**1.06**	**0.03**	**0.12**
TCG-NBFS-O		**21.50**	**14.28**	**8.58**	**2.12**	**1.08**	**0.24**	**48.50**	**0.43**	**1.33**	**0.03**	**0.13**
CLSM-O		**23.70**	**6.68**	**9.16**	**2.39**	**1.06**	**0.24**	**53.40**	**0.14**	**1.46**	**0.03**	**0.13**
Equilibrated in N_2 Chamber - Removed After Batch Equilibration 130 to 140 days (Batch Rep A)												
TCG-N2		**17.40**	**26.69**	**5.74**	**1.44**	**2.60**	**0.21**	**41.40**	**1.42**	**1.03**	**0.03**	**0.12**
TCG-NBFS-N2		**21.70**	**13.98**	**8.52**	**2.17**	**1.08**	**0.23**	**48.60**	**0.51**	**1.32**	**0.04**	**0.12**
CLSM-N2		**23.80**	**6.67**	**9.26**	**2.42**	**1.05**	**0.21**	**53.10**	**0.26**	**1.48**	**0.03**	**0.13**
Equilibrated in Coy Chamber - Removed After Batch Equilibration 130 to 140 days (Batch Rep A)												
TCG-C		**17.30**	**26.81**	**5.72**	**1.39**	**2.63**	**0.17**	**41.50**	**1.40**	**1.04**	**0.03**	**0.12**
TCG-NBFS-C		**21.60**	**14.07**	**8.51**	**2.12**	**1.12**	**0.23**	**48.70**	**0.44**	**1.34**	**0.03**	**0.13**
CLSM-C		**23.70**	**6.71**	**9.20**	**2.42**	**1.05**	**0.26**	**53.20**	**0.21**	**1.46**	**0.02**	**0.13**
		Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	SiO_2	SO_3	TiO_2	ZnO	SrO
Enhanced Leaching - Oxic Environment (Batch Rep C)												
TCG-O		**18.00**	**25.63**	**5.78**	**1.24**	**2.57**	**0.17**	**42.20**	**1.27**	**1.06**	**0.03**	**0.12**
TCG-NBFS-O		**21.70**	**13.57**	**8.70**	**1.97**	**1.09**	**0.19**	**49.30**	**0.25**	**1.35**	**0.03**	**0.13**
CLSM-O		**23.70**	**6.62**	**9.26**	**2.35**	**1.06**	**0.24**	**53.50**	**0.05**	**1.47**	**0.02**	**0.13**
Enhanced Leaching - Coy Chamber Reducing Environment (Batch Rep C)												
TCG-C		**17.50**	**26.34**	**5.80**	**1.17**	**2.64**	**0.13**	**41.80**	**1.41**	**1.05**	**0.03**	**0.12**
TCG-NBFS-C		**21.50**	**13.68**	**8.60**	**1.80**	**1.09**	**0.22**	**49.50**	**0.32**	**1.34**	**0.03**	**0.13**
CLSM-C		**23.70**	**6.44**	**9.32**	**2.29**	**1.03**	**0.24**	**53.70**	**0.10**	**1.48**	**0.02**	**0.13**

Table 9. XRF results for the K_2O and Na_2O contents of initial TCG formulations and samples exposed to extended leaching, compared to mass balance estimates of K_2O and Na_2O lost during batch equilibration, i.e., delta K_2O and Na_2O.

		K_2O	Delta K_2O*	Na_2O	Delta Na_2O*
		%		%	
Enhanced Leaching - Oxic Environment (Batch Rep C)					
TCG		1.51	0.29	0.26	0.10
TCG-O		1.24		0.17	
TCG-NBFS		2.24	0.37	0.25	0.09
TCG-NBFS-O		1.97		0.19	
CLSM		2.39	0.15	0.26	0.04
CLSM-O		2.35		0.24	
Enhanced Leaching - Coy Chamber Reducing Environment (Batch Rep C)					
TCG		1.51	0.43	0.26	0.12
TCG-C		1.17		0.13	
TCG-NBFS		2.24	0.51	0.25	0.11
TCG-NBFS-C		1.80		0.22	
CLSM		2.39	0.20	0.26	0.05
CLSM-C		2.29		0.24	

*The amount of a component removed as a weight percentage from batch samples based on the ICP-MS analysis of sampled pore solutions, e.g., an estimate of the difference on K_2O for TCG minus that in the leached TCG-O (see Fig. 10).

XRD Analysis of TCG Paste Formulations

Data presented in this section is related to phase identification and quantification for the individual dry feeds (i.e., BFS, FA, and OPC) and the pre- and post-leached tank closure paste samples from the batch equilibrations (i.e., the tank closure grout formulations without the sand component). A more thorough discussion of the XRD data (including XRD patterns) is provided in **Appendix B**, with additional documentation supporting XRD peak assignments and the matching ICDD PDFs provided in **Appendix C** and **Appendix D**, respectively.

The phases and their proportions identified for the BFS, FA, and OPC are presented in **Tables 10, 11,** and **12**, respectively. BFS (**Table 10**) is primarily composed of amorphous silicate glasses and Lehigh add approximately 2-3 wt% gypsum ($CaSO_4.2H_2O$) and limestone (calcite – $CaCO_3$), as an activator and grinding aid, respectively. BFS will exhibit pozzolanic activity in the presence of the cement reaction product portlandite ($Ca(OH_2)$) to form calcium (aluminum) silicate hydrates (C-A-S-H), which are partially responsible for the strength gain associated with BFS-containing cementitious blends. FA (**Table 11**) is a semi-amorphous material composed of silicate glasses and crystalline mullite ($3Al_2O_3•2SiO_2$), quartz (SiO_2), hematite (Fe_2O_3), and magnetite (Fe_3O_4). Similar to BFS, the

glassy phase of FA can undergo pozzolanic activity in the presence of portlandite but the crystalline phases are essentially inert. As such, the crystalline phases are expected to persist in the hydrated tank closure grout samples. The typical (and anticipated) cement phases (**Table 12**) are alite (Ca_3SiO_5), belite (Ca_2SiO_4), tri-calcium aluminate ($Ca_3Al_2O_6$), and tetracalcium aluminoferrite (Ca_2AlFeO_5). In addition, gypsum ($CaSO_4•2H_2O$), bassanite (hemihydrate - $CaSO_4•0.5H_2O$), limestone (calcite – $CaCO_3$), and trace ettringite ($Ca_6Al_2S_3O_{18}•32H_2O$) and portlandite ($Ca(OH)_2$) are also present. Gypsum is added to OPC as a set retarder and this mineral can actually lose water to form bassanite at the elevated temperatures that may occur during the clinker grinding process. Portlandite and ettringite are reaction products of cement hydration and indicate that the cement likely underwent mild hydration due to air-moisture contact during storage. The phases present in pre-hydrated cement react during hydration and are thus not anticipated to be present in the hydrated material. The primary hydration products of cement include calcium silicate hydrate (C-S-H), portlandite, AFt (alumina-ferric oxide-tri) phases (e.g., ettringite) and AFm (alumina-ferric oxide-mono) phases (e.g., monosulfoaluminate) (refer to **Appendices A** and **C** for additional information on cement hydration products). Though portlandite is a primary reaction product of cement it is expected to be consumed by secondary cement reactions, by carbonation, and by the pozzolanic reactions of BFS and FA.

Table 10. Phases and wt% in BFS.

Batch ID	**Phase (wt%)**			
	Amorphous	*Gypsum*	*Calcite*	*Quartz*
2019-IR-05-0261*	95.63	2.15	2.22	Trace**

* The manufacturer certification report (MCR) for BFS is provided in **Appendix A**.

** The quartz polymorphs described for BFS in **Appendix B** were barely above background and not included in Rietveld quantification.

Table 11. Phases and wt% in FA.

Batch ID	**Phase (wt%)**				
	Amorphous	*Mullite*	*Quartz*	*Hematite*	*Magnetite*
2019-IR-05-0195*	63.92	17.63	13.33	2.50	2.61

* The MCR for FA is provided in **Appendix A**.

Table 12. Phases and wt% in OPC.

Batch ID	Phase (wt%)								
	Alite	*Belite*	*Aluminate*	*Ferrite*	*Gypsum*	*Bassanite*	*Calcite*	*Portlandite*	*Ettringite*
2019-IR-05-0201*	64.06	10.67	2.17	15.12	2.24	1.55	3.28	0.90	Trace **

* The MCR for OPC is provided in **Appendix A**.

** Trace is indicated for ettringite because the peak is barely above background and its inclusion (at such low concentrations) in the Rietveld refinement resulted in pattern simulation anomalies; hence it was omitted during Rietveld quantification (refer to **Appendix B** for additional information).

Table 13 contains a summary of the phases identified in each of the batch tested pre- and post-leached tank closure paste formulations with a qualitative indication of confidence in phase identification. The bases for the confidence levels are discussed in **Appendix B**.

Tables 14, 15, and **16** provide phase proportions for CLSM, TCG-NBFS, and TCG, respectively. It is important to note, however, that not all of the phases identified by XRD were quantifiable since in many cases the crystal structure information required for Rietveld refinement was missing from the ICDD PDFs. Phase identification and quantification data are presented in greater detail in **Appendices B** and **C** and what follows is a brief data summary.

X-ray diffraction patterns of both pre-leached and leached samples were dominated by amorphous phases presumed to be associated with unreacted silicate glasses in the BFS and FA components, and amorphous calcium silicate hydrate (C-S-H) and calcium aluminosilicate hydrate (C-A-S-H) gels formed via the hydration/pozzolanic reactions of the OPC, BFS, and FA. As anticipated the poorly reacting crystalline phases of FA (i.e., quartz, mullite, hematite, and magnetite) were also detected in all pre-leached and leached samples. In addition to the aforementioned phase constituents, all pre-leached samples indicated the presence of strätlingite (**Appendix C-2**), calcite (**Appendix C-1**), ettringite (**Appendix C-3**), and varied AFm carbonates and/or sulfates (including hemicarboaluminate, monocarboaluminate, and/or monosulfoaluminate) (**Appendices C-4 and C-5**). Ettringite was not detected in any leached samples, and the AFm phases either disappeared in the leached samples or their proportions were significantly reduced. Strätlingite persisted to some degree in almost all of the leached samples irrespective of the leaching environment; however, it was barely detectable (and therefore not quantifiable) for the CLSM, TCG-NBFS, or TCG subjected to a reducing environment. In addition, for all TCG samples (containing BFS) strätlingite was barely apparent above background irrespective of the leaching environment. Calcite persisted in all samples and the highest calcite proportions were detected for samples subjected to the oxic environment during leaching. These results were anticipated and presumed due to sample carbonation in the CO_2-containing oxic environment. Hydrotalcite (**Appendix C-8**) and a phase tentatively identified as kuzelite (or monosulfoaluminate) (**Appendix C-7**) were also observed but only in the BFS-containing TCG samples; both minerals persisted in all the leached TCG samples irrespective of leaching environment. Hydrotalcite was

anticipated due to the magnesia (MgO) content of the BFS (approximately 6 wt% measured via XRF). Monosulfoaluminate is a reaction product of tricalcium aluminate (a primary cement phase) and ettringite (a cement hydration product).

Table 13. Summary of phases identified by XRD analysis of batch-treated TCG samples.

PHASE	SAMPLES OBSERVED IN			CONFIDENCE LEVEL
	CLSM	TCG-NBFS	TCG	
Strätlingite $Ca_2Al_2SiO_7.8H_2O$	• CLSM-REF • CLSM-OPEN • CLSM-CLOSED • CLSM-N_2	• TCG-NBFS-REF • TCG-NBFS-OPEN • TCG-NBFS-N_2	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2	HIGH
Ettringite $Ca_6Al_2(SO_4)_3(OH)_{12}(H_2O)_{26}$	• CLSM-REF	• TCG-NBFS-REF	• TCG-REF	HIGH
Kuzelite $Ca_2Al(SO_4)_{0.5}(OH)_6(H_2O)_3$	• Not Observed	• Not Observed	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2	MEDIUM
Calcium Iron Oxide Sulfite Hydrate $Ca_4Fe_2O_6(SO_3).12H_2O$	• Not Observed	• Not Observed	• TCG-REF • TCG-CLOSED • TCG-N_2	LOW
Calcium Aluminum Silicate Hydrate $CaAl_2Si_7O_{18}.1.7H_2O$	• Not Observed	• TCG-NBFS-REF	• Not Observed	LOW
Calcium Aluminum Oxide Carbonate Sulfate Hydroxide Hydrate $3CaO.Al_2O_3.0.17CaSO_4.0.5Ca(OH)_2.0.33CaCO_3.xH_2O$	• CLSM-REF	• Not Observed	• TCG-REF • TCG-OPEN	LOW
Calcium Aluminum Carbonate Hydroxide Hydrate (Hemicarboaluminate) $Ca_2Al(CO_3)_{0.25}(OH)_{6.5}(H_2O)_2$	• CLSM-REF	• TCG-NBFS-REF	• TCG-REF	MEDIUM
Calcium Aluminum Iron Oxide Carbonate Hydroxide Hydrate $Ca_8Al_2Fe_2O_{12}CO_3(OH)_2.22H_2O$	• CLSM-REF	• TCG-NBFS-REF • TCG-NBFS-CLOSED • TCG-NBFS-N_2	• TCG-OPEN • TCG-CLOSED • TCG-N_2	LOW
Hydrotalcite $Mg_{0.67}Al_{0.33}(CO_3)_{0.17}(OH)_2(H_2O)_{0.5}$	• Not Observed	• Not Observed	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2	MEDIUM
Calcium Aluminum Carbonate Hydroxide Hydrate (Monocarboaluminate) $Ca_4Al_2(CO_3)(OH)_{12}(H_2O)_5$	• CLSM-REF	• TCG-NBFS-REF • TCG-NBFS-CLOSED • TCG-NBFS- N_2	• Not Observed	MEDIUM

PHASE	SAMPLES OBSERVED IN			CONFIDENCE LEVEL
	CLSM	TCG-NBFS	TCG	
Mullite *General: $3Al_2O_3.2SiO_2$* *Actual: $Al_2(Al_{2.588}Si_{1.412})O_{9.706}$*	• All Samples	• All Samples	• All Samples	HIGH
Portlandite *$Ca(OH)_2$*	• Not Observed	• Not Observed	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2	MEDIUM
Quartz *SiO_2*	• All Samples	• All Samples	• All Samples	HIGH
Calcite *$CaCO_3$*	• All Samples	• All Samples	• All Samples	HIGH
Hematite *Fe_2O_3*	• All Samples	• All Samples	• All Samples	HIGH
Silicon *Si*	• All Samples	• All Samples	• All Samples	HIGH
Calcium Silicate Hydrates *C-S-H*	• Possibly present in all samples but most predominant in TCG-REF			MEDIUM
Magnetite *Fe_3O_4*	• All Samples	• All Samples	• All Samples	HIGH

Table 14. Results for the Rietveld Refinement of the batch-treated CLSM formulations.

CLSM Sample	Phase (wt%)									
	Amorphous	*Mullite*	*Quartz*	*Hematite*	*Magnetite*	*Calcite*	*Strätlingite*	*Ettringite*	*Hemicarbo-aluminate*	*Monocarbo-aluminate*
REF (Non-Leached)	66.12	14.11	10.23	2.09	1.10	0.88	2.72	0.49	1.88	0.38
OPEN	65.55	14.75	10.95	2.12	1.48	3.01	2.14	-	-	-
CLOSED	68.79	14.75	11.19	2.12	1.64	1.51	Trace *	-	-	-
N_2	66.92	15.24	11.24	2.14	1.69	1.10	1.71	-	-	-

* Trace is indicated for strätlingite because the peak is barely above background and its inclusion (at such low concentrations) for Rietveld refinement resulted in pattern simulation anomalies; hence it was omitted during Rietveld quantification

Table 15. Results for the Rietveld Refinement of the batch-treated TCG-NBFS formulations.

TCG-NBFS Sample	Phase (wt%)									
	Amorphous	*Mullite*	*Quartz*	*Hematite*	*Magnetite*	*Calcite*	*Strätlingite*	*Ettringite*	*Hemicarbo-aluminate*	*Monocarbo-aluminate*
REF (Non-Leached)	66.97	11.69	8.64	1.79	0.99	2.63	1.85	1.41	2.61	1.43
OPEN	68.66	11.87	9.07	1.81	1.01	6.08	1.51	-	-	-
CLOSED	72.58	11.32	9.12	1.73	1.07	2.79	-	-	-	1.39
N_2	71.17	11.77	8.55	1.86	0.79	2.27	1.75	-	-	1.84

Table 16. Results for the Rietveld Refinement of the batch-treated TCG formulations.

TCG Sample	Phase (wt%)											
	Amorphous	*Mullite*	*Quartz*	*Hematite*	*Magnetite*	*Calcite*	*Ettringite*	*Strätlingite*	*Hydrotalcite*	*Kuzelite*	*Portlandite*	*Hemicarbo-aluminate*
REF (Non-Leached)	78.40	6.06	5.44	1.22	0.14	1.14	0.91	Trace *	1.42	0.78	0.42	4.07
OPEN	77.85	6.55	5.64	1.30	0.22	5.37	-	Trace	2.67	0.41	-	-
CLOSED	78.92	5.48	5.28	1.29	0.15	3.38	-	Trace	3.07	1.75	0.08	-
N_2	79.66	6.64	5.96	1.44	0.24	1.30	-	Trace	2.78	1.77	0.20	-

* Trace is indicated for strätlingite because the peak is barely above background and its inclusion (at such low concentrations) for Rietveld refinement resulted in pattern simulation anomalies; hence it was omitted during Rietveld quantification.

CONCLUSIONS

SREL conducted a series of batch and column studies to address uncertainty in the realistic pH and E_h ranges associated with grouted waste tank systems, including an evaluation of three candidate TCG paste formulations. After curing for 90 days, the size reduced pastes were equilibrated under different atmospheric conditions, including open to the laboratory atmosphere (i.e., oxidizing), under a constant N_2 purge, and within an anaerobic Coy Chamber with anoxic conditions maintained by addition of a 95% N_2/5% H_2 gas mixture. The grout materials were equilibrated with pore water simulant for 150+ days, with pH and E_h monitored weekly and small aliquots of the pore waters collected for chemical analysis. Column tests were also performed in which the size-reduced TCG formulations were constantly leached under saturated conditions with the pore-water simulant that was either in equilibrium with the lab atmosphere or purged with UHP N_2 to reduce dissolved O_2 levels.

A summary of the solution chemistry results from the current set of experiments is provided in **Table 17**. The observed results were consistent with previous laboratory tests aimed at defining achievable E_h and pH conditions in tank waste grouted systems. The pH results were consistent with both the values derived from geochemical modeling and more recent laboratory testing. The TCG displayed the highest pH followed by the TCG-NBFS and finally the CLSM, with both the TCG and TCG-NBFS maintaining a higher pH (i.e., buffer the system) than the CLSM under all three test atmospheres. The observed E_h values, however, were less extreme than values used in WRMs to represent various stages in the aging of reducing tank closure grout. The lowest E_h values were observed for all samples equilibrated in the anaerobic Coy Chamber under a H_2/N_2 atmosphere, with the N_2 glovebag yielding the next lowest values and the batch samples open to the lab atmosphere yielding the highest E_h values. Even though there was a great deal of scatter in the data, the TCG materials containing the BFS generally provided the lowest E_h values (i.e., most reducing) under all batch and column test conditions, followed by TCG-NBFS and then CLSM.

The original dry feed materials, the three initially cured TCG paste formulations, and the TCG paste materials that were subjected to various batch treatments were extensively characterized by XRF and XRD analysis. The composition of the dry feed materials and the TCG paste formulations were consistent with previous analyses. Only minor changes to Na_2O and K_2O contents were observed for batch samples that had been subjected to extended leaching, but such changes were insufficient to correlate with any significant changes in mineralogy of the batch tested materials.

Irrespective of sample formulation or testing environment, XRD patterns were dominated by amorphous phases and unreacted quartz, mullite, hematite, and magnetite from the FA. In addition, all pre-leached samples indicated the presence of strätlingite, calcite, ettringite, and varied AFm carbonates and/or sulfates. Ettringite was not detected in any leached samples, and the AFm phases either disappeared in the leached samples or their proportions were significantly reduced. Strätlingite persisted to some degree in almost all of the leached samples irrespective of the leaching environment; however, it was barely detectable (and therefore not quantifiable) for the CLSM, TCG-NBFS, or TCG subjected to a reducing environment. In addition, for all TCG samples (containing BFS) strätlingite

was barely apparent above background irrespective of the leaching environment. Calcite persisted in all samples and the highest calcite proportions were detected for samples subjected to the oxic environment during leaching. These results were anticipated and presumed due to sample carbonation in the CO_2-containing oxic environment. Hydrotalcite and a phase tentatively identified as kuzelite (or monosulfoaluminate) were also observed but only in the BFS-containing TCG samples; both minerals persisted in all the leached TCG samples irrespective of leaching environment.

Table 17. Summary of batch equilibration results compared to geochemical modeling values for reducing grouts (SRNL-STI-2012-00404; SRR-CWDA-2016-00086).

	pH	Eh (volts)	Ca^{2+} (molar)	Na^{+} (molar)	Mg^{2+} (molar)	K^{+} (molar)
Leaching solution prescribed in SRNL-STI-2012-00404						
	4.68		2.1E-06	8.7E-06	1.3E-06	
Chemical Conditions of Reducing Grout Pore Water (SRNL-STI-2012-00404; SRR-CWDA-2016-00086)						
Red. Region II	11.1	-0.47	4.0E-03	1.0E-03		
Ox. Region II	11.1	0.56	4.0E-03	1.0E-03		
Ox. Region III	9.2	0.68	6.6E-05	1.0E-03		
Test Conditions	Current Study					
Batch Test - Open Atmosphere*	pH Range	Eh Range (Volts)	Ca^{2+} (molar)	Na^{+} (molar)	Mg^{2+} (molar)	K^{+} (molar)
TCG	11.1-12.6	0.12-0.26	2.0E-04	2.3E-04	3.6E-06	7.1E-04
TCG-NBFS	10.1-12.4	0.16-0.28	3.1E-04	2.2E-04	6.0E-06	8.2E-04
CLSM	9.2-11.9	0.20-0.35	3.5E-04	1.0E-04	1.1E-05	2.3E-04
Batch Test - N_2 Purged Atmosphere						
TCG	12.1-12.7	(-0.12)-0.18	ND	ND	ND	ND
TCG-NBFS	11.5-12.2	0.003-0.22	ND	ND	ND	ND
CLSM	10.8-11.8	0.02-0.27	ND	ND	ND	ND
Batch Test Coy Chamber - Reducing Atmosphere*						
TCG	11.6-12.8	(-0.42)-0.16	2.8E-04	1.3E-04	1.6E-06	4.3E-04
TCG-NBFS	11.0-12.4	(-0.36)-0.23	5.8E-05	2.3E-04	5.3E-07	1.2E-03
CLSM	9.22-12.1	(-0.45)-0.30	3.1E-04	1.1E-04	4.5E-06	3.4E-04
Column Test - Open Atmosphere**						
TCG	11.3-12.5	0.01-0.26	3.4E-04	6.0E-05	4.5E-07	7.3E-05
TCG-NBFS	10.9-12.3	0.13-0.35	7.7E-04	7.3E-05	1.8E-06	1.7E-04
CLSM	10.7-11.9	0.17-0.35	7.5E-04	5.6E-05	3.4E-06	8.8E-05
Column Test - N_2 Purged Atmosphere**						
TCG	11.3-12.5	(-0.03)-0.17	9.1E-04	6.5E-05	7.0E-07	1.1E-04
TCG-NBFS	11.0-12.1	0.10-0.25	8.0E-04	5.8E-05	1.4E-06	1.3E-04
CLSM	10.6-12.0	0.13-0.31	6.1E-04	4.9E-05	2.1E-06	1.0E-04

*Cation data for batch tests reflect the final solution after the "enhanced leaching" treatment.
**Cation data for column experiments reflect the final effluent composition.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the assistance of M. Shapiro and J. Lott in the lab.

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APPENDIX A: Manufacturer Certification Reports for Dry Feeds

Lehigh Grade 120 BFS

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LEHIGH
HEIDELBERGCEMENT Group

Material Certification Report

Brand Name: Lehigh Slag Cement
Material: GGBFS
Type: ASTM C989 Grade 120

Date 1-Feb-19
Silo # 611/612

General Information

Supplier: Lehigh Cement Company Address: 575 Cargo Road Cape Canaveral, FL 32920	Source Location: Lehigh Cement Company 575 Cargo Road Cape Canaveral, Florida 32920

The following information is based on monthly average test data. The data is typical of GGBFS shipped by Lehigh Cement Company, Cape Canaveral, FL Plant. Individual shipments may vary.

Test Data on ASTM "Standard" Requirements

Chemical (C989, Table 2)			Physical (C989, Table1)		
Item	Limit	Result	Item	Limit	Result
			45 µm (No. 325) Sieve (...)	-	0.4
			Blaine Fineness (m2/kg)	-	550
			Air Content (%)	12 max	2.1
Sulfide S (%)	2.5 max	0.9	Expansion in Water (C-1038) (%)	0.020 max	0.012
Sulfate Ion - SO_3 (%)	NA	3.2	Slag Activity Index (SAI %)		
			Average of Last 5 Samples:		
			Avg 7 Day Index	-	92
Aluminum Oxide - Al2O3 (%)	NA	13.8	Avg 28 Day Index	115 min	118
			Current Samples:		
			7 Day Index		91
			28 Day Index	110 min	116

Test Data on CCRL Reference Cement

Chemical			Physical		
Item	Limit	Result	Item	Limit	Result
Total Alkalies as Na_2O (%)	0.80 - 0.90	0.78	Blaine Fineness (m2/kg)	-	382
C_3S	-	57	Compressive Strength MPa (psi):		
C_2S	-	15	7 Day	-	4628
C_3A	-	7	28 Day	34.5 (5000) min	40.5 (5875)
C_4AF		8			

Optional Test Data

Chemical			Physical		
Item	Limit	Result	Item	Limit	Result
% Total Alkalies	-	0.42	Specific Gravity (Latest Result)	-	2.86
%Cl (Chloride)	-	<0.01	1 Day Accelerated (C-1073) psi	-	2770

Certification Statement

Lehigh Slag Cement meets Section 929-1 and 929-5 of FDOT Specifications. This product meets ASTM C989 Grade 120. This C989 slag cement is manufactured without the use of grinding agent as it is not required or beneficial. PO# SRR A078187.

Laboratory Manager: Inna Reed

SRRA078187R-4

SEFA Class F FA

TEC Services
Testing • Engineering • Consulting

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Client: Mr. Tom Hendrix
The SEFA Group
P.O. Box 6
Moncks Corner, SC 29461

Date: February 5, 2019
TEC Services I.D.: TEC 06-0509
Lab No.: 18-1378-WI

REPORT OF FLY ASH TESTS

Sample I.D. No.: WI113018
Manufacturer: Winyah Station (Thermally Beneficiated)

Date Sampled: November 30, 2018
Date Received: December 4, 2018

Chemical Analysis**	Results (wt%)	Specification (Class F) ASTM C618-08a	Specification (Class F) AASHTO M295-11
Silicon Dioxide (SiO_2)	53.0	----	----
Aluminum Oxide (Al_2O_3)	28.6	----	----
Iron Oxide (Fe_2O_3)	10.56	----	----
Sum of Silicon Dioxide, Iron Oxide & Aluminum Oxide ($SiO_2+Al_2O_3+Fe_2O_3$)	92.2	70 % min.	70 % min.
Calcium Oxide (CaO)	1.5	----	----
Magnesium Oxide (MgO)	1.1	----	----
Sodium Oxide (Na_2O)	0.34	----	----
Potassium Oxide (K_2O)	2.45	----	----
"Sodium Oxide Equivalent ($Na_2O+0.658K_2O$)"	1.95	----	----
Sulfur Trioxide (SO_3)	0.08	5 % max.	5 % max.
Loss on Ignition	0.2	6 % max.	5 % max.
Moisture Content	0.0	3 % max.	3 % max.
Total Chlorides	0.007	----	----
Available Alkalies**			
Sodium Oxide (Na_2O) as Available Alkalies	0.17	----	----
Potassium Oxide (K_2O) as Available Alkalies	1.31	----	----
Available Alkalies as "Sodium Oxide Equivalent ($Na_2O+0.658K_2O$)"	1.03	----	1.5 % max.*

Physical Analysis	Test Date			
Fineness (Amount Retained on #325 Sieve)	12/7/18	25.4%	34 % max.	34 % max.
Strength Activity Index (Using Lehigh Leeds Alabama Portland Cement)				
At 7 Days: Control Average, psi: 4790 / Test Average, psi: 3870	12/21/18	81%	75 % min.† (of control)	75 % min.† (of control)
At 28 Days: Control Average, psi: 6050 / Test Average, psi: 4860	1/11/19	80%	75 % min.† (of control)	75 % min.† (of control)
Water Requirements (Test H_2O/Control H_2O) Control, mls: 242 / Test, mls: 237	12/14/18	98%	105% max.† (of control)	105% max.† (of control)
Autoclave Expansion:	12/7/18	-0.03%	± 0.8 % max.	± 0.8 % max.

Uniformity Requirements				Test Date	Variation		
Specific Gravity:	2.34	Average:	2.34	12/4/18	-0.1%	5 % max. from average	5 % max. from average
% Retained #325 Sieve:	25.4	Average:	23.5	12/7/18	1.9%	5 % max. from average	5 % max. from average

† Meeting the 7 day or 28 day strength activity index will indicate specification compliance

* Optional

**Chemical Analysis performed on January 4, 2019.

The results of our testing indicate that this sample complies with ASTM C618-08a and AASHTO M295-11 (2015) specifications for Class F pozzolans.

Respectfully Submitted,
Testing, Engineering & Consulting Services, Inc.

Dean Roosa
Project Manager

Shawn McCormick
Laboratory Principal

ISO 17025 ACCREDITED LABORATORY
US Army Corps of Engineers
Testing, Engineering & Consulting Services, Inc.
235 Buford Drive | Lawrenceville, GA 30046
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AASHTO R18
ASTM INTERNATIONAL

Holcim Type I/II Cement

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Holcim NSF

Material Certification Report

Material: Portland Cement
Type: I-II (MH)
Test Period: 17-Jan-2019 to 17-Jan-2019
Date Issued: 14-Feb-2019

Certification

This cement meets the specifications of ASTM C150 and AASHTO M85 for Type I-II (MH) cement.

General Information

Supplier:	Holcim (US) Inc. d/b/a LafargeHolcim US	Source Location:	Holly Hill Plant Silo: 25
Address:	8700 West Bryn Mawr Ave Chicago, IL 60631		2173 Gardner Blvd Holly Hill, SC 29059
Contact:		Contact:	Scott Poaps / (803) 496-2995

The following is based on average test data during the test period. The data is typical of product shipped from this source; individual shipments may vary.

Test Data on ASTM Standard Requirements

Chemical			Physical		
Item	Limit [1]	Result	Item	Limit [1]	Result
SiO_2 (%)	-	19.4	Air Content (%)	12 max	6
Al_2O_3 (%)	6.0 max	4.6	Blaine Fineness (m^2/kg)	260-430	396
Fe_2O_3 (%)	6.0 max	3.3			
CaO (%)	-	63.1	Autoclave Expansion (%) (C151)	0.80 max	0.02
MgO (%)	6.0 max	1.3	Compressive Strength MPa (psi)		
SO_3 (%) [2]	3.0 max	3.3	3 day	10.0 (1450) min	28.8 (4180)
Loss on Ignition (%) [4]	3.5 max	2.3	7 day	17.0 (2470) min	34.3 (4970)
Insoluble Residue (%)	1.50 max	0.36			
CO_2 (%)	-	1.3	Initial Vicat (minutes)	45-375	80
$CaCO_3$ in Limestone (%)	70 min	84			
Potential Phase Compositions [5]:			Mortar Bar Expansion (%) (C1038)	0.020 max	0.009
C_3S (%)	-	58			
C_2S (%)	-	12			
C_3A (%)	8 max	7			
C_4AF (%)	-	10			
$C_3S + 4.75C_3A$ (%)	100 max	90			

Test Data on ASTM Optional Requirements

Chemical			Physical		
Item	Limit [1]	Result	Item	Limit [1]	Result
Equivalent Alkalies (%)	0.60 max	0.47	Heat of Hydration kJ/kg (cal/g) (ASTM C1702) 3 Days [4]	-	276 (66)

Notes (*1-9)

1 - Dashes in the Limit / Result columns mean Not Applicable.
2 - It is permissible to exceed the specification limit provided that ASTM C1038 Nortar Bar Expansion does not exceed 0.020% at 14 days.
3 - Adjusted per Annex A1.6 of ASTM C150 and AASHTO M85.
4 - Test results represent the most recent value and is provided for information only.
5 - Limit = 3.0 when limestone is not an ingredient in the final cement product
1/17/2019
Grind 17
This cement was ground with the proper amount of gypsum and grinding aid.

PO# 5RRA078919R-3

Additional Data

Item	Limestone	Inorganic Processing Addition	Base Cement Phase Composition	Result
Amount (%)	3.6	-	C_3S (%)	60
SiO_2 (%)	2.3	-	C_2S (%)	12
Al_2O_3 (%)	0.6	-	C_3A (%)	7
Fe_2O_3 (%)	0.5	-	C_4AF (%)	10
CaO (%)	51.3	-		
SO_3 (%)	0.1	-		

Printed: 2/14/2019 1:59:41 PM
Version: 180412

Scott Poaps
Scott Poaps,
Quality Manager

APPENDIX B: XRD Analysis of Tank Closure Grouts

APPENDIX B: XRD Analysis of Tank Closure Grouts

X-ray diffraction data is presented for the following sample types:

1. The BFS, FA, and cement (OPC) that were used to prepare the tank closure pastes for the batch leaching experiments. These are analyzed since they are the starting point for all the tank closure paste mixes and some of the phases present in these materials will likely persist in the hydrated samples.
2. Hydrated tank closure pastes that had ***not*** been subjected to batch leaching treatments; these were used as reference samples to indicate the pre-treatment mineralogy.
3. Tank closure pastes subjected to batch leaching treatments under OPEN (oxic), CLOSED (reducing), and anoxic (N_2) environments.

Before presenting the XRD data it is important to acknowledge some complexities that exist with respect to phase identification and quantification when analyzing XRD scans for multicomponent systems. Such complexities are not necessarily associated with analysis of the dry feeds because those systems have, in general, been well-studied, partial information about the physical and chemical make-up of the samples is available from the manufacturer, and their chemistry and/or mineralogy is, to some degree, controlled by industrial standards. However, there are limitations that exist (with respect to phase identification and ultimately quantification) when analyzing unknown systems, such as the hydrated and leached samples from this study; these include:

1. **Peak Overlap** – different phases present in a sample may exhibit peaks at similar 2θ diffraction angles. As such, the individual peaks are not discrete and the XRD indicates a peak with shape and intensity matching the sum of the individual peaks (refer to **Figure B-1**). For abundant phases (>10 wt%), peak overlap is often relatively easy to detect but overlap of trace (<2 wt%)) or minor (<10 wt%) phases (especially if the overlapping peak is associated with a major phase) can make definitive phase identification difficult.

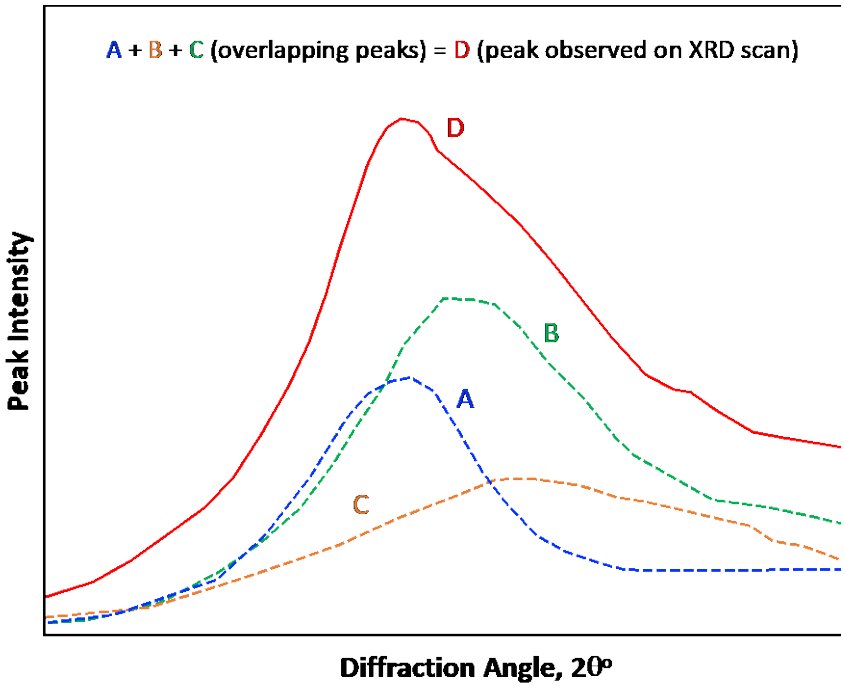


Figure B-1: Peak overlap from different phases in XRD scans.

2. **Peak Shift** – XRD phase identification of multiphase samples is achieved by comparing the XRD scan for the material of interest with verified scans for individual phases from a crystallographic database, such as the International Centre for Diffraction Data (ICDD). Matching is partly achieved by comparing the peak positions in the measured sample with the peak positions of the verified database material. Note that the peak positions are dependent on crystallographic structure, in particular interplanar spacing (refer to https://serc.carleton.edu/research_education/geochemsheets/BraggsLaw.html). However, the database entries are not limitless and phases in the measured sample may differ from the database entries due to slight differences in stoichiometry, anion and cation substitutions, and water content in the case of cementitious/pozzolanic materials. All of these factors can influence the crystal structure and the interplanar spacings, which may result in the characteristic peak shift illustrated in **Figure B-2**.

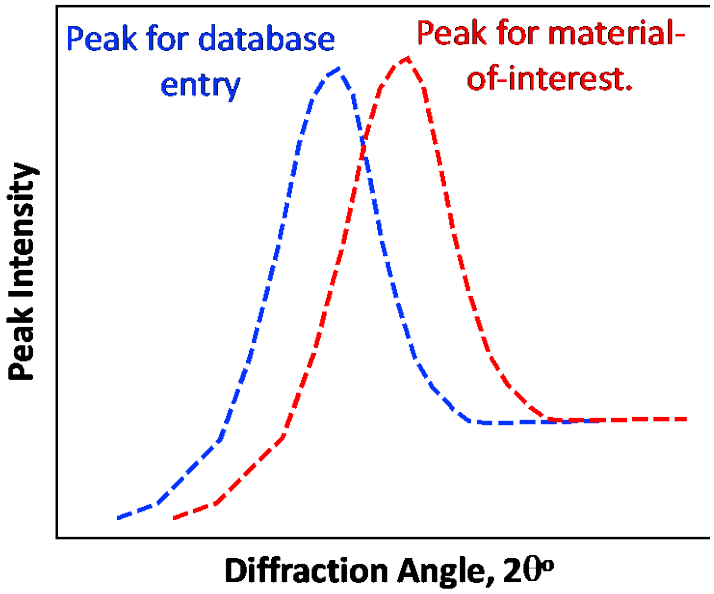
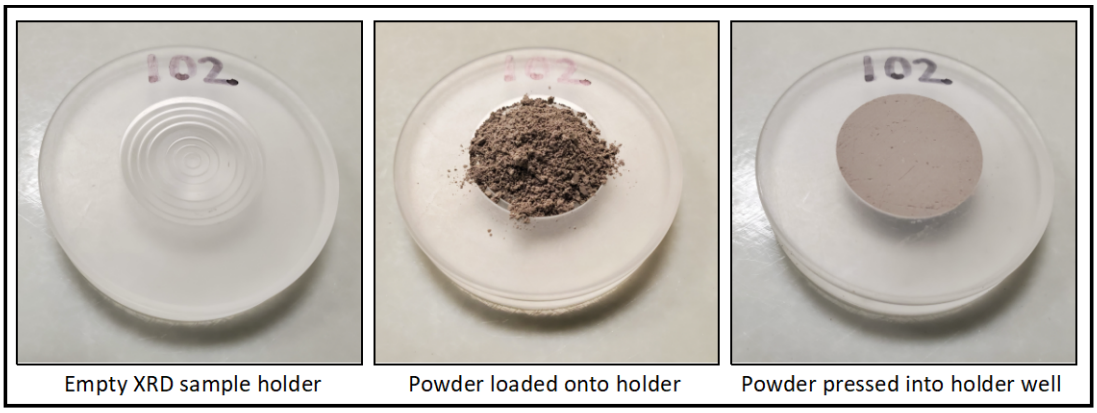


Figure B-2: Peak shift between material-of-interest and crystallographic database entry.

3. **Peak Intensity** – in addition to peak position (or diffraction angle 2θ), relative peak intensity is also used in the identification of unknown phases. Each peak is based on the diffraction of X-rays from a particular atomic plane within the crystal structure. Powder XRD relies on the sample being composed of millions of randomly orientated grains that are ideally <10 μm in diameter, and this helps to ensure that all the crystal planes are randomly orientated and, additionally, that the most abundant planes within the crystal are characterized as such on the XRD scan. If, however, one crystal plane is preferentially orientated with respect to the X-ray incident beam, then that plane will be indicated as the highest intensity peak on the XRD scan though it may not be the most abundant plane in the crystal system. Some materials are in the form of platelets and these can be orientated during XRD sample preparation (refer to **Figure B-3**). As the powder samples are pressed into the well of the XRD sample holder, plate-like crystals can be forced to lie in a particular or preferred orientation rather than being randomly orientated. The preferred orientation results in a specific crystallographic plane becoming orientated towards the XRD incident beam, and the diffraction peak intensity associated with that crystallographic plane is enhanced. With respect to this study, it is noteworthy that both ground gypsum and portlandite exhibit plate-like morphologies and are subject to preferred orientation.



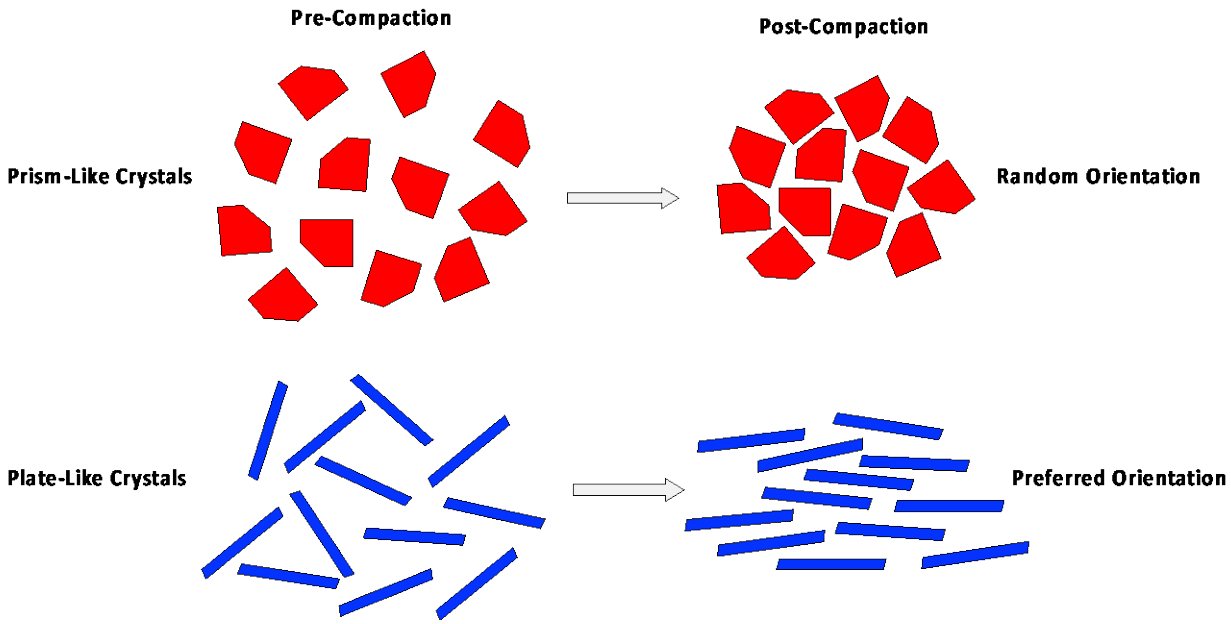


Figure B-3: Preferred orientation of plate-like crystals.

4. **Phase Proportion** – as the proportion of a phase in a multicomponent system decreases, it becomes more difficult to identify since the lower-intensity characteristic peaks are absent. Strategies available to evaluate the overlap of two abundant phases are not necessarily applicable if a trace/minor phase is overlapped by a more abundant phase. In addition, many of characteristics peaks of trace/minor phases may be masked by background noise. Hence, for trace and minor phases the analyst may be relying on a single peak to identify a given phase; this is problematic unless the analyst has prior knowledge of the system and expectations of the phases that should be present.

5. **Phase Structure Information** – in order to conduct phase quantification, a phase must first be identified; identification is, of course, dependent on the previously mentioned complexities of XRD analysis (i.e., Items 1-4). In addition, while it may be possible to identify a phase via comparison with verified patterns from a crystallographic database, the database may not contain the structure information that is needed to conduct quantification

via Rietveld refinement. This topic is beyond the scope of this report but it should be noted that a number of phases have been tentatively identified in the hydrated/leached samples, but the database information was ultimately missing the structure data required for quantification. Additional information regarding the fundamentals of Rietveld refinement is provided at http://profex.doebelin.org/wp-content/uploads/2015/02/Lesson-1-XRD-and-Rietveld-Refinement.pdf.

DRY FEEDS XRD DATA

Lehigh Grade 120 BFS

Figure B-4 (A-C) indicates the XRD data for the Lehigh Grade 120 BFS used to prepare the samples in this study. **Table B-1** also indicates the phases observed, the 2θ diffraction angles at which peaks were discernible, the ICDD powder diffraction file (PDF) for each phase, and additional pertinent information. In addition, the ICDD PDF data sheets for each phase are provided in **Appendix D**.

Table B-1: Phases observed in BFS.

PHASE	OBSERVED PEAKS (2θ)	ICDD PDF	COMMENTS
Gypsum *Calcium Sulfate Dihydrate* $CaSO_4•2H_2O$	11.63° 20.72° 29.09° 31.14° 33.37°	04-015-8262	• Additive to BFS to enhance reactivity. • Indicates preferred orientation (texture) at [h k l] = [0 2 0] (2θ = 11.63°).
Calcite (Limestone) *Calcium Carbonate* $CaCO_3$	23.08° 29.41°	01-078-4614	• Additive to BFS to enhance grinding.
β-Quartz *Silicon Dioxide* SiO_2	26.25°	00-047-1144	• Potential quartz carryover from smelting process.
Quartz *Silicon Dioxide* SiO_2	26.63°	00-046-1045	• Potential quartz carryover from smelting process.

The XRD scan (**Figure B-4 (A)**) is characterized by well-defined peaks associated with either crystalline gypsum ($CaSO_4•2H_2O$), limestone (calcite – $CaCO_3$), and the 10 wt% NIST Si standard (which is added for peak alignment and as an internal standard for phase quantification). Lehigh adds 2-3 wt% each of gypsum and limestone as an activator and grinding aid, respectively. The scan is also characterized by an "amorphous hump" that correlates with the glassy (amorphous) BFS phases. Amorphous materials, such as BFS aluminosilicate glasses, have no long-range atomic order and produce a broad low intensity hump, rather than the high-intensity, well-defined diffraction peaks associated with the long-range atomic order of crystalline materials. Thus, with XRD it is not possible to determine any phase information for the amorphous portion of BFS. Magnification of the XRD scan (**Figure B-4 (B)**) reveals two trace peaks between 26-27° 2θ; these have tentatively been identified as two quartz polymorphs. Any quartz present during the iron smelting process will ultimately be contained in the slag byproduct. One final point of note is

indicated in **Figure B-4 (C)**. With respect to XRD, crystalline materials are characterized by peaks which exhibit specific 2θ diffraction angles and specific relative intensities; each peak is associated with diffraction from a specific crystal plane denoted by unit cell coordinates [h k l] (refer to https://www.doitpoms.ac.uk/tlplib/miller_indices/printall.php regarding crystal plane designations). As shown in **Figure B-4 (C)**, the relative peak intensities of the ICDD reference sample are denoted by the vertical blue lines, but the peak intensity associated with the [0 2 0] plane in the measured sample is three times larger than the reference sample. This anomaly is referred to as preferred orientation or texture. Gypsum, when ground during the BFS manufacturing process, forms flat, plate-like crystals that tend to lie on the flat crystal faces (**Grattan-Bellew, 1975**) particularly, as discussed in the earlier text, during sample preparation for XRD analysis in which powders are pressed into shallow sample holder wells (refer to **Figure B-3**). Thus, sample preparation preferentially orientates the [0 2 0] gypsum plane to the incident X-ray beam, which produces the highest intensity peak.

BFS phase proportions (determined by the internal standard Rietveld method) are provided in **Table B-2**. These proportions are in line with those reported in **SREL DOC. No. R-20-0001**, which detailed the compositions of multiple BFS batches previously utilized at the Savannah River Site (SRS). The GOF value refers to the "Goodness of Fit" between the XRD pattern for the actual sample and the simulated pattern. The relevance of GOF and other pattern-simulation discrepancy values are discussed by **Toby (2006)**, though as the author notes, GOF does not provide a clear basis for rejecting or accepting a simulated pattern. In this study, the GOF values are provided merely as a means of comparison to indicate deviations between the actual and simulated scans. Discrepancies between the measured and simulated patterns can be visualized when using the Rietveld refinement software (i.e., DIFFRAC-TOPAS for this study). **Figure B-5** shows the measured and simulated patterns for the BFS used in this study, and indicates that pattern discrepancy is predominantly associated with the peaks for the NIST Si standard. DIFFRAC-TOPAS does include a number of options for enhancing pattern fit and the authors evaluated a number of said options in addition to consulting the XRD Applications Team at Bruker Nano Inc. (Madison, WI). Ultimately, the simulated pattern provided an acceptable fit to the measured data, and the observed minor discrepancies are not thought to impact the validity of the phase quantification data.

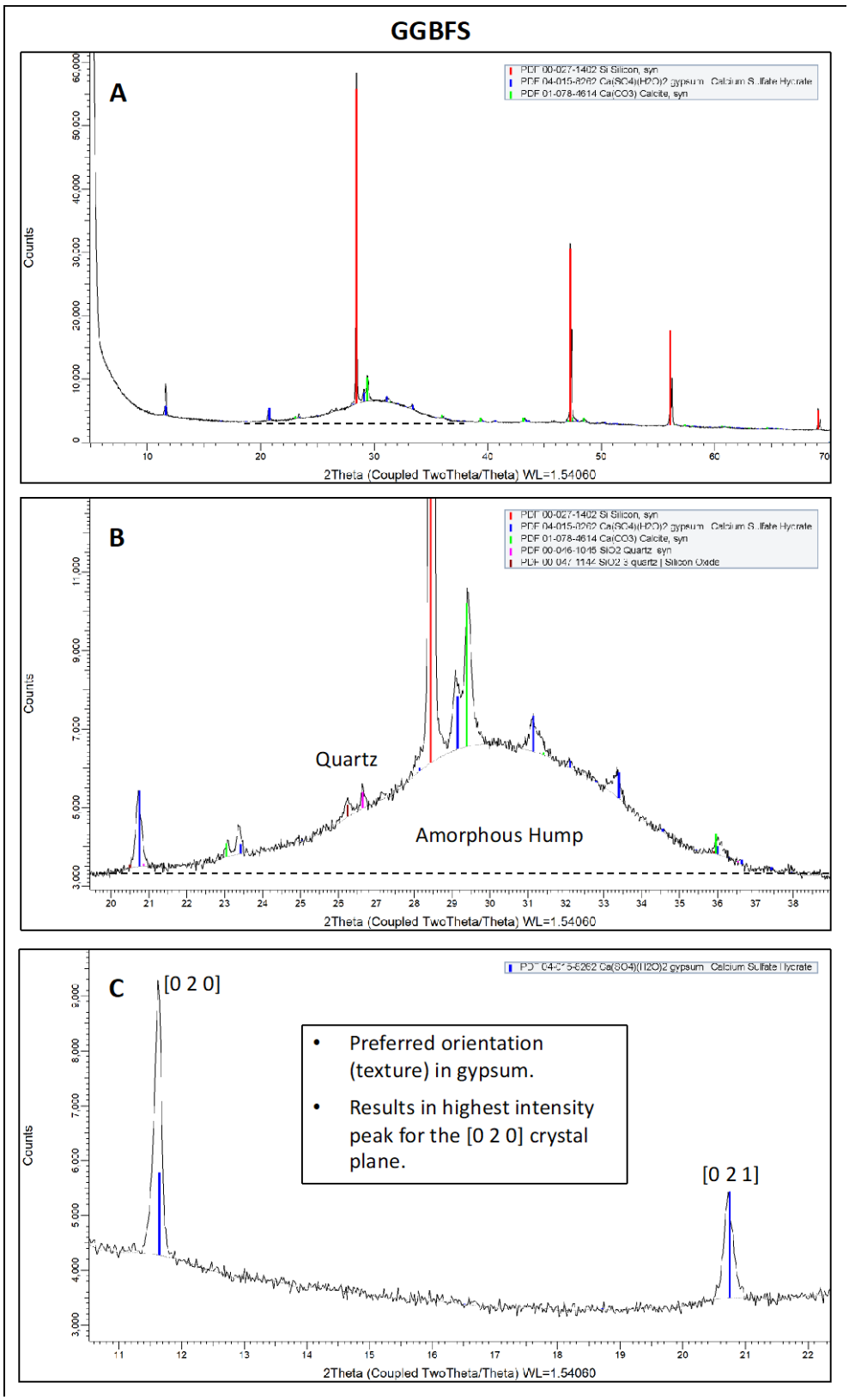


Figure B-4: XRD scan for BFS used to produce study samples (A = complete scan; B & C = magnified portions of scan).

Table B-2: Crystalline and amorphous phase wt% in BFS.

Batch ID	Phase (wt%)				GOF
	Amorphous	*Gypsum*	*Calcite*	*Quartz***	
2019-IR-05-0261*	95.63	2.15	2.22	Trace	2.43

* The MCR for BFS is provided in **Appendix A**.

** The quartz polymorphs evident in **Figure B-4 (B)** were barely above background and not included in Rietveld refinement.

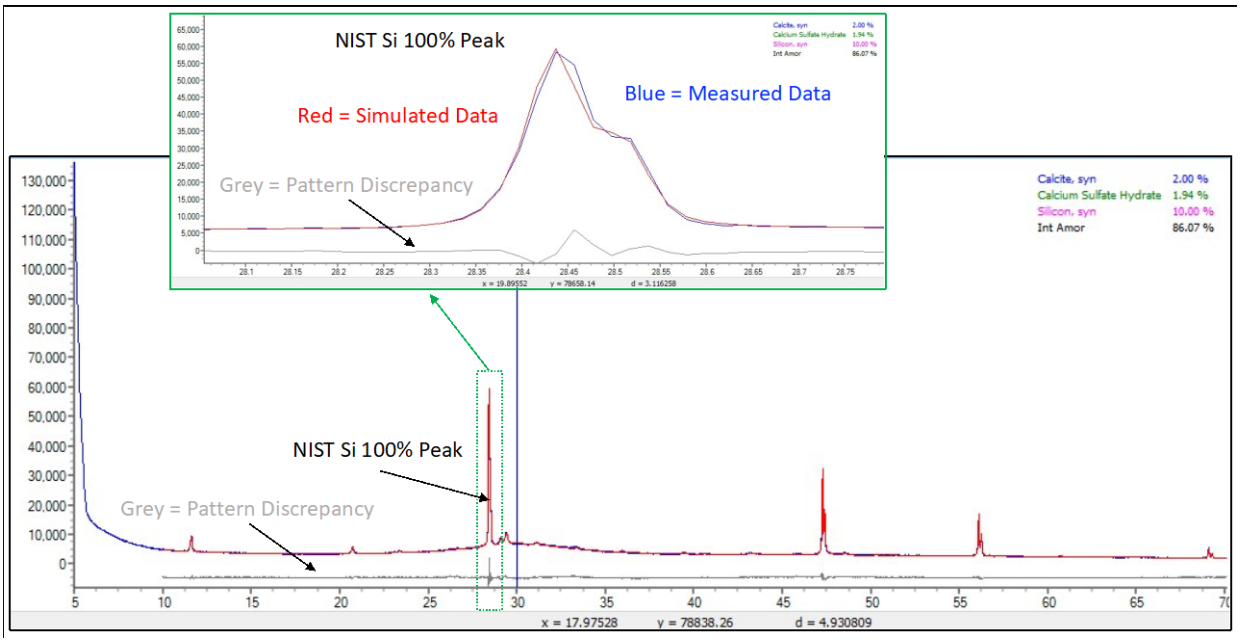


Figure B-5: Rietveld refinement data illustrating discrepancies between measured and simulated patterns for BFS.

SEFA Class F FA

Figure B-6 indicates the XRD scan for the SEFA Class F FA used in the preparation of samples for this study. Information regarding the diffraction angles at which each phase was observed and the matching phase from the ICDD are provided in **Table B-3**. In addition, the data sheets for each ICDD PDFs are provided in **Appendix D**.

FA is a semi-amorphous silicate glass containing multicomponent oxides of silicon, aluminum, iron, and calcium, and the FA scan is characterized by crystalline peaks for mullite ($3Al_2O_3•2SiO_2$), quartz (SiO_2), hematite (Fe_2O_3), and magnetite (Fe_3O_4), all of which are identified in **ACI 232.2R, *Report on the Use of Fly Ash in Concrete***, as potential FA mineral phases. The amorphous portion of the FA (characterized by the "amorphous hump in **Figure B-6 (B)**) is

susceptible to pozzolanic activity but the crystalline phases are considered low activity components; refer to **SREL DOC. No. R-20-0001** for additional information on FA activity. The crystalline and amorphous phase proportions are indicated in **Table B-4**. Discrepancies between the measured XRD pattern and the Rietveld simulated pattern are presented in **Figure B-7**. Similar to BFS the main discrepancy is for the NIST Si peaks but the FA also indicates discrepancies for the quartz phase. Ultimately, the discrepancies observed in the data are relatively insignificant and the quantification numbers are considered valid.

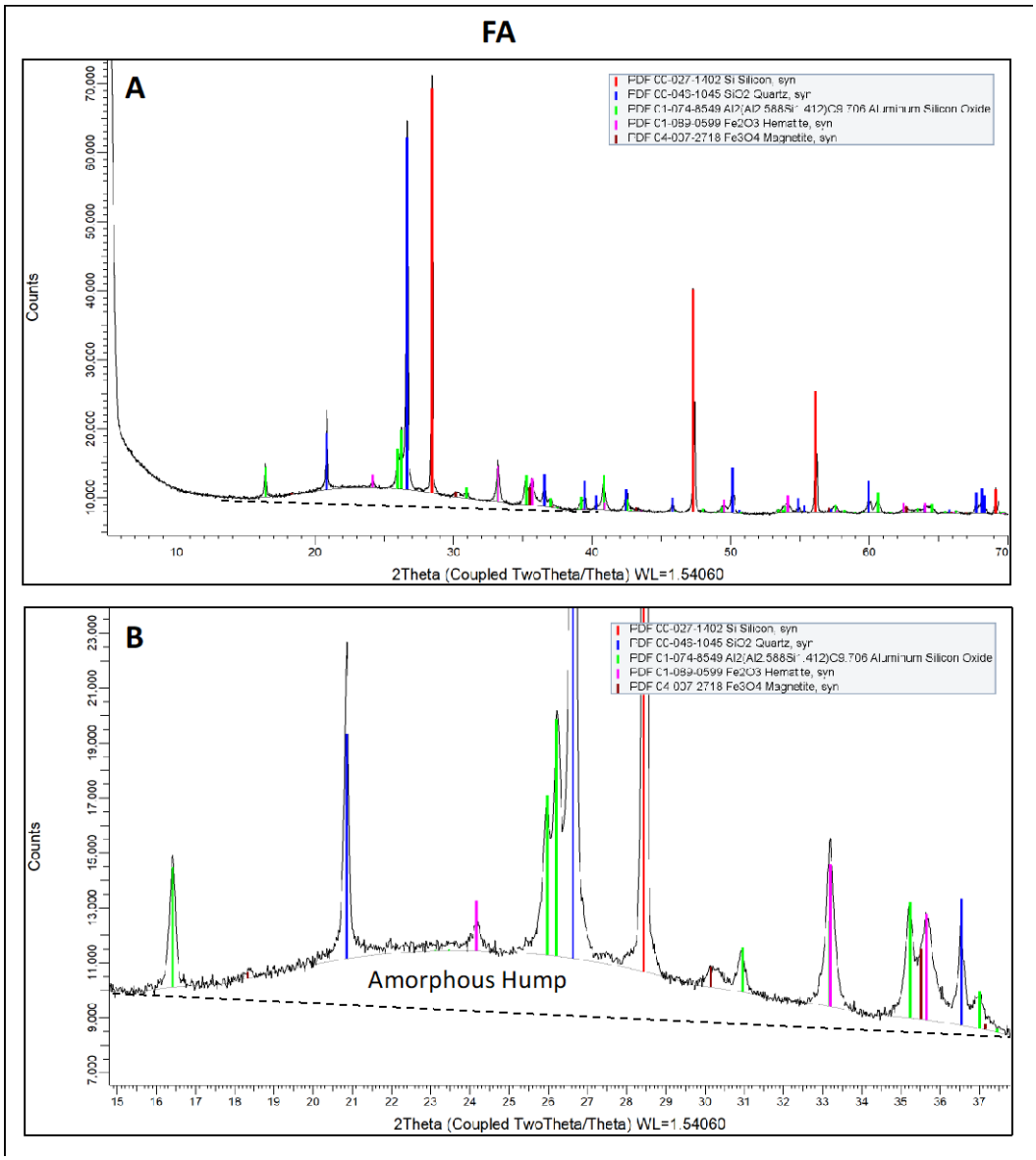


Figure B-6: XRD scan for FA used to produce study samples (A = complete scan; B = magnified portion of scan).

Table B-3: Phases observed in FA.

PHASE	PROMINENT PEAKS (2θ)	ICDD PDF	COMMENTS
Mullite *Aluminum Silicate* $3Al_2O_3•2SiO_2$	16.42° 25.96° 26.23° 35.21° 40.81°	01-074-8549	• Primary phase in low-Ca FA.
Quartz *Silicon Dioxide* SiO_2	20.85° 26.63° 50.11°	00-046-1045	• Primary phase in low-Ca FA.
Hematite *Iron (III) Oxide or Ferric Oxide* Fe_2O_3	24.17° 33.19° 35.66°	01-089-0599	• Primary phase in low-Ca FA.
Magnetite *Iron (II,III) Oxide or Ferrous-Ferric Oxide* Fe_3O_4	30.21°	04-007-2718	• Potential phase in low-Ca FA.

Table B-4: Crystalline and amorphous phase wt% in FA.

Batch ID	Phase (wt%)					GOF
	Amorphous	*Mullite*	*Quartz*	*Hematite*	*Magnetite*	
2019-IR-05-0195*	63.93	17.62	13.34	2.50	2.61	2.08
* The MCR for FA is provided in **Appendix A**.						

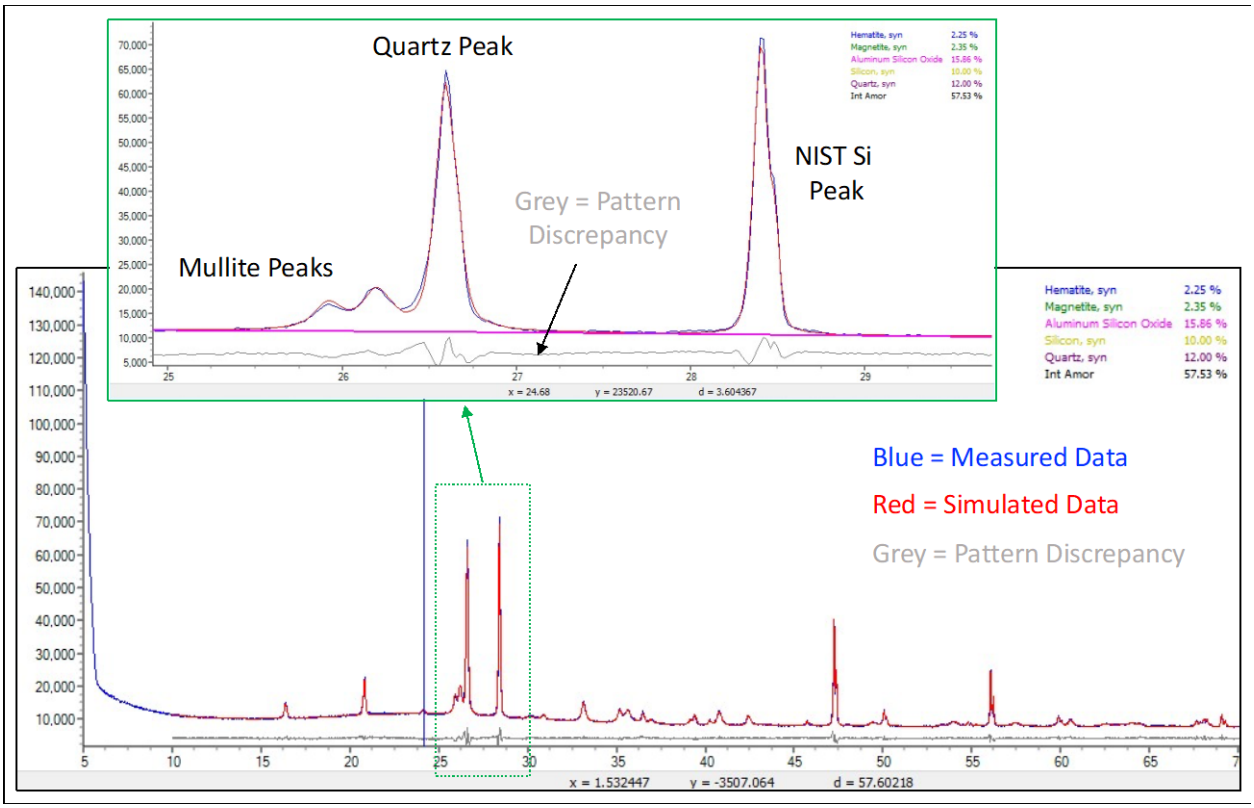


Figure B-7: Rietveld refinement data illustrating discrepancies between measured and simulated patterns for FA.

Holcim Type I/II Cement

Figure B-9 indicates the XRD scan for the cement used in the preparation of grout samples for this study. Information regarding the diffraction angles at which each phase was observed and the matching phase from the ICDD database are provided in **Table B-5**. In addition, the data sheets for each ICDD PDF are provided in **Appendix D**.

The typical (and anticipated) cement phases (per **ACI 225R, *Guide to the Selection and Use of Hydraulic Cements***) are alite (Ca_3SiO_5), belite (Ca_2SiO_4), tri-calcium aluminate ($Ca_3Al_2O_6$), and tetracalcium aluminoferrite (Ca_2AlFeO_5). In addition, gypsum ($CaSO_4•2H_2O$), bassanite (hemihydrate - $CaSO_4•0.5H_2O$), limestone (calcite – $CaCO_3$), and trace ettringite ($Ca_6Al_2S_3O_{18}•32H_2O$) and portlandite ($Ca(OH)_2$) are also observed (refer to **Figure B-8 (C and D)**). Gypsum is added to OPC as a set retarder and this mineral can actually lose water to form bassanite at the elevated temperatures that may occur during the clinker grinding process (**Kilic et al, 2007; Pott et al., 2020**). Limestone (calcite) is also added to the Holcim cement as a filler material and a grinding aid (**Ingram et al., 1991**). Portlandite and ettringite (**Figure B-8 (D)**) are reaction products of cement hydration and indicate that the cement likely underwent mild hydration due to air-moisture contact during storage. The cement phase proportions are identified in **Table B-6**. The main cement phases are also measured by the manufacturer and presented in

the cement MCR in **Appendix A** and **Table B-6**. The alite and belite are in fairly good agreement but the vendor values for tri-calcium aluminate (typically termed *aluminate*) and tetra-calcium aluminoferrite (typically termed *ferrite*) are significantly different. Since the peaks for aluminate and ferrite in the measured sample are very discrete and not susceptible to significant overlap with other phases, the authors are confident in the proportions presented in **Table B-6**. It is conceivable, of course, that a multiple-ton batch of cement will exhibit heterogeneity, and such heterogeneities would be expected to manifest when analyzing gram-sized XRD samples from ton-sized batches of material. The Rietveld discrepancy plot is presented in **Figure B-9**, and indicates more phase discrepancies (confirmed by the GOF = 3.65 in **Table B-6**), which is not surprising since the cement is a multi-phase material subject to significant peak overlap between phases. However, the figure also demonstrates that ultimately the simulated and measured patterns are well matched.

Table B-5: Phases observed in cement.

PHASE	OBSERVED PEAKS (2θ)	ICDD PDF	COMMENTS
Ettringite *Calcium Sulfoaluminate* $3CaO.Al_2O_3.3CaSO_4.32H_2O$	9.11°	04-013-3691	• AFt phase produced via the early reaction between gypsum ($CaSO_4•2H_2O$) (added to cement) and tricalcium aluminate ($3CaO•Al_2O_3$).
Gypsum *Calcium Sulfate Dihydrate* $CaSO_4.2H_2O$	11.65° 20.73° 29.15°	04-015-8262	• Additive to cement to reduce flash setting. • Indicates preferred orientation (texture) at [h k l] = [0 2 0] (2θ = 11.65°).
Bassanite *Calcium Sulfate Hemihydrate* $CaSO_4.0.5H_2O$	14.74° 25.65° 29.68°	00-041-0224	• Gypsum added to cement can dehydrate due to heat associated with milling cement clinker.
Alite (Hatrurite) Tricalcium Silicate Ca_3SiO_5	14.90° 25.19° 32.15° 32.53° 34.31°	00-055-0738	• Primary phase in cement.
Portlandite *Calcium Hydroxide* $Ca(OH)_2$	18.02°	00-044-1481	• Portlandite is a principal hydration product of cement resulting from the reactions of alite and belite with water. • Possible preferred orientation (texture) at [h k l] = [0 0 1] (2θ = 18.02°).
Ferrite *Tetracalcium Aluminoferrite* $Ca_2(Al,Fe)_2O_5$	24.36° 33.53° 33.90°	04-008-6822	• Primary phase in cement.
Calcite (Limestone) *Calcium Carbonate* $CaCO_3$	29.35°	01-078-4614	• Additive to cement as a filler/grinding aid. • Main (100%) calcite peak overlapped by alite.
Aluminate *Tricalcium Aluminate* $Ca_3Al_2O_6$	21.82° 26.65° 33.22°	00-006-0495	• Primary phase in cement.
Belite (Larnite) *Dicalcium Silicate* Ca_2SiO_4	31.02° 26.65° 33.22°	01-080-8935	• Primary phase in cement. • Most belite peaks are severely overlapped by alite.

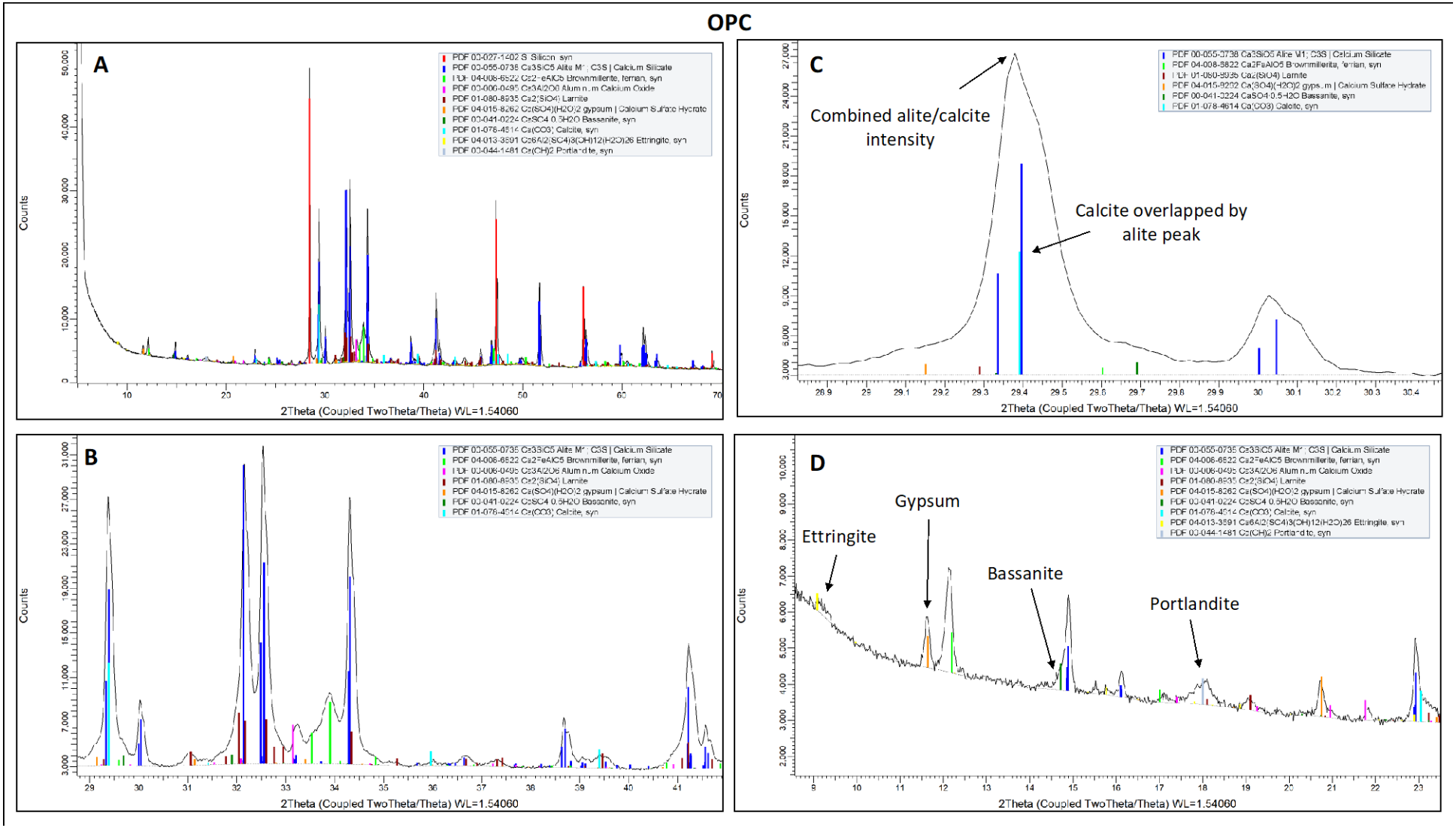


Figure B-8: XRD scan for cement used to produce study samples (A = complete scan; B, C & D = magnified scan portions).

Table B-6: Crystalline phase wt% in cement.

Batch ID	Analysis Source	Phase (wt%)									GOF
		Alite	*Belite*	*Aluminate*	*Ferrite*	*Gypsum*	*Bassanite*	*Calcite*	*Portlandite*	*Ettringite*	
2019-IR-05-0201*	This Study	64.06	10.67	2.17	15.12	2.24	1.55	3.28	0.90	Trace **	3.58
	MCR	58	12	7	10	-	-	-	-	-	N/A

* The cement MCR is provided in **Appendix A**.

** Trace is indicated for ettringite because the peak is barely above background and its inclusion (at such low concentrations) in the Rietveld refinement resulted in pattern simulation anomalies; hence it was omitted during Rietveld quantification.

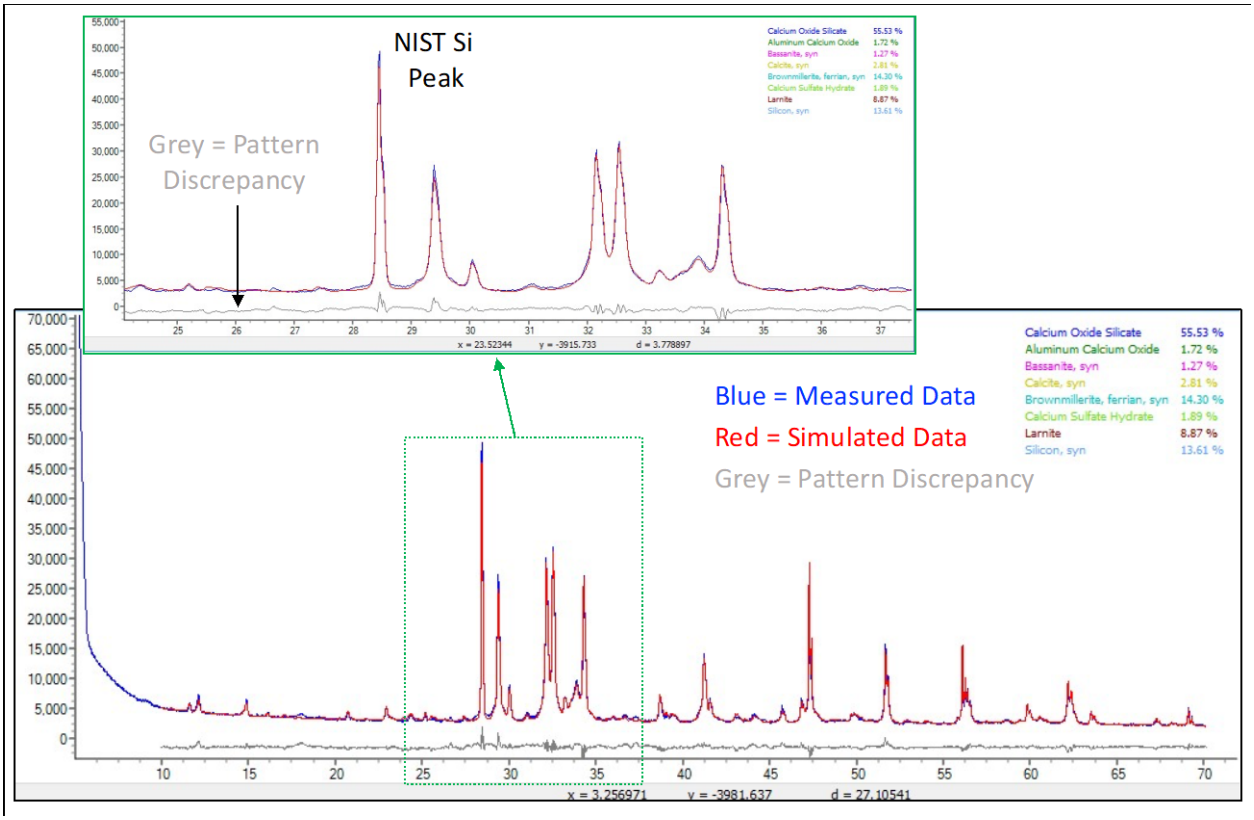


Figure B-9: Rietveld refinement data illustrating discrepancies between measured and simulated patterns for cement.

TANK CLOSURE PASTES

The tank closure paste samples used in batch testing contain the following dry cementitious material proportions (**Table B-7**); this information is relevant regarding the phases formed during hydration/pozzolanic reactions and because of the residual unreacted phases (from each dry feed) that may persist in the scans of hydrated/leached samples.

Table B-7: Dry feed component proportions in tank closure grouts.

Tank Closure Grout Sample	Component (wt%)		
	Lehigh Grade 120 GBFS	SEFA Class F FA	Holcim Type I/II Cement
CLSM	0	92.3	7.7
TCG-NBFS	0	82.1	17.9
TCG	30.1	52.0	17.9

The phases identified (or tentatively identified) in the various tank closure paste samples are indicated in **Table B-8**, which also indicates the 2θ diffraction angles at which peaks were discernible, the best-matching ICDD PDF, and the specific samples in which each phase was observed. The reader should also refer to **Appendix C**, which includes more detailed information regarding the rationale for the appearance of the indicated phases and **Appendix D**, which contains the ICDD PDF data sheets for each phase. Note also that many of the technical references regarding the observed phases are indicated in the tables of **Appendix C** rather than directly in this text. Regarding the certainty of phase identification, in **Table B-8** mineral phases are marked with a confidence level of HIGH, MEDIUM, and LOW. It should be noted that the assigned confidence levels are somewhat arbitrary and highly dependent on the analyst's knowledge and perception of the data. However, the authors have spent considerable time analyzing the data, in addition to reviewing research data for similar systems to provide rationales for inclusion of a particular phase. Phases marked LOW or MEDIUM are thus marked because they suffer from one or more of the following deficiencies:

1. Trace phase barely above background.
2. Phase may exhibit only one observable peak.
3. Peak shift compared to the ICDD pattern.
4. Significant peak overlap (especially between 10.6° and 11.6° 2θ).
5. The authors were unable to find previous research studies that specifically identified the same, or similar, phases associated with cement/FA and cement/FA/BFS systems.

Table B-8: Phases identified via XRD analysis of tank closure grouts.

PHASE	OBSERVED PEAKS (2θ)	ICDD PDF	SAMPLES OBSERVED IN			CONFIDENCE LEVEL
			CLSM	TCG-NBFS	TCG	
Strätlingite $Ca_2Al_2SiO_7.8H_2O$	7.09° 14.15° 21.35°	00-029-0285	• CLSM-REF • CLSM-OPEN • CLSM-CLOSED • CLSM-N_2	• TCG-NBFS-REF • TCG-NBFS-OPEN • TCG-NBFS-N_2	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2	HIGH
Ettringite $Ca_6Al_2(SO_4)_3(OH)_{12}(H_2O)_{26}$	9.11° 15.80° 18.94° 22.97°	04-013-3691	• CLSM-REF	• TCG-NBFS-REF	• TCG-REF	HIGH
Kuzelite $Ca_2Al(SO_4)_{0.5}(OH)_6(H_2O)_3$	9.92°	04-013-3303	• Not Observed	• Not Observed	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2	MEDIUM
Calcium Iron Oxide Sulfite Hydrate $Ca_4Fe_2O_6(SO_3).12H_2O$	10.52°	00-044-0448	• Not Observed	• Not Observed	• TCG-REF • TCG-CLOSED • TCG-N_2	LOW
Calcium Aluminum Silicate Hydrate $CaAl_2Si_7O_{18}.1.7H_2O$	10.66° 21.29°	00-021-0132	• Not Observed	• TCG-NBFS-REF	• Not Observed	LOW
Calcium Aluminum Oxide Carbonate Sulfate Hydroxide Hydrate $3CaO.Al_2O_3.0.17CaSO_4.0.5Ca(OH)_2.0.33CaCO_3.xH_2O$	10.70°	00-060-0312	• CLSM-REF	• Not Observed	• TCG-REF • TCG-OPEN	LOW
Calcium Aluminum Carbonate Hydroxide Hydrate (Hemicarboaluminate) $Ca_2Al(CO_3)_{0.25}(OH)_{6.5}(H_2O)_2$	10.80° 21.71° 30.99°	04-018-9908	• CLSM-REF	• TCG-NBFS-REF	• TCG-REF	MEDIUM
Calcium Aluminum Iron Oxide Carbonate Hydroxide Hydrate $Ca_8Al_2Fe_2O_{12}CO_3(OH)_2.22H_2O$	11.06°	00-045-0572	• CLSM-REF	• TCG-NBFS-REF • TCG-NBFS-CLOSED • TCG-NBFS-N_2	• TCG-OPEN • TCG-CLOSED • TCG-N_2	LOW

PHASE	OBSERVED PEAKS (2θ)	ICDD PDF	SAMPLES OBSERVED IN			CONFIDENCE LEVEL
			CLSM	TCG-NBFS	TCG	
Hydrotalcite $Mg_{0.67}Al_{0.33}(CO_3)_{0.17}(OH)_2(H_2O)_{0.5}$	11.60°	04-015-4253	• Not Observed	• Not Observed	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2	MEDIUM
Calcium Aluminum Carbonate Hydroxide Hydrate (Monocarboaluminate) $Ca_4Al_2(CO_3)(OH)_{12}(H_2O)_5$	11.68° 23.51°	04-011-4223	• CLSM-REF	• TCG-NBFS-REF • TCG-NBFS-CLOSED • TCG-NBFS- N_2	• Not Observed	MEDIUM
Mullite *General:* $3Al_2O_3.2SiO_2$ *Actual:* $Al_2(Al_{2.588}Si_{1.412})O_{9.706}$	16.42° 25.96° 26.23° 35.21° 40.81°	01-074-8549	• All Samples	• All Samples	• All Samples	HIGH
Portlandite $Ca(OH)_2$	18.02° 34.09°	00-044-1481	• Not Observed	• Not Observed	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2	MEDIUM
Quartz SiO_2	20.85° 26.63° 50.11°	00-046-1045	• All Samples	• All Samples	• All Samples	HIGH
Calcite $CaCO_3$	23.08° 29.41°	01-078-4614	• All Samples	• All Samples	• All Samples	HIGH
Hematite Fe_2O_3	24.17° 33.19° 35.66°	01-089-0599	• All Samples	• All Samples	• All Samples	HIGH
Silicon *Si*	28.44° 47.30° 56.12°	00-027-1402	• All Samples	• All Samples	• All Samples	HIGH
Calcium Silicate Hydrates *C-S-H*	29.35°	00-012-0739 00-009-0329 00-015-0641	• Possibly present in all samples but most predominant in TCG-REF			MEDIUM

PHASE	OBSERVED PEAKS (2θ)	ICDD PDF	SAMPLES OBSERVED IN			CONFIDENCE LEVEL
			CLSM	TCG-NBFS	TCG	
Magnetite Fe_3O_4	30.21°	04-007-2718	• All Samples	• All Samples	• All Samples	HIGH

CLSM

XRD scans for the pre-leached CLSM sample and samples exposed to the various leaching environments are provided in **Figures B-10 through B-13**; tentatively identified phases are indicated in grey text on the XRD scans. **Figure B-14** provides a direct scan comparison to all CLSM samples (note that the background noise has been removed for enhanced clarity in **Figure B-14**). **Table B-9** indicates the quantitative data determined by Rietveld refinement using 10 wt% NIST Si as an internal standard (note that phases identified as MEDIUM or LOW confidence, per are in grey). Not surprisingly, since the CLSM contains 92.3% FA and 7.7% cement, all samples are dominated by the presence of the FA-containing "amorphous hump" and crystalline peaks associated with poorly-reacting quartz, mullite, hematite, and magnetite. Note also that the proportions of these phases are fairly consistent from one sample to another. The pre-leached sample indicates the presence of calcite, strätlingite, ettringite, hemicarboaluminate, monocarboaluminate, and a Ca-Al-CO_3-SO_3 hydrate. Rationales for the presence of these phases is as follows:

- **Calcite** – an additive to cement and BFS but also associated with the carbonation of cement hydration products, such as portlandite and calcium silicate hydrates (refer to **Appendix C-1**). It is noteworthy, however, that the calcite peak at approximately 29.4° 2θ (refer to **Figure B-10 (C)**) may be overlapped with peaks associated with calcium silicate hydrate (C-S-H) or calcium aluminosilicate hydrate (C-A-S-H) (i.e., the primary reaction products from hydration/pozzolanic reactions that serve to bind the cured cementitious structure) though the presence of C-S-H is perhaps more apparent when evaluating the TCG samples.
- **Strätlingite** – formed in aluminum-rich cement systems (e.g., cement/FA blends) (refer to **Appendix C-2**).
- **Ettringite** – AFt phase produced via the early reaction in cement between gypsum and tri-calcium aluminate (refer to **Appendix C-3**).

  Note: AFt stands for "alumina-ferric oxide-tri" and has a general formula of $[Ca_3(Al,Fe)(OH)_6 \cdot 12\ H_2O]_2 \cdot X_3 \cdot nH_2O$, where X_3 denotes three (tri) anions such as CO_3^{2-} or SO_4^{2-}.
- **Hemicarboaluminate / Monocarboaluminate** – AFm phases produced in cement systems with added limestone; carbonate anions can substitute for sulfate anions in monosulfoaluminate (a cement hydration product caused by the reaction of ettringite with tricalcium aluminate) (refer to **Appendix C-4**).

  Note: AFm stands for "alumina-ferric oxide-mono" and has a general formula of $[Ca_2(Al,Fe)(OH)_6)] \cdot X \cdot nH_2O$, where X equals a single (mono) anion, such as CO_3^{2-} or SO_4^{2-} .
- **Ca-Al-CO_3-SO_3 Hydrate** – AFm phase similar to the carboaluminates in which carbonate anions have been partially substituted by sulfate anions (refer to **Appendix C-5**).

Ettringite and the carboaluminate phases were only observed in the pre-leached samples. The proportion of ettringite would be expected to decrease as hydration continues due to ongoing reactions with tri-calcium aluminate in the cement to form monosulfoaluminate; however, the role of leaching in the disappearance of this phase must also be considered. Mineral phase solubility will ultimately be considered as part of a fiscal year 2021 (FY21) effort directed towards understanding the aqueous chemistry of tank closure grouts under various environmental conditions. Equally, both carboaluminate phases disappear for all leached samples; under non-leaching conditions and in the presence of "free" carbonate anions, hemicarboaluminate would be expected to convert to monocarboaluminate. Thus, it is conceivable that the complete loss of the carboaluminate phases is potentially associated with leaching. Only calcite and strätlingite persist in the leached samples though the proportion of strätlingite decreases, particularly for the CLOSED and N_2 treatments. The calcite proportion increases in all the leached samples (when compared to the non-leached reference sample), and as expected, the concentration is highest in the OPEN system with exposure to higher CO_2 concentrations and enhanced carbonation.

Regarding the Rietveld quantification data, **Table B-9** indicates GOFs in the range of 2.37 – 2.60. This is comparable to the GOF of 2.08 for the FA dry feed indicated in **Table B-4**. **Figure B-15** indicates the comparison between the measured and simulated patterns and as with the FA, the main discrepancies for CLSM are associated with the NIST Si and the quartz phases.

Table B-9: Rietveld quantification data for CLSM samples.

CLSM Sample	Phase (wt%)										GOF
	Amorphous	*Mullite*	*Quartz*	*Hematite*	*Magnetite*	*Calcite*	*Strätlingite*	*Ettringite*	*Hemicarbo-aluminate*	*Monocarbo-aluminate*	
REF (Non-Leached)	66.12	14.11	10.23	2.09	1.10	0.88	2.72	0.49	1.88	0.38	2.58
OPEN	65.55	14.75	10.95	2.12	1.48	3.01	2.14	-	-	-	2.60
CLOSED	68.79	14.75	11.19	2.12	1.64	1.51	Trace *	-	-	-	2.45
N_2	66.92	15.24	11.24	2.14	1.69	1.10	1.71	-	-	-	2.37

* Trace is indicated for strätlingite because the peak is barely above background and its inclusion (at such low concentrations) for Rietveld refinement resulted in pattern simulation anomalies; hence it was omitted during Rietveld quantification.

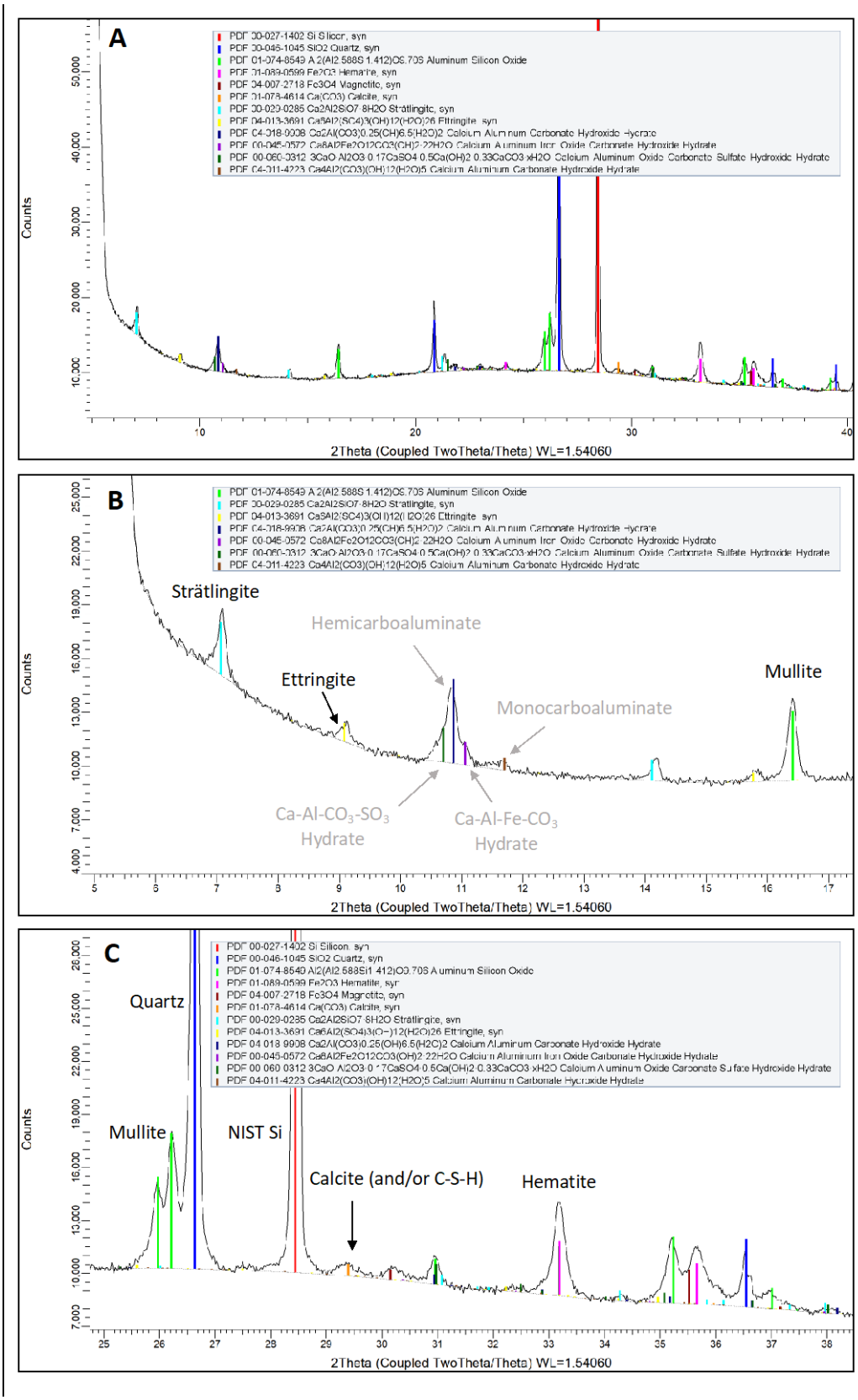


Figure B-10: XRD scan of pre-leached CLSM reference sample.

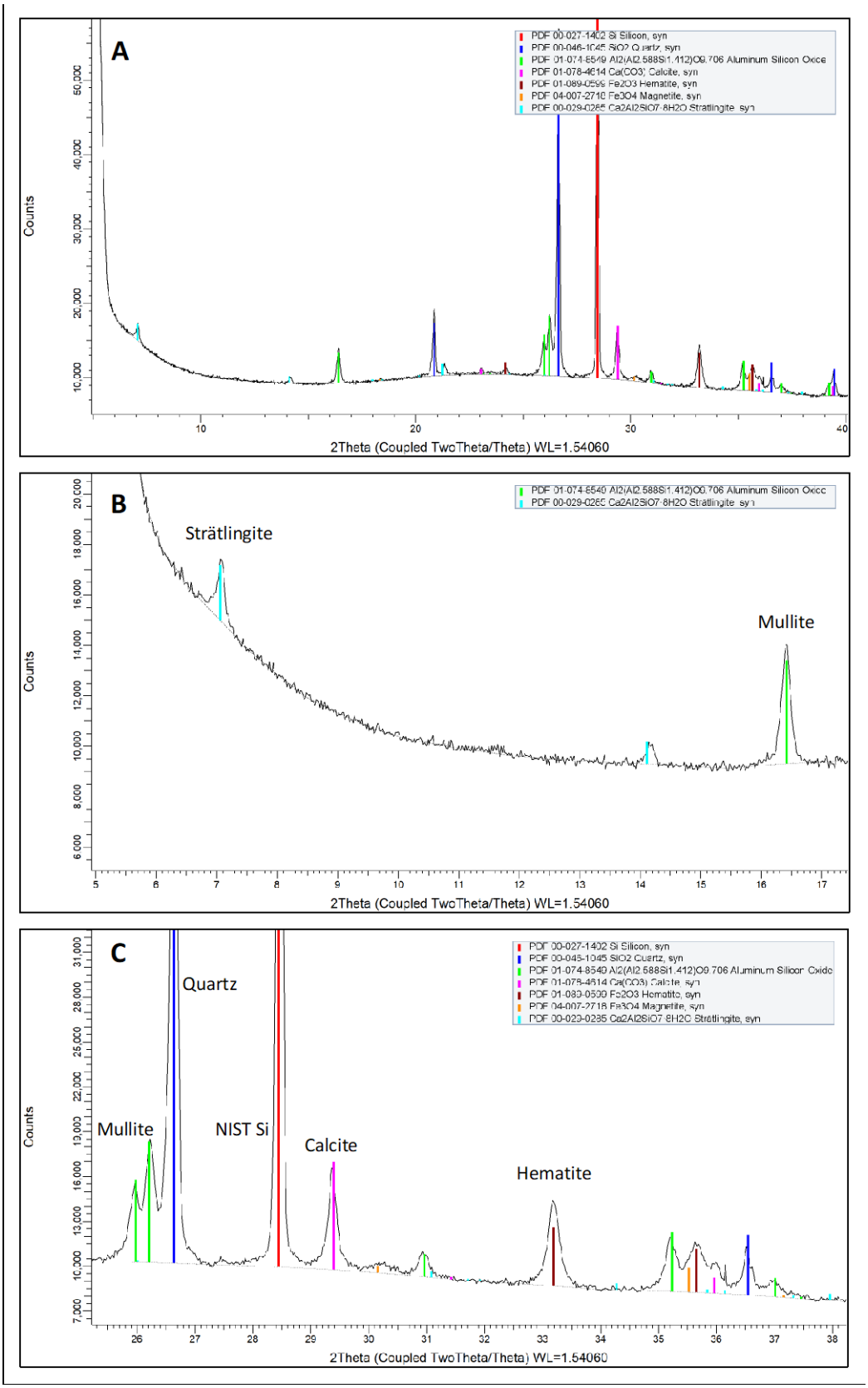


Figure B-11: XRD scan of CLSM-OPEN sample.

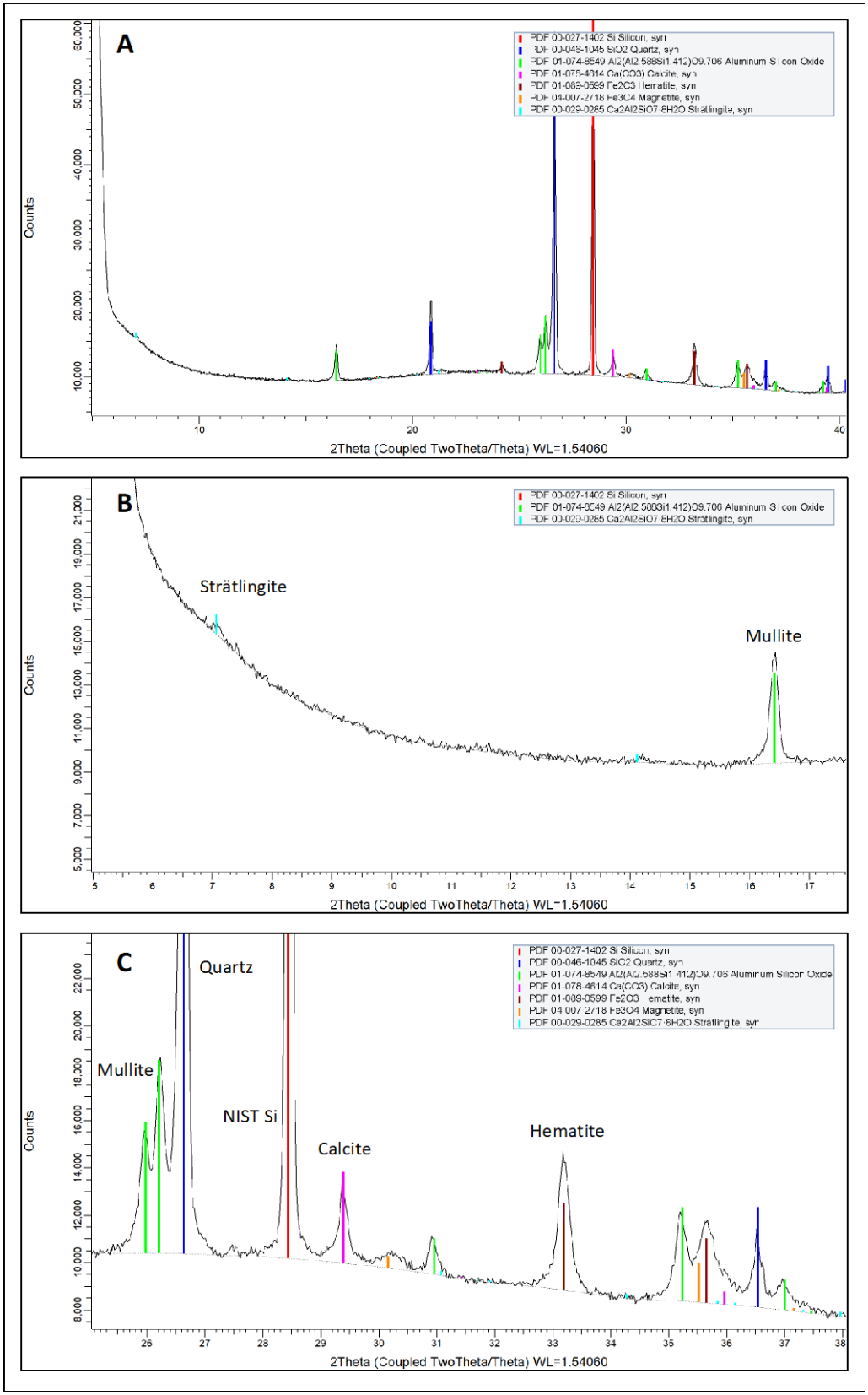


Figure B-12: XRD scan of CLSM-CLOSED sample.

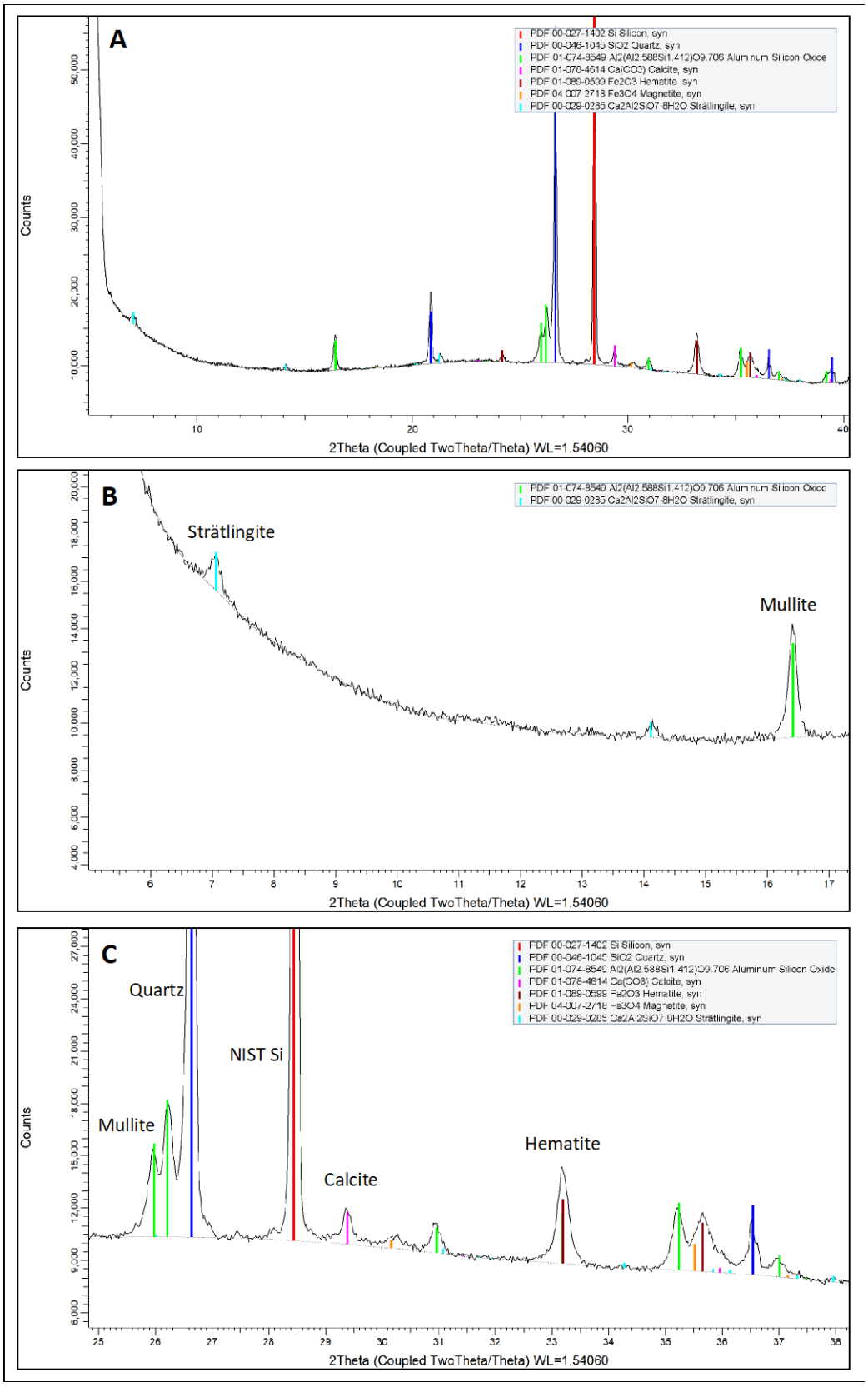


Figure B-13: XRD scan of CLSM-N_2 sample.

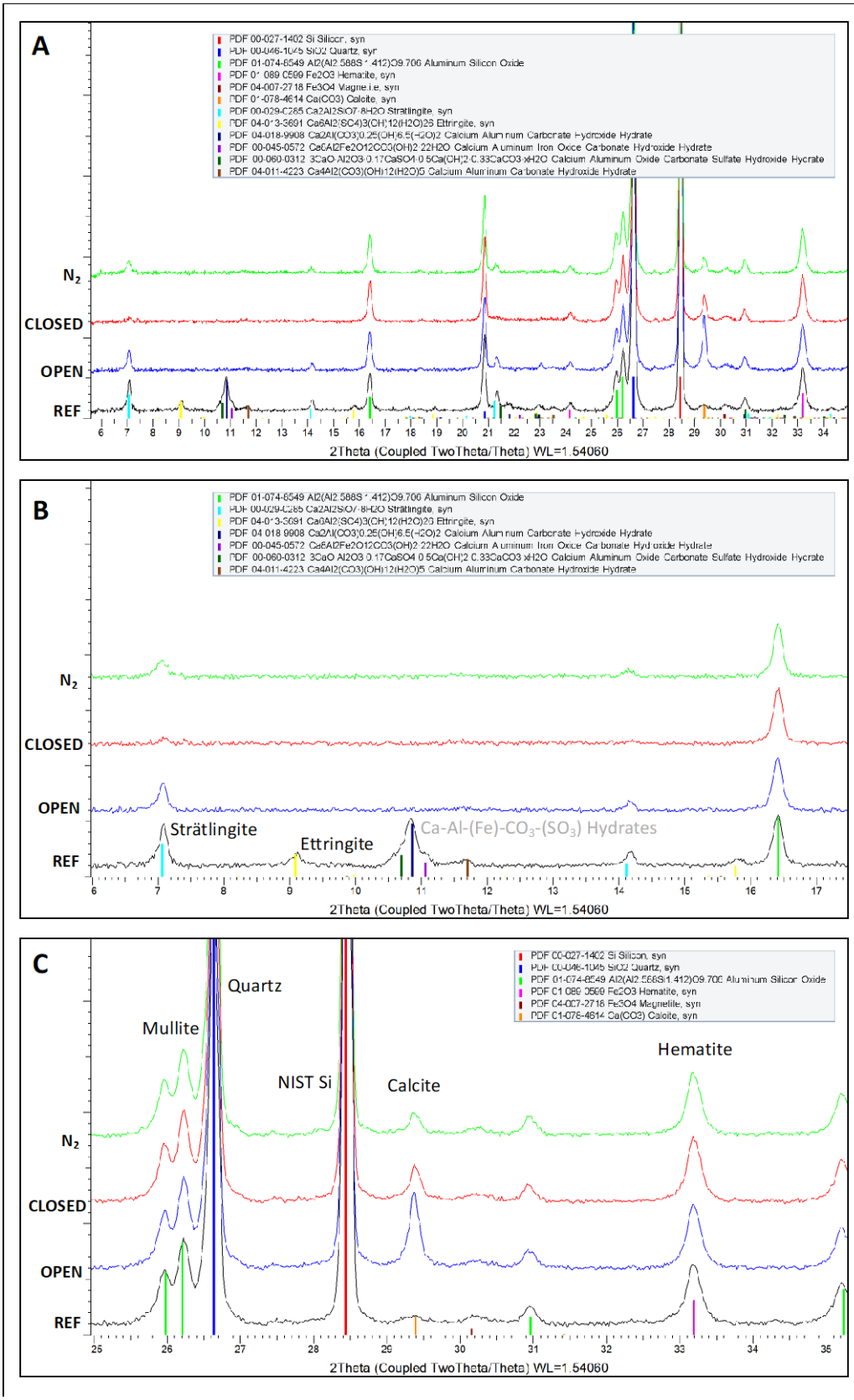


Figure B-14: XRD scan comparison for all CLSM samples.

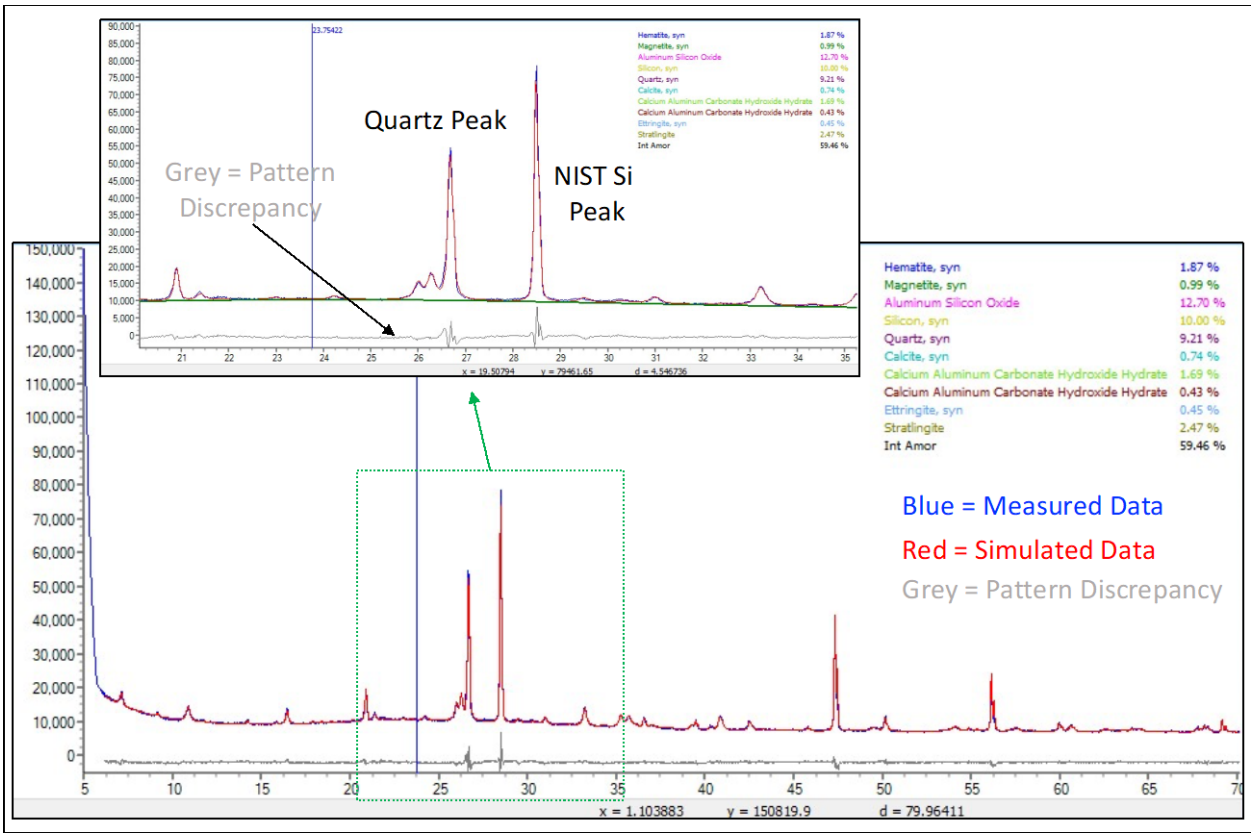


Figure B-15: Rietveld refinement data illustrating discrepancies between measured and simulated patterns for the non-leached CLSM sample.

TCG-NBFS

XRD scans for the pre-leached TCG-NBFS samples and samples exposed to the various leaching environments are provided in **Figures B-16** through **B-19**. **Figure B-20** provides a direct scan comparison between all TCG-NBFS samples. **Table B-10** indicates the quantitative data determined by Rietveld refinement. Phases that were detected by XRD but were lacking crystal structure data in the ICDD PDF have not been quantified.

The TCG-NBFS samples contain 82.1 wt% FA and 17.9 wt% cement and as with the CLSM samples, the XRD scans are dominated by the amorphous and poorly reacting crystalline phases associated with FA. The non-leached TCG reference sample indicates the presence of calcite, strätlingite, ettringite, and hemi- and mono-carboaluminates, all of which were also observed in the non-leached CLSM sample. Also, note that as with the CLSM, the calcite peak may overlap peaks associated with C-SH and/or C-A-S-H phases, though these are more apparent in the TCG reference sample. In addition to the aforementioned phases, some Ca-Fe-Al-CO_3 and Ca-Al-Si hydrates were also tentatively identified. Rationales for the presence of these additional phases is as follows:

- **Ca-Fe-Al-CO_3 Hydrate** – this phase is essentially an Fe-substituted carboaluminate phase; in multicomponent systems Fe can substitute for Al many AFm phases (refer to **Appendix C-5**).
- **Ca-Al-Si Hydrate** – in Al-rich cement systems (e.g., cement/FA blends) the Al may be substituted into the calcium silicate hydrates (C-S-H) to form calcium aluminosilicate hydrates (C-A-S-H); refer to **Appendix C-6**).

As with CLSM many of the phases observed in the non-leached reference sample are not detected in the leached samples; these phases may have disappeared as the hydration reactions progress or alternatively mineral dissolution and subsequent leaching may be occurring. For the OPEN sample (**Figure B-17**) only strätlingite and calcite are apparent. For the CLOSED sample, however, the low-angle strätlingite peak is not observed but trace amounts of the Fe-substituted carboaluminate and the monocarboaluminate are tentatively identified. The strätlingite peak returns in the N_2 sample and the carboaluminate phases observed in the CLOSED sample are also indicated. Calcite is present in all samples and, as expected, the highest proportion is detected for the OPEN sample.

Regarding the Rietveld quantification data, **Table B-10** indicates GOFs in the range of 2.45 – 2.85. **Figure B-21** indicates the comparison between the measured and simulated patterns and as with the CLSM, the main discrepancies for the TCG-NBFS are associated with the NIST Si and the quartz phases.

Table B-10: Rietveld quantification data for TCG-NBFS samples

TCG-NBFS Sample	Phase (wt%)										GOF
	Amorphous	*Mullite*	*Quartz*	*Hematite*	*Magnetite*	*Calcite*	*Strätlingite*	*Ettringite*	*Hemicarbo -aluminate*	*Monocarbo-aluminate*	
REF (Non-Leached)	66.97	11.69	8.64	1.79	0.99	2.63	1.85	1.41	2.61	1.43	2.45
OPEN	68.66	11.87	9.07	1.81	1.01	6.08	1.51	-	-	-	2.62
CLOSED	72.58	11.32	9.12	1.73	1.07	2.79	-	-	-	1.392	2.85
N_2	71.17	11.77	8.55	1.86	0.79	2.27	1.75	-	-	1.843	2.48

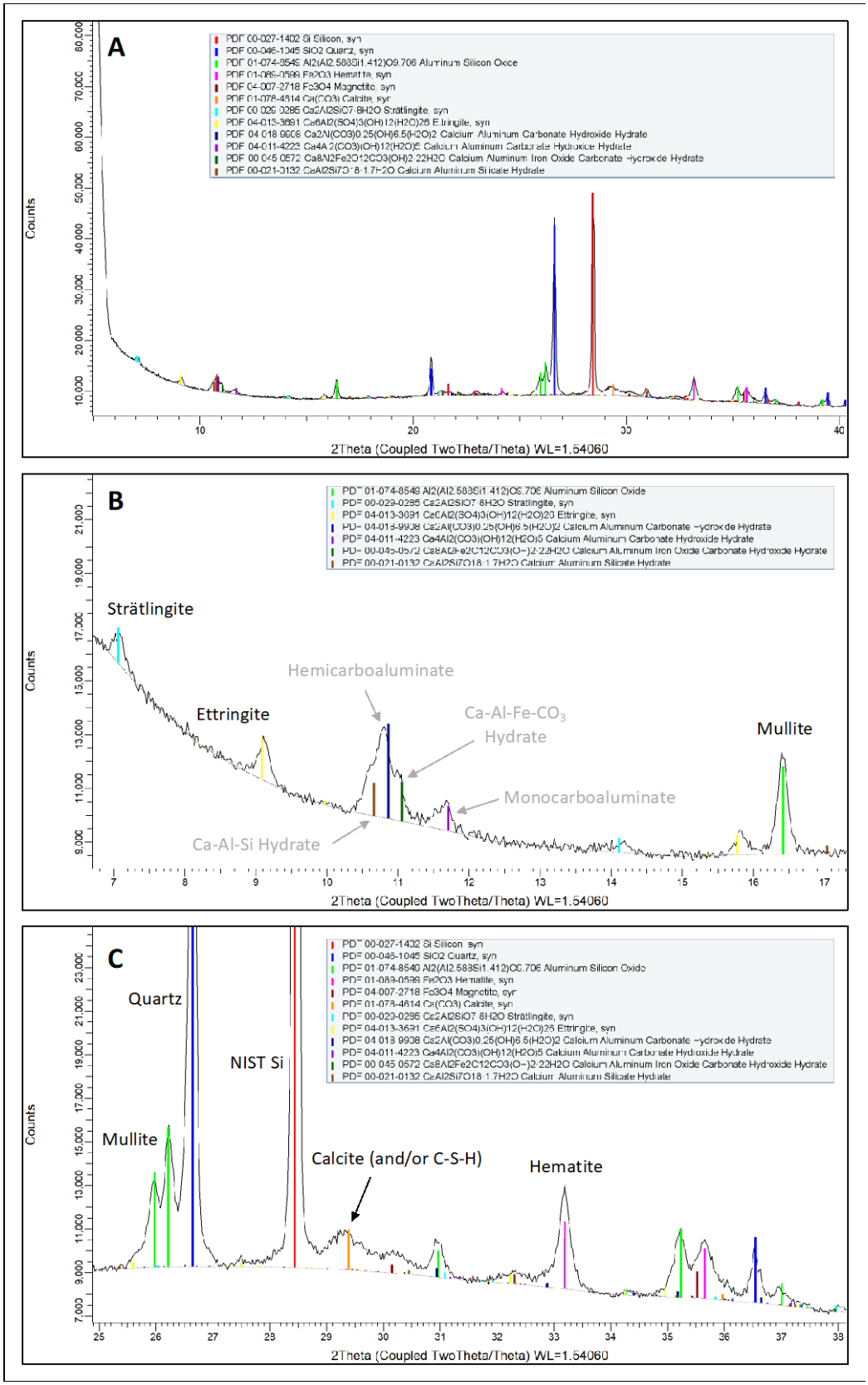


Figure B-16: XRD scan of pre-leached TCG-NBFS reference sample.

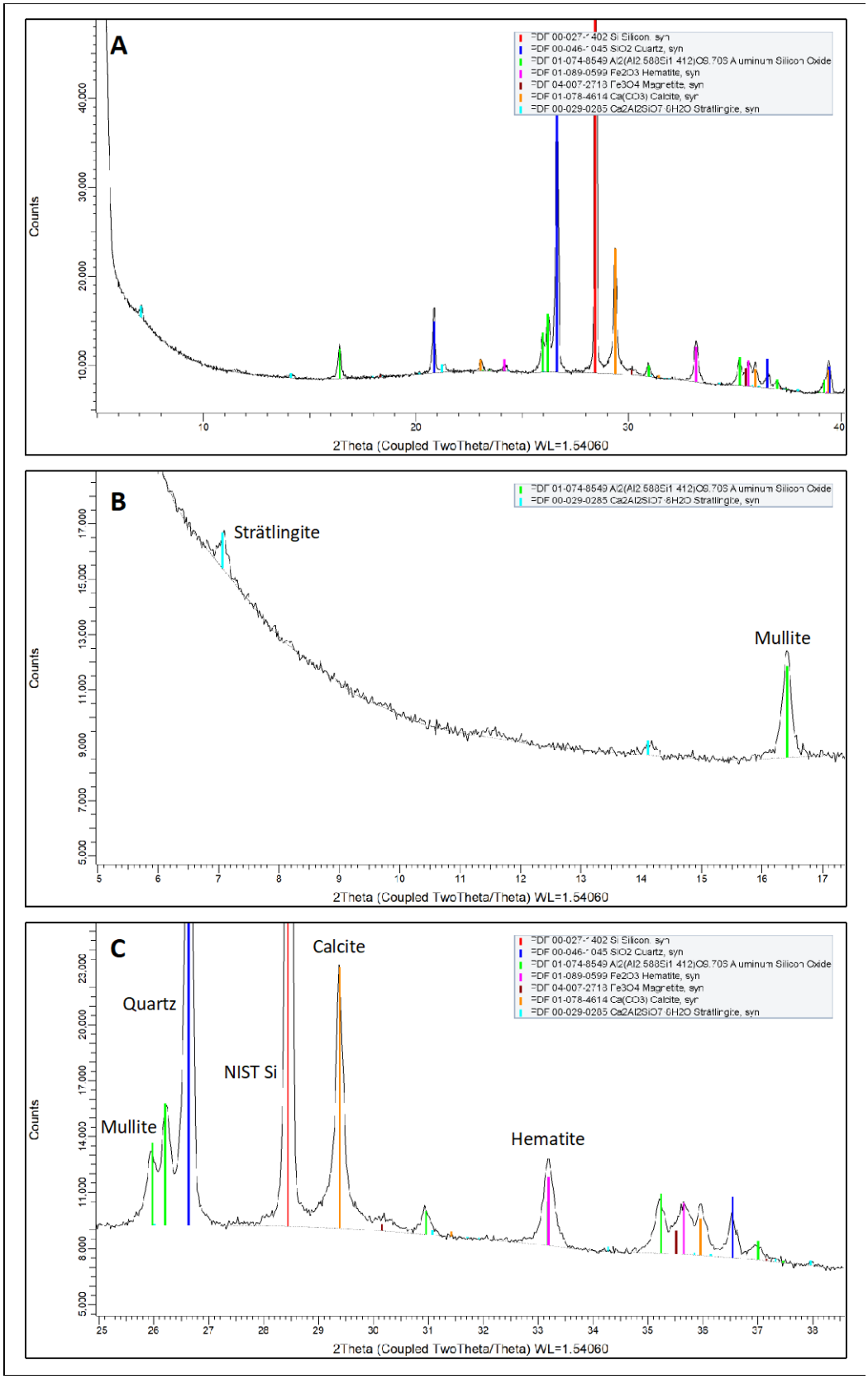


Figure B-17: XRD scan of TCG-NBFS-OPEN sample.

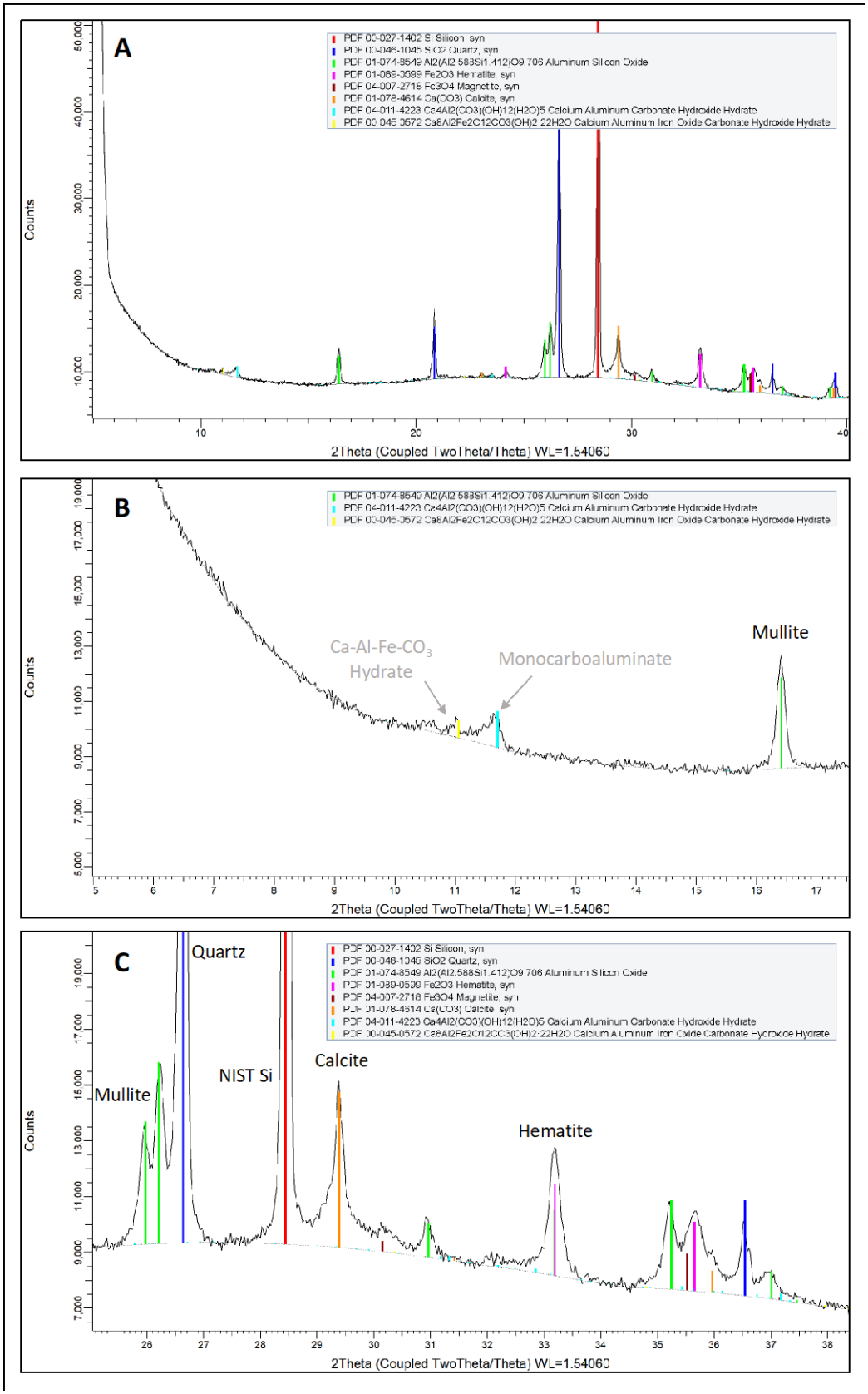


Figure B-18: XRD scan of TCG-NBFS-CLOSED sample.

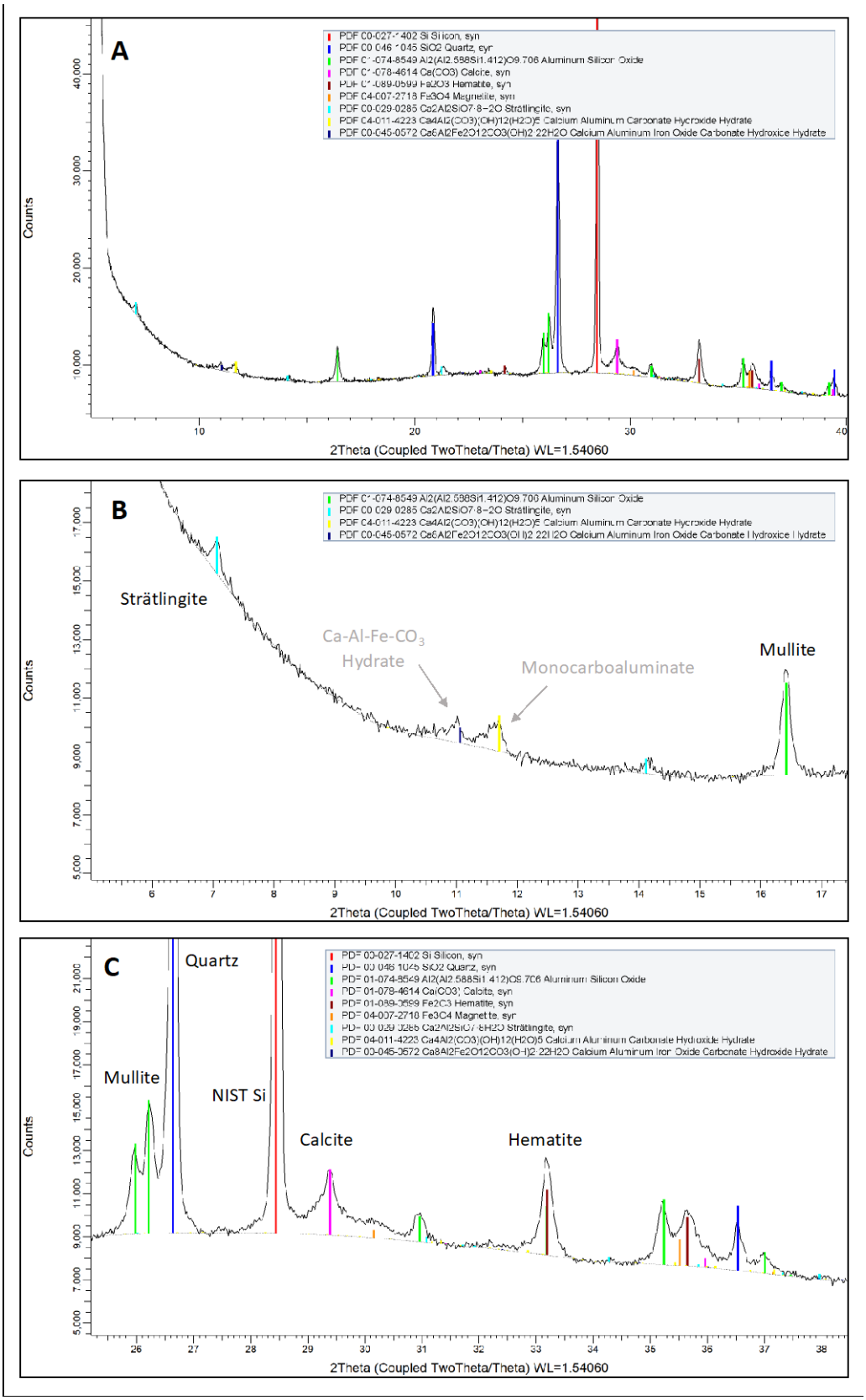


Figure B-19: XRD scan of TCG-NBFS-N_2 sample.

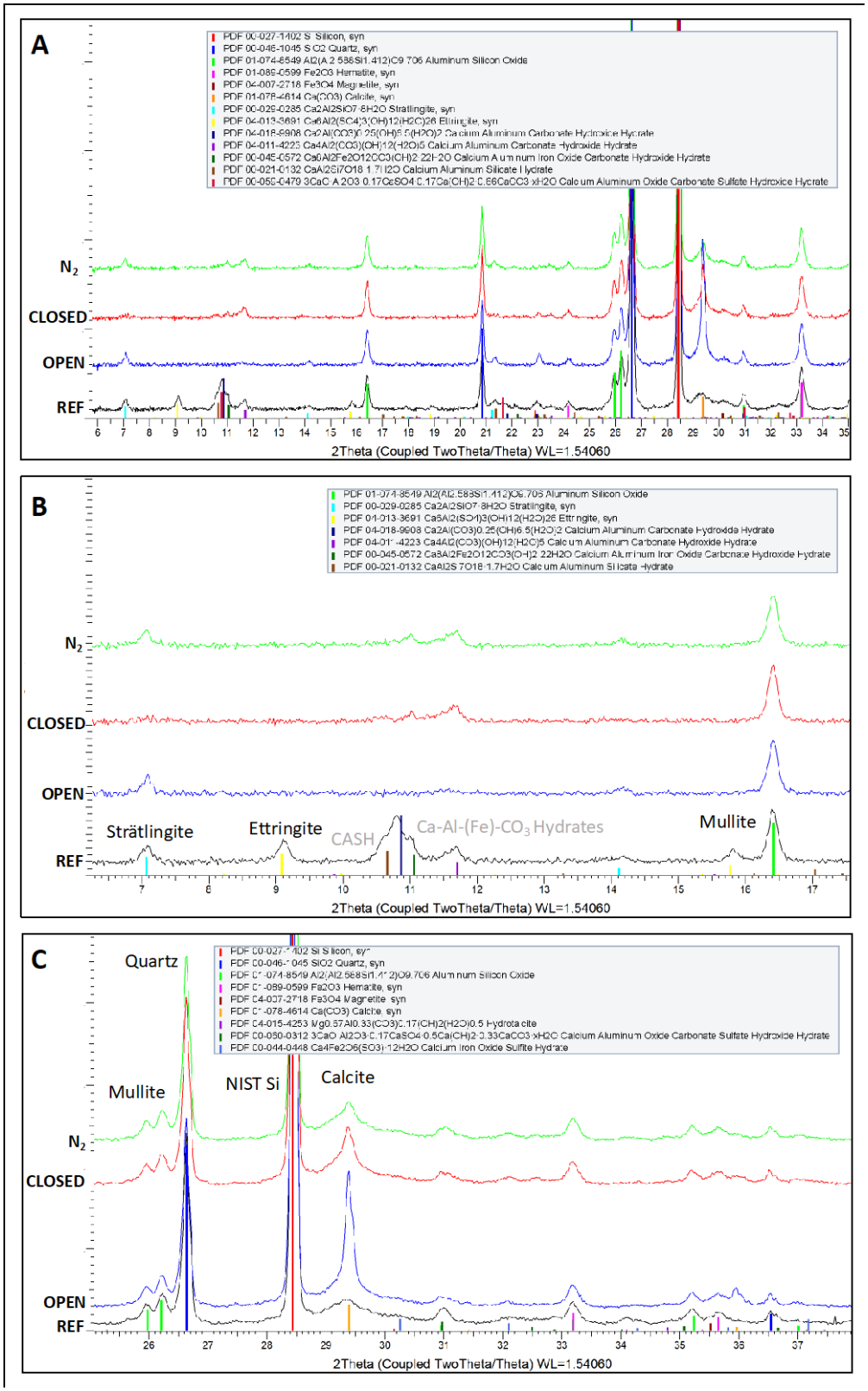


Figure B-20: XRD scan comparison for all TCG-NBFS samples.

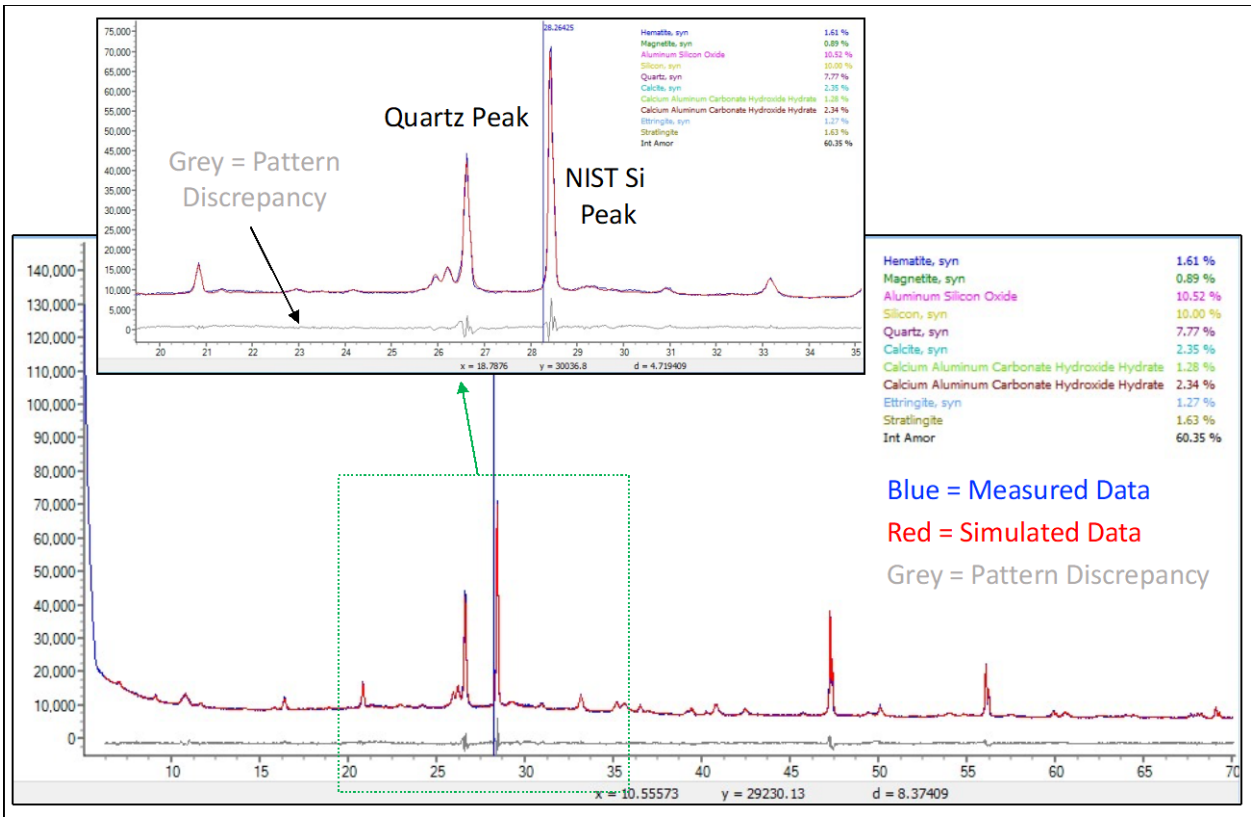


Figure B-21: Rietveld refinement data illustrating discrepancies between measured and simulated patterns for the non-leached TCG-NBFS sample.

TCG

XRD scans for the pre-leached TCG sample and samples exposed to the various leaching environments are provided in **Figures B-22** through **B-25**. **Figure B-26** provides a direct scan comparison between all TCG samples. **Table B-11** indicates the quantitative data determined by Rietveld refinement. Phases that were detected by XRD but were lacking crystal structure data in the ICDD PDF have not been quantified.

The TCG samples contain 30.1 wt% BFS, 52.0 wt% FA, and 17.9 wt% cement. As expected, the amorphous phase content has increased for the TCG (in comparison to the CLSM and TCG-NBFS samples) with the addition of BFS (approximately 78 wt% compared to 65-70 wt%). Additionally, the proportions of the FA crystalline phases of quartz, mullite, hematite, and magnetite have decreased due to the incorporation of the BFS.

The non-leached TCG reference sample indicates the presence of calcite, strätlingite, ettringite, and hemicarboaluminate, which were also observed in the CLSM and TCG-NBFS reference samples. It is important to note that despite the detection of strätlingite, it is barely above background and could ultimately not be quantified. Indeed, inclusion of this phase in Rietveld refinement resulted in simulation data anomalies, and thus the phase was omitted from the evaluation. A number of additional phases, including kuzelite, hydrotalcite, portlandite, and a Ca-Fe-SO_3 hydrate, were detected in the TCG reference sample (**Figure B-22 (B)**). There is also a broad peak around 29.3° 2θ (**Figure B-22 (C)**), which was somewhat apparent in the CLSM and TCG-NBFS samples, but appears more prominent in the TCG samples. Whilst this peak position also corresponds to calcite, other studies reveal that C-S-H and C-A-S-H phases indicate crystalline peaks around the same 2θ angle. It is thus possible that the calcite wt% indicated in Table X.12 are slightly high though the analyst has attempted to ensure that the fit of the calcite peak compensates for the presence of the broad C-S-H peak.

Rationales for the presence of these additional phases is as follows:

- **Kuzelite** – kuzelite is the mineral name for the AFm monosulfoaluminate phase which is formed via the reaction of tri-calcium aluminate (in cement) and the cement hydration product ettringite (refer to **Appendix C-7**).
- **Hydrotalcite** – formed in BFS-containing systems due to high Mg concentrations (refer to **Appendix C-8**). Hydrotalcite has the potential for XRD peak overlap with gypsum (added to both cement and BFS) at 11.6° 2θ. However, since, in both cement and BFS, gypsum is added to influence the hydration/pozzolanic reactions, it is assumed that gypsum has been consumed.
- **Portlandite** – portlandite is a primary cement hydration product but its presence in TCG samples is unexpected, partly because it was not observed in the CLSM or TCG-NBFS samples, but also because it is assumed that portlandite will be consumed during the alkali-dissolution and subsequent reaction of BFS. For these reasons the authors were unsure

about designating the observed peak as portlandite. However, when the portlandite peak at 18° 2θ emerges, a second (yet extremely small) characteristic portlandite peak also emerges at 34° 2θ (see **Figure B-22 (C)**). It should also be noted that the size of the portlandite peak is misleading since, with a plate-like morphology, portlandite exhibits preferred orientation (which was discussed earlier in this section), and the proportion of portlandite determined by Rietveld analysis is actually less than 0.5 wt% (refer to **Appendix C-9**).

- **Ca-Fe-SO_3 Hydrate** – similar to sulfoaluminate AFm phase in which the Al has been substituted with Fe to produce a sulfoferrite phase (refer to **Appendix C-5**).
- **C-S-H / C-A-S-H** – C-S-H is the primary reaction product of cement-based systems; C-A-S-H is formed in high-Al systems such as cement blends with FA or BFS (refer to **Appendix C-6**).

As with the CLSM and TCG-NBFS samples, many of the aforementioned phases disappear during the batch leaching experiments though both kuzelite and hydrotalcite appear to persist for all the environmental treatments. The persistence of these phases (under the various environments) will likely be considered as part of the FY21 studies related to the aqueous chemistry of tank closure grouts. As anticipated, the trend for calcite is identical to the CLSM and TCG-NBFS samples in that the calcite proportion is highest for the OPEN sample with higher CO_2 concentrations.

Regarding the Rietveld quantification data, **Table B-11** indicates GOFs in the range of 3.19 – 3.87, which is higher than any of the other samples presented evaluated in this study. **Figure B-27** indicates the comparison between the measured and simulated patterns, and it is apparent that the discrepancies are associated with those phases that could not be modeled due to a lack of complete crystal structure data.

Table B-11: Rietveld quantification data for TCG samples.

TCG Sample	Phase (wt%)												GOF
	Amorphous	*Mullite*	*Quartz*	*Hematite*	*Magnetite*	*Calcite*	*Ettringite*	*Strätlingite*	*Hydrotalcite*	*Kuzelite*	*Portlandite*	*Hemicarbo-aluminate*	
REF (Non-Leached)	78.40	6.06	5.44	1.22	0.14	1.14	0.91	Trace *	1.42	0.78	0.42	4.07	3.87
OPEN	77.85	6.55	5.64	1.30	0.22	5.37	-	Trace	2.67	0.41	-	-	3.19
CLOSED	78.92	5.48	5.28	1.29	0.15	3.38	-	Trace	3.07	1.75	0.08	-	3.50
N_2	79.66	6.64	5.96	1.44	0.24	1.30	-	Trace	2.78	1.77	0.20	-	3.44

* Trace is indicated for strätlingite because the peak is barely above background and its inclusion (at such low concentrations) for Rietveld refinement resulted in pattern simulation anomalies; hence it was omitted during Rietveld quantification.

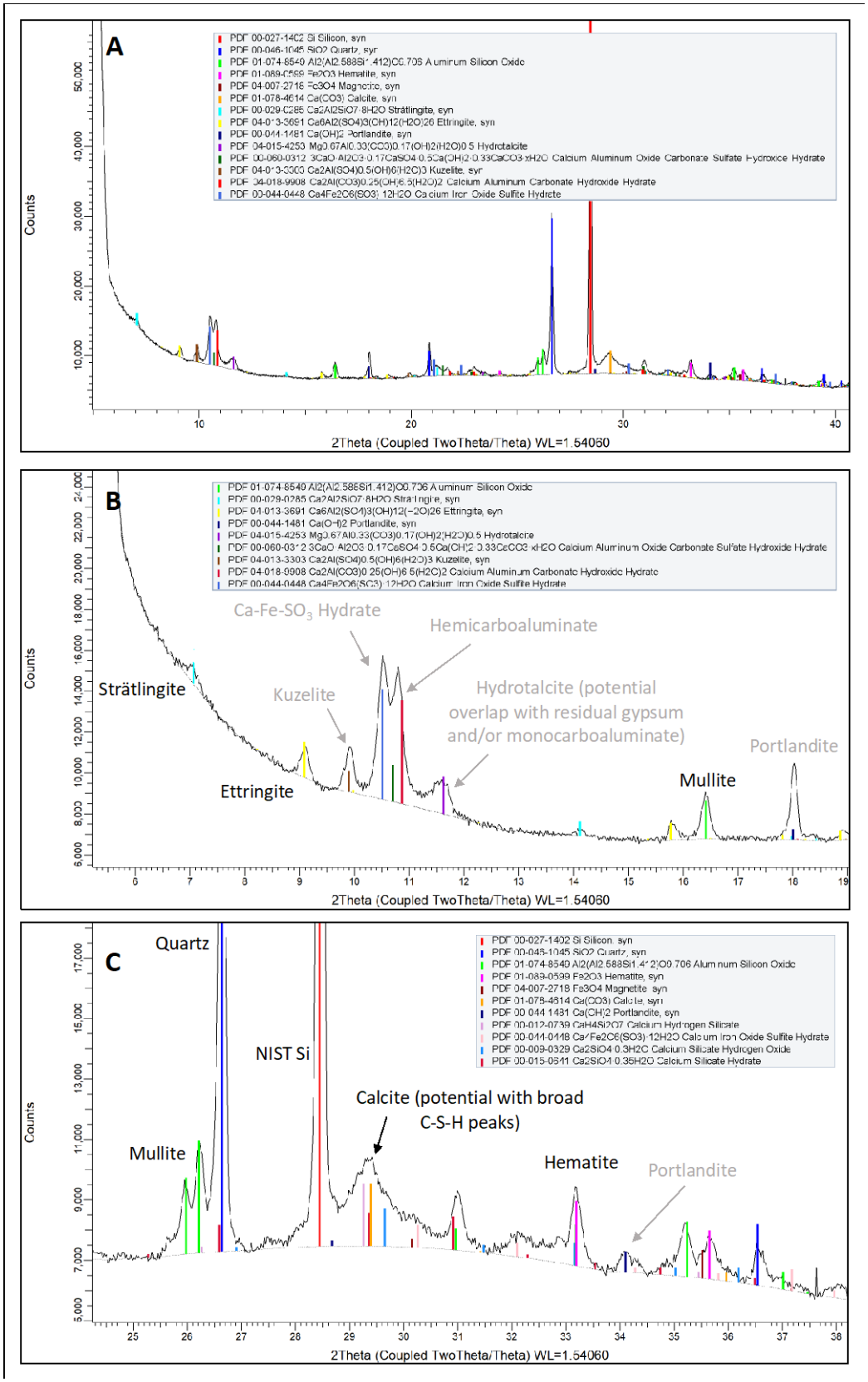


Figure B-22: XRD scan of pre-leached TCG reference sample.

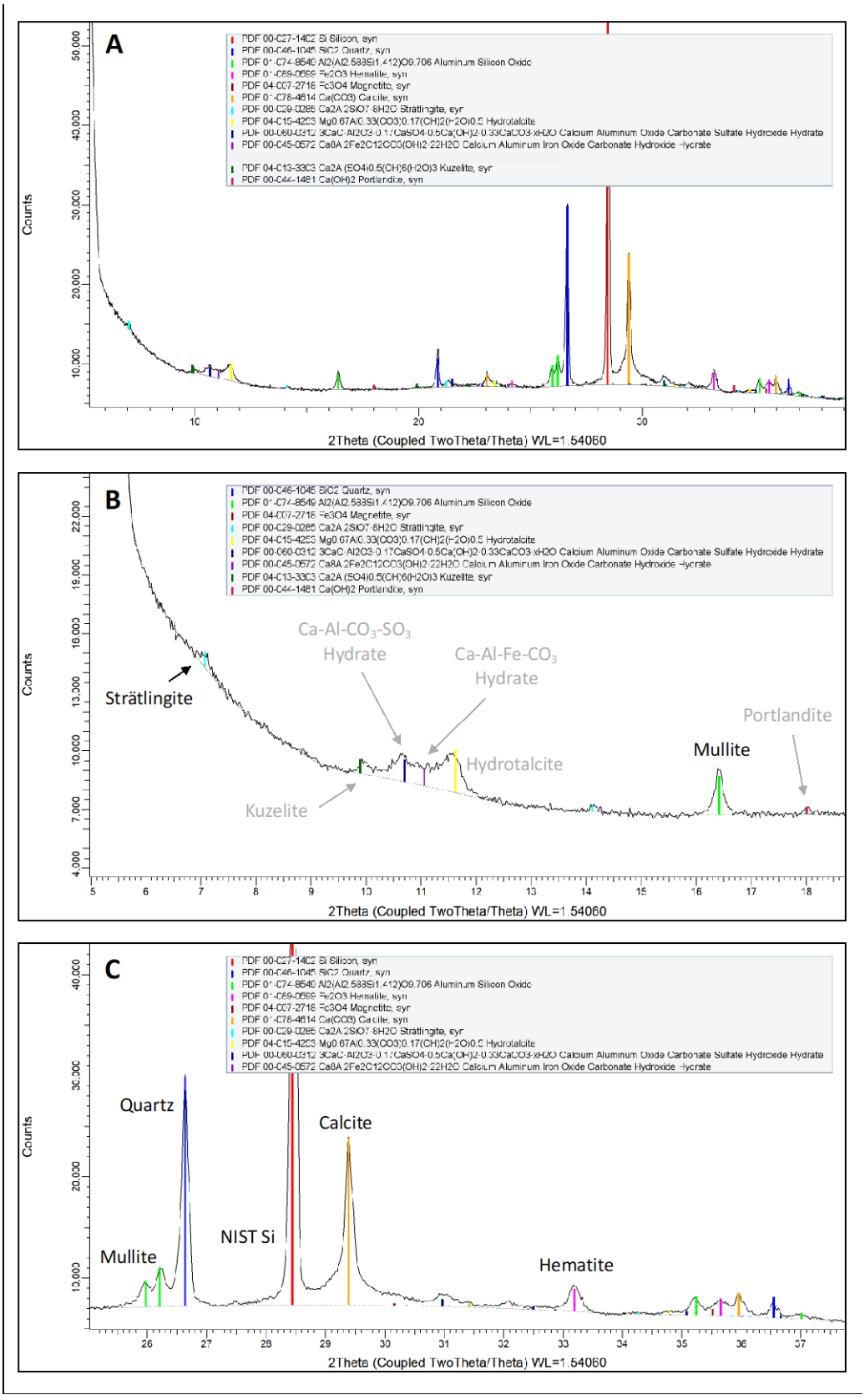


Figure B-23: XRD scan of TCG-OPEN sample.

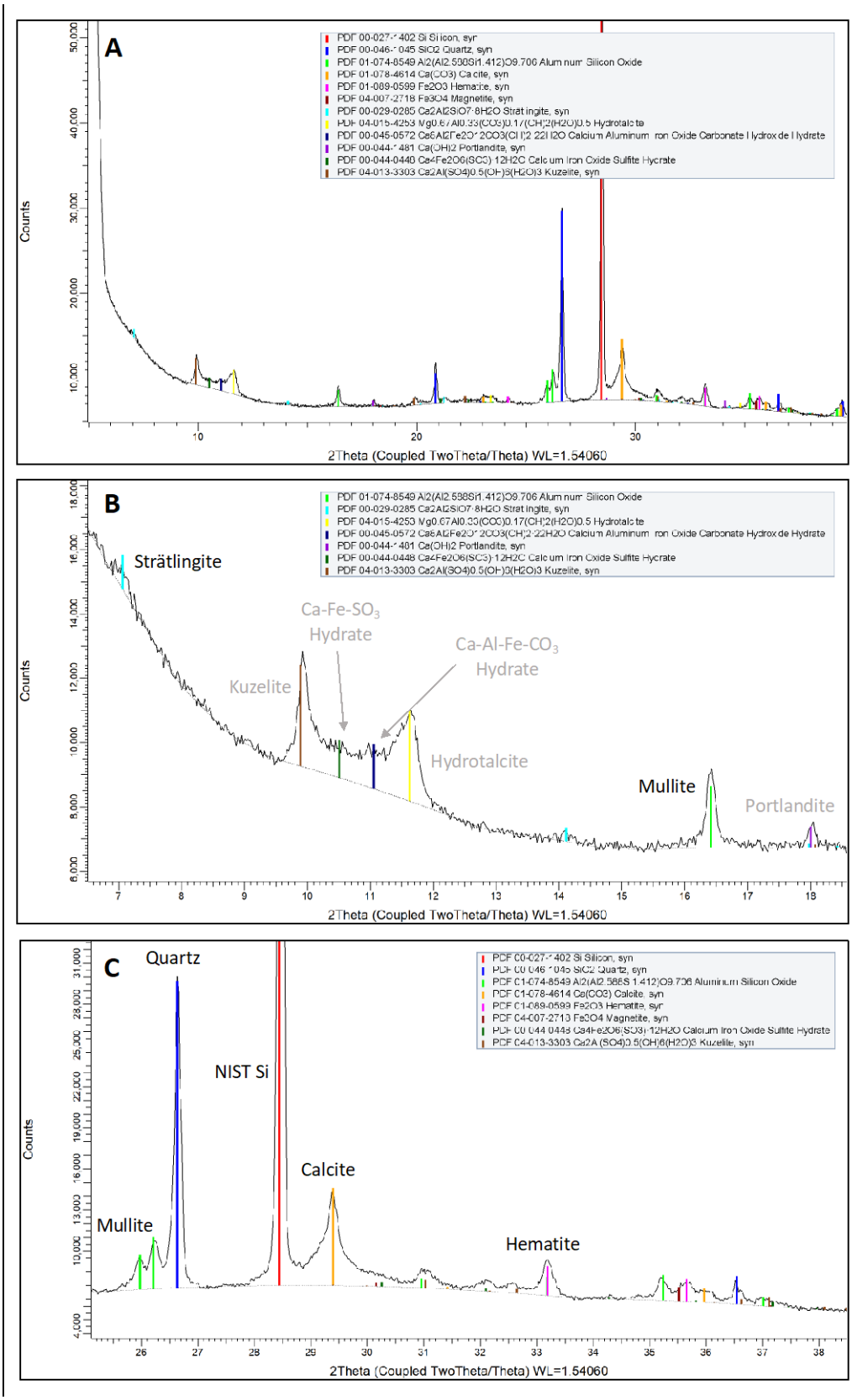


Figure B-24: XRD scan of TCG-CLOSED sample.

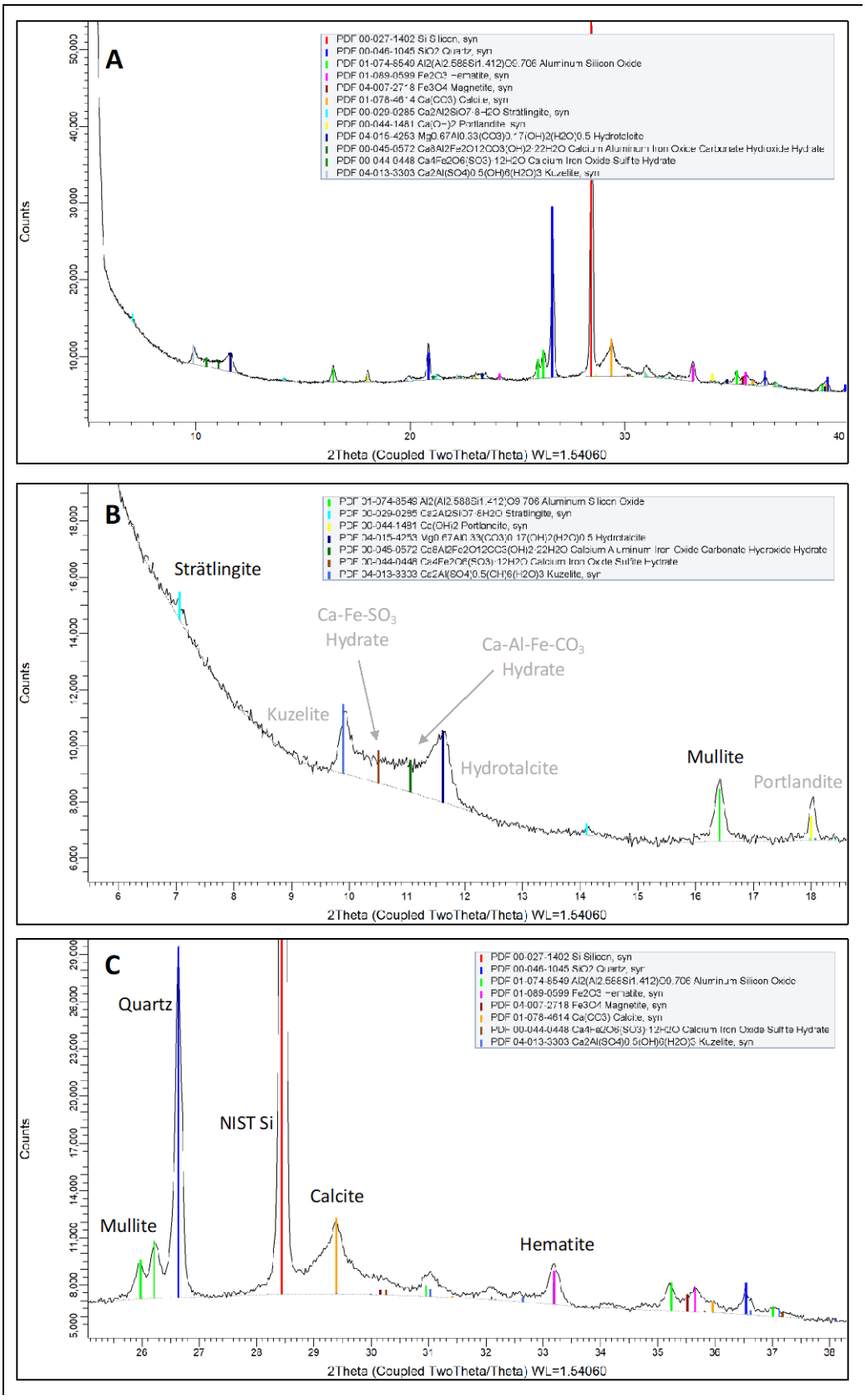


Figure B-25: XRD scan of TCG-N_2 sample.

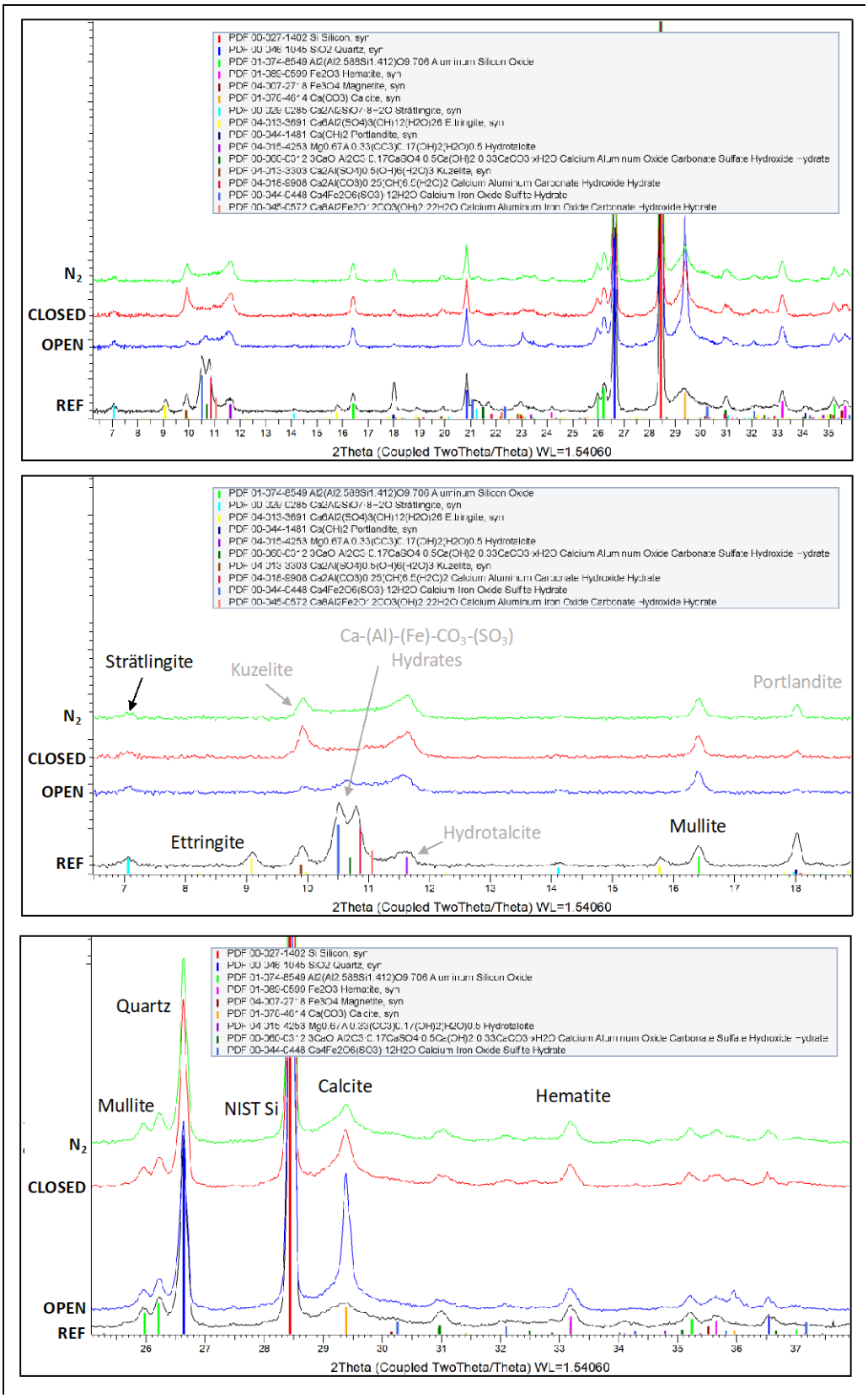


Figure B-26: XRD scan comparison for all TCG samples.

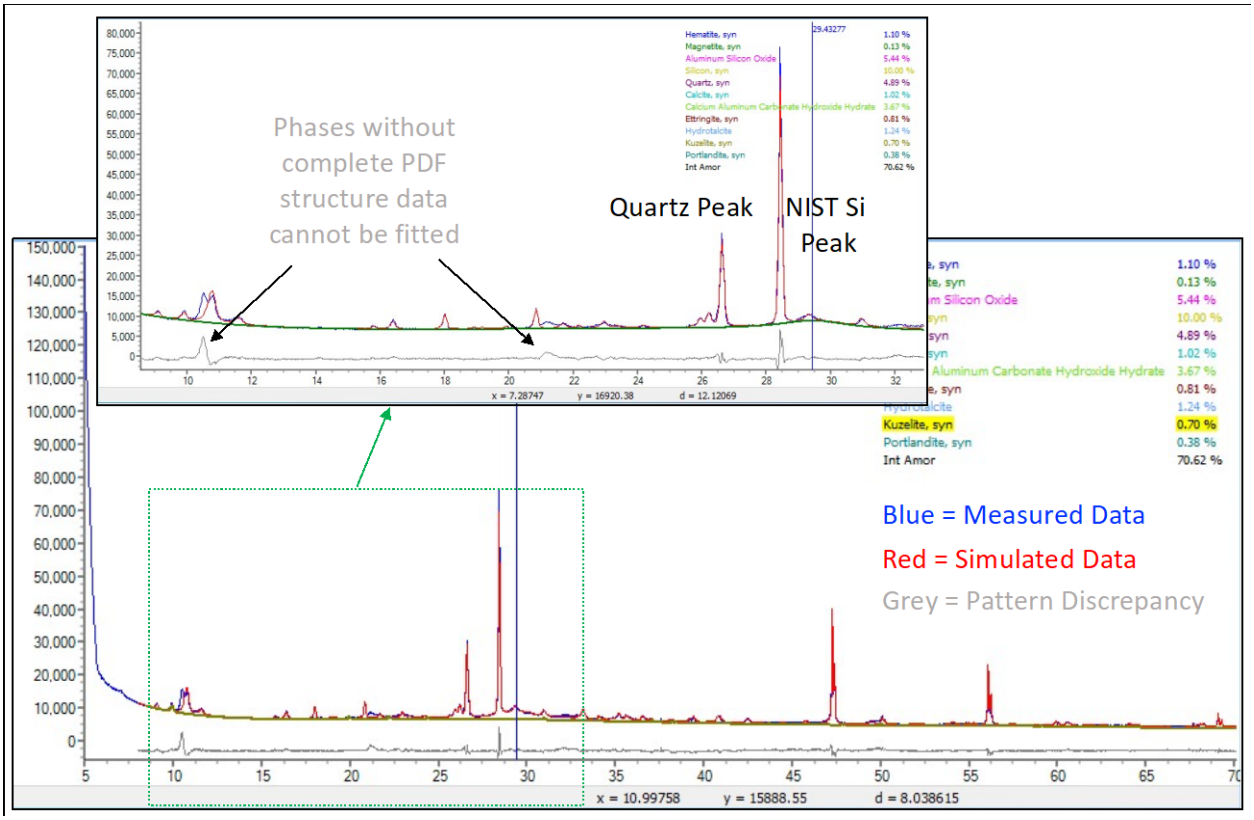


Figure B-27: Rietveld refinement data illustrating discrepancies between measured and simulated patterns for the non-leached TCG sample.

APPENDIX C: Rationale for XRD Peak Assignments

(**Note**: References identified in Appendix C are detailed at the end of Appendix B)

APPENDIX C-1: Calcite

PHASE	PEAKS OBSERVED (2θ)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	*TCG-NBFS*	*TCG*
Calcite (Limestone) $CaCO_3$	23.08° 29.41°	01-078-4614	• All samples	• All samples	• All samples

- Limestone (predominantly calcite – $CaCO_3$) is added to cement and BFS but is also the product of carbonation reactions.
- For cement, it was originally added as a substitute for cement clinker to reduce the energy consumption and costs associated with cement production (**Ingram et al., 1991**); for BFS, limestone is added as a grinding aid.
- Free calcite can interact with monosulfoaluminate (refer to page **Appendix C-7**); carbonate anions can displace the sulfate anions and produce calcium carboaluminate compounds (e.g., $3CaO•Al_2O_3•CaCO_3•11H_2O$) (refer to hemi- and mono-carboaluminates in **Appendix C-4**).
- Calcite may also react directly with tricalcium aluminate to form calcium hemi- and mono-carboalumiantes with the general formulas of $3CaO•Al_2O_3•CaCO_3•12H_2O$ and $3CaO•Al_2O_3•3CaCO_3•32H_2O$, respectively (**Ingram et al., 1991**).
- Calcite may form due to the carbonation[1] of cement hydration products including calcium hydroxide ($Ca(OH)_2$ - portlandite) and calcium silicate hydrates (C-S-H gels) (**Savija et al., 2016**).

$$Ca(OH)_2 + CO_2 = CaCO_3\,^{[2]} + H_2O$$

$$(CaO)_x(SiO_2)(H_2O) + xCO_2 = xCaCO_3 + SiO_2(H_2O)_t\,^{[3]} + (z\text{-}t)H_2O$$

- Hence, portlandite and C-S-H are expected to decrease as calcite increases.

Notes:

[1] Carbonation occurs when CO_2 in the presence of moisture reacts with Ca-bearing phases to form $CaCO_3$.

[2] Calcite is only one polymorph of calcium carbonate that may result from the carbonation of cement-based systems; other polymorphs include vaterite and aragonite though both are meta-stable and will ultimately convert to calcite (**Savija et al., 2016**).

[3] $SiO_2(H_2O)_t$ = amorphous silica gel.

APPENDIX C-2: Strätlingite

PHASE	PEAKS OBSERVED (2θ)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	***TCG-NBFS***	***TCG***
Strätlingite $Ca_2Al_2SiO_7.8H_2O$	7.09° 14.15° 21.35°	00-029-0285	• CLSM-REF • CLSM-OPEN • CLSM-CLOSED • CLSM-N_2	• TCG-NBFS-REF • TCG-NBFS-OPEN • TCG-NBFS-N_2	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2

- Strätlingite is an example of a Al-substituted calcium silicate hydrate (C-A-S-H) (also termed calcium aluminosilicate hydrate).
- **Bae et al. (2014)** indicated the formation of strätlingite in binary Type I/II cement / Class F FA systems; formed by the pozzolanic reaction of amorphous aluminosilicate FA with portlandite.
- Also predicted by thermodynamic modeling in cement / FA systems with > 33 wt% FA (**Loftenbach et al., 2011**).

APPENDIX C-3: Ettringite

PHASE	PEAKS OBSERVED (2θ)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	*TCG-NBFS*	*TCG*
Ettringite $Ca_6Al_2(SO_4)_3(OH)_{12}(H_2O)_{26}$	9.11° 15.80° 18.94° 22.97°	04-013-3691	• CLSM-REF	• TCG-NBFS-REF	• TCG-REF

- Ettringite (or calcium trisulfoaluminate hydrate) is an AFt[1] phase and is produced via the early and rapid reaction[2] between gypsum ($CaSO_4•2H_2O$) (added to both cement and BFS) and tricalcium aluminate ($3CaO•Al_2O_3$) in cement.

$$3CaO•Al_2O_3 + 3CaSO_4•2H_2O + 26H_2O = 3CaO•Al_2O_3•3CaSO_4.32H_2O \text{ } (or\ Ca_6Al_2(SO_4)_3(OH)_{12}•26H_2O)$$

- Ettringite subsequently reacts with tricalcium aluminate to form the AFm[3] phase calcium monosulfoaluminate hydrate (refer to **Appendix C-7**) per the following reaction:

$$2(3CaO•Al_2O_3) + 3CaO•Al_2O_3•3CaSO_4•32H_2O + 4H_2O = 3(3CaO•Al_2O_3•CaSO_4•12H_2O)$$

- Monosulfoaluminate can react with limestone (calcite – $CaCO_3$) (added to both cement and BFS); carbonate anions can displace the sulfate anions and produce calcium carboaluminate compounds (e.g., $3CaO•Al_2O_3•CaCO_3•11H_2O$) (refer to **Appendix C-4**).

- The displaced sulfate anions may subsequently interact with residual calcium monosulfoaluminate to reform ettringite.

Notes:

[1] AFt phases have the general formula $Ca_3(Al,Fe)(OH)_6•12\ H_2O]_2•X_3•nH_2O$, where X denotes an anion such as SO_4^{2-} and CO_3^{2-}.

[2] Typical cement reactions are detailed in **Portland Cement Association, *Concrete Information: Ettringite Formation and the Performance of Concrete,* PCA R&D Serial No. 2166.**

[3] AFm phases have the general formula $Ca_2(Al,Fe)(OH)_6)] •X•nH_2O$, where X denotes an anion such as SO_4^{2-} and CO_3^{2-}.

APPENDIX C-4: Carboaluminates

PHASE	PEAKS OBSERVED (2θ)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	***TCG-NBFS***	***TCG***
Calcium Aluminum Carbonate Hydroxide Hydrate (Hemicarboaluminate) $Ca_2Al(CO_3)_{0.25}(OH)_{6.5}(H_2O)_2$	10.80° 21.71° 30.99°	04-018-9908	• CLSM-REF	• TCG-NBFS-REF	• TCG-REF
Calcium Aluminum Carbonate Hydroxide Hydrate (Monocarboaluminate) $Ca_4Al_2(CO_3)(OH)_{12}(H_2O)_5$	11.68° 23.51°	04-011-4223	• CLSM-REF	• TCG-NBFS-REF • TCG-NBFS-CLOSED • TCG-NBFS-N_2	• Not observed

- AFm carboaluminate phases can be formed in cement systems that contain limestone ($CaCO_3$) additives or are open to CO_2-containing environments.
- Carbonate anions can displace sulfate anions in the hydration product monosulfoaluminate to produce calcium carboaluminate compounds (**Ipavec et al., 2011; Matschei et al., 2007**).
- Hemicarbonate appears during early hydration but can covert to moncarboaluminate if carbonate anions (either from residual limestone additive or dissolved CO_2) are available (**Ipavec et al, 2011.; Matschei et al., 2007**).

APPENDIX C-5: AFm-Type Phases

PHASE	PEAKS OBSERVED (2θ)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	***TCG-NBFS***	***TCG***
Calcium Iron Oxide Sulfite Hydrate $Ca_4Fe_2O_6(SO_3).12H_2O$	10.52°	00-044-0448	Not observed	Not observed	• TCG-REF • TCG-CLOSED • TCG-N_2
Calcium Aluminum Oxide Carbonate Sulfate Hydroxide Hydrate $3CaO.Al_2O_3.0.17CaSO_4.0.5Ca(OH)_2.0.33CaCO_3.xH_2O$	10.70°	00-060-0312	• CLSM-REF	Not observed	• TCG-REF • TCG-OPEN
Calcium Aluminum Iron Oxide Carbonate Hydroxide Hydrate $Ca_8Al_2Fe_2O_{12}CO_3(OH)_2.22H_2O$	11.06°	00-045-0572	• CLSM-REF	• TCG-NBFS-REF • TCG-NBFS-CLOSED • TCG-NBFS-N_2	• TCG-OPEN • TCG-CLOSED • TCG-N_2

- The above phases were tentalively identified by XRD; they are essentially AFm phases with full or partial cation and/or anion substitution (i.e., Al ⇔ Fe and SO_4^{2-}⇔ CO_3^{2-}).
- Anion substitution in AFm phases was previously discussed with respect to the formation of monocarboaluminates via the displacement of SO_4^{2-} by CO_3^{2-} in monosulfoaluminate (refer to **Appendix C-3**).
- **Dilnesa et al. (2011)** report on the formation of Fe-containing monocarbonate AFm phases with the general formula $Ca_3(Al_xFe_{2-x})O_6•CaCO_3•nH_2O$.

APPENDIX C-6: CSH-Type Phases

PHASE	PEAKS OBSERVED (2θ)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	***TCG-NBFS***	***TCG***
Calcium Silicate Hydrate *C-S-H*	≈ 29.2° [1]	00-012-0739 00-009-0329 00-015-0641	Most evident in TCG-REF but potentially present in other samples but hidden by calcite peak overlap.		
Calcium Aluminum Silicate Hydrate $CaAl_2Si_7O_{18}.1.7H_2O$	10.66° 21.29°	00-021-0132	• TCG-NBFS-REF		

- C-S-H is the main hydration product formed during the hydration of cementitious materials.[2]
- Al-substituted C-S-H (or C-A-S-H) can be formed in cement-based systems containing BFS and/or FA.
- C-S-H can be amorphous but can also C-S-H with long range order can also produce well-defined (but often broad) XRD diffraction peaks.

Note:

[1] Work by **Hunnicutt (2013)** indicates C-S-H and C-A-S-H peaks located at approximately 29.2° 2θ.

[2] **SREL DOC No. R-20-0001** provides information on the C-S-H and C-A-S-H hydration/pozzolanic products for blended cement systems.

APPENDIX C-7: Kuzelite

PHASE	PEAKS OBSERVED (2θ)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	*TCG-NBFS*	*TCG*
Kuzelite (Monosulfoaluminate) $Ca_2Al(SO_4)_{0.5}(OH)_6(H_2O)_3$	9.92°	04-013-3303	Not observed	Not observed	• TCG-REF • TCG-OPEN • TCG-CLOSED • TCG-N_2-C

- Ettringite, formed during early hydration reactions (refer to **Appendix C-2**) reacts with tricalcium aluminate (in the cement) to form the AFm[1] phase calcium monosulfoaluminate hydrate (mineral name: kuzelite) per the following reaction:

$$2(3CaO.Al_2O_3) + 3CaO•Al_2O_3•3CaSO_4•32H_2O + 4H_2O = 3(3CaO•Al_2O_3•CaSO_4•12H_2O)$$

- Monosulfoaluminate can react with limestone (calcite – $CaCO_3$) (added to both cement and BFS); carbonate anions can displace the sulfate anions and produce calcium carboaluminate compounds (e.g., $3CaO•Al_2O_3•CaCO_3•11H_2O$) (refer **Appendix C-4**).

- The displaced sulfate anions may subsequently interact with residual calcium monosulfoaluminate to reform ettringite.

Note:

[1] AFm phases have the general formula $Ca_2(Al,Fe)(OH)_6)]•X•nH_2O$, where X denotes an anion such as SO_4^{2-} and CO_3^{2-}.

APPENDIX C-8: Hydrotalcite

PHASE	PEAKS OBSERVED (2q)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	***TCG-NBFS***	***TCG***
Hydrotalcite [1] $Mg_{0.67}Al_{0.33}(CO_3)_{0.17}(OH)_2(H_2O)_{0.5}$	11.60°	04-015-4253	Not observed	Not observed	All samples

- For this study, hydrotalcite was only observed in BFS-containing samples.
- Empirical studies of ternary cement-BFS-FA systems indicate the formation of hydrotalcite (**Claret et al., 2018; Snyder et al., 2009**)
- Hydrotalcite is also thermodynamically predicted in BFS-containing systems (**Gruskovnjak et al., 2006**).

Notes:

[1] The hydrotalcite has potential XRD peak overlap with gypsum, though it is assumed that the gypsum (added to the cement and BFS) has been consumed during hydration/pozzolanic reactions.

APPENDIX C-9: Portlandite

PHASE	PEAKS OBSERVED (2θ)	ICDD PDF	OBSERVED IN WHICH SAMPLES		
			CLSM	*TCG-NBFS*	*TCG*
Portlandite $Ca(OH)_2$	18.02° [1] 34.09°	00-044-1481	Not observed	Not observed	All samples [2]

- Portlandite is a principal hydration product of cement resulting from the reactions of tricalcium silicate (alite) and dicalcium silicate (belite) with water.[3]

$$2(3CaO{\bullet}SiO_2) + 6H_2O = 3CaO{\bullet}2SiO_2{\bullet}3H_2O\ (C\text{-}S\text{-}H) + 3Ca(OH)_2$$

$$2(2CaO{\bullet}SiO_2) + 4H_2O = 3CaO{\bullet}2SiO_2{\bullet}3H_2O\ (C\text{-}S\text{-}H) + Ca(OH)_2$$

- Portlandite can subsequently be consumed in the following hydration reactions with the other cement phases tricalcium aluminate (to form tetracalcium aluminate hydrate) and tetracalcium aluminoferrite (to form calcium aluminoferrite hydrate).

$$3CaO{\bullet}Al_2O_3 + Ca(OH)_2 + 12H_2O = 3CaO{\bullet}Al_2O_3{\bullet}Ca(OH)_2{\bullet}12H_2O$$

$$4CaO{\bullet}Al_2O_3{\bullet}Fe_2O_3 + 2(Ca(OH)_2) + 10H_2O = 6CaO{\bullet}Al_2O_3{\bullet}Fe_2O_3{\bullet}12H_2O$$

- Portlandite is also consumed by carbonation and by the pozzolanic reactions of FA and BFS.

Note:

[1] ICDD PDF 00-044-1481 (**Appendix D**) indicates the highest intensity peak for portlandite at 34.1° 2θ. However, preferred orientation can result in the highest peak intensity occurring at 18.02° 2θ as observed in this study. The reader is referred to https://rruff.info/portlandite/names/asc/, which indicates an XRD pattern for portlandite with the highest intensity peak at approximately 18° 2θ.

[2] Portlandite was tentatively identified in the TCG samples though the authors acknowledge that its presence is counter intuitive since portlandite would be expected to be consumed by pozzolanic reactions with BFS, which was only contained within the TCG samples.

[3] Typical cement reactions are detailed in **Portland Cement Association, *Concrete Information: Ettringite Formation and the Performance of Concrete,* PCA R&D Serial No. 2166**.

Appendix D: ICDD Powder Diffraction Files

00-006-0495 Mav 15. 2020 1:29 PM (Steve Simner)

Status Deleted **Quality Mark:** Star **Environment:** Ambient **Temp:** 298.0 K **Chemical Formula:** Ca3 Al2 O6
Empirical Formula: Al2 Ca3 O6 **Weight %:** Al19.97 Ca44.50 O35.53 **Atomic %:** Al18.18 Ca27.27 O54.55
Compound Name: Aluminum Calcium Oxide **Entry Date:** 09/01/1956

Radiation: CuKα1 (1.5405 Å) **d-Spacing:** Diffractometer **Intensity:** Diffractometer - Peak

Crystal System: Cubic **SPGR:** Pa-3 (205)
Author's Unit Cell [a: 15.262 Å **Volume:** 3554.96 Å³ **Z:** 24.00 **MolVol:** 148.12 **]**
Calculated Density: 3.029 g/cm³ **Color:** Colorless **SS/FOM:** F(30) = 43.7(0.0146, 47)

Space Group: Pa-3 (205) **Molecular Weight:** 270.19 g/mol
Crystal Data [a: 15.262 Å **b:** 15.262 Å **c:** 15.262 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00°
XtlCell Vol: 3554.96 Å³ **XtlCell Z:** 24.00 **a/b:** 1.000 **c/b:** 1.000 **]**
Reduced Cell [a: 15.262 Å **b:** 15.262 Å **c:** 15.262 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **RedCell Vol:** 3554.96 Å³ **]**

Atomic parameters are cross-referenced from PDF entry 04-008-8069 **AC Space Group:** Pa-3 (205)
AC Unit Cell [a: 15.263(3) Å **b:** 15.263(3) Å **c:** 15.263(3) Å **α:** 90° **β:** 90° **γ:** 90° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	6	x,-y+1/2,z+1/2	11	z+1/2,-x+1/2,-y	16	z+1/2,x,-y+1/2	21	-y,z+1/2,-x+1/2
2	-x,-y,-z	7	-x+1/2,-y,z+1/2	12	-z+1/2,x+1/2,y	17	y,z,x	22	y,-z+1/2,x+1/2
3	x+1/2,-y+1/2,-z	8	x+1/2,y,-z+1/2	13	-z,x+1/2,-y+1/2	18	-y,-z,-x	23	-y+1/2,-z,x+1/2
4	-x+1/2,y+1/2,z	9	z,x,y	14	z,-x+1/2,y+1/2	19	y+1/2,-z+1/2,-x	24	y+1/2,z,-x+1/2
5	-x,y+1/2,-z+1/2	10	-z,-x,-y	15	-z+1/2,-x,y+1/2	20	-y+1/2,z+1/2,x		

ADP Type: U

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Uiso	AET
Ca	1	4a	.-3.	0.0	0.0	0.0	1.0	0.006	
Ca	2	4b	.-3.	0.5	0.0	0.0	1.0	0.0084	
Ca	3	8c	.3.	0.2561	0.2561	0.2561	1.0	0.0079	
Ca	4	8c	.3.	0.375	0.375	0.375	1.0	0.0117	
Ca	5	24d	1	0.1386	0.3763	0.1272	1.0	0.01307	
Ca	6	24d	1	0.38	0.3838	0.1209	1.0	0.00827	
Al	7	24d	1	0.2526	0.0133	0.0197	1.0	0.00647	
Al	8	24d	1	0.2444	0.2335	0.0046	1.0	0.00673	
O	9	24d	1	0.2777	0.1241	0.0103	1.0	0.01477	
O	10	24d	1	0.4835	0.1315	0.2536	1.0	0.01373	
O	11	24d	1	0.2664	0.2841	0.1049	1.0	0.0121	
O	12	24d	1	0.235	0.4047	0.2921	1.0	0.01423	
O	13	24d	1	0.3491	-0.0385	-0.0174	1.0	0.0132	
O	14	24d	1	0.1509	-0.0104	-0.0242	1.0	0.01207	

Anisotropic Displacement Parameters:

Atom	Num	Uani11	Uani22	Uani33	Uani12	Uani13	Uani23
Ca	1	0.006	0.006	0.006	0.0014	0.0014	0.0014
Ca	2	0.0084	0.0084	0.0084	5.0E-5	5.0E-5	5.0E-5
Ca	3	0.0079	0.0079	0.0079	6.5E-4	6.5E-4	6.5E-4
Ca	4	0.0117	0.0117	0.0117	0.0014	0.0014	0.0014
Ca	5	0.0079	0.009	0.0223	0.0013	0.0026	-5.0E-4
Ca	6	0.006	0.0092	0.0096	-5.5E-4	0.0012	5.5E-4
Al	7	0.0056	0.0058	0.008	-0.0011	-2.0E-4	-5.5E-4
Al	8	0.0078	0.0059	0.0065	-5.5E-4	7.5E-4	5.0E-4
O	9	0.017	0.0097	0.0176	-1.0E-4	-0.0013	-0.0011
O	10	0.0138	0.0086	0.0188	-0.0011	7.0E-4	-6.5E-4
O	11	0.0083	0.0182	0.0098	-0.0019	0.0034	-0.0031
O	12	0.0142	0.0094	0.0191	-5.5E-4	0.0039	0.0027
O	13	0.009	0.0159	0.0147	0.0022	-0.0031	-0.0041
O	14	0.0066	0.0154	0.0142	0.0031	0.0024	5.0E-4

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Cement and Hydration Product, Common Phase, Inorganic, Superconducting Material **Pearson Symbol:** cP264.00

Cross-Ref PDF #'s: 04-008-8069 (Primary)

References:

Type	DOI	Reference
Primary Reference		Swanson et al. Natl. Bur. Stand. (U. S.), Circ. 539 1995.
Crystal Structure		Crystal Structure Source: LPF.

00-006-0495 Mav 15. 2020 1:29 PM (Steve Simner)

Database Comments: Analysis: Spectrographic analysis (wt.%): <1 "Na"; <0.1 "Fe", "Mn"; <0.01 "Cu", "Mg", "Si", "Ag", "Sr"; <0.001 "Cr", "Sn". Deleted Or Rejected By: Deleted by corrected NBS card Set 8. Temperature of Data Collection: 298 K.

d-Spacings (40) - Ca3 Al2 O6 - 00-006-0495 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
17.40825	5.090000	5	2	2	1		38.18362	2.355000	2	5	4	1	
19.26252	4.604000	4	3	1	1		39.54511	2.277000	3	6	3	0	
20.95907	4.235000	10	2	3	0		40.91247	2.204000	11	4	4	4	
21.76484	**4.080000**	16	3	2	1		41.36357	2.181000	2	6	3	2	
23.29095	3.816000	1	4	0	0		42.23486	2.138000	<1	7	1	1	
23.99896	3.705000	2	4	1	0		43.10144	2.097000	3	6	4	1	
25.42008	3.501000	2	3	3	1		43.51553	2.078000	2	7	2	1	
26.73269	3.332000	5	4	2	1		44.36874	2.040000	5	6	4	2	
27.40299	3.252000	1	3	3	2		44.80839	2.021000	<1	7	2	2	
28.58647	3.120000	<1	4	2	2		45.59369	1.988000	4	7	3	1	
29.23742	3.052000	3	4	3	0		46.40788	1.955000	4	6	5	0	
29.82700	2.993000	3	4	3	1		46.78797	1.940000	<1	7	3	2	
31.54276	2.834000	6	4	3	2		47.62072	**1.908000**	36	8	0	0	
32.08892	2.787000	14	5	2	1		48.02170	1.893000	4	8	1	0	
33.15224	**2.700000**	100	4	4	0		48.78922	1.865000	1	7	3	3	
34.72816	2.581000	3	5	3	1		49.55379	1.838000	3	8	2	1	
35.71373	2.512000	<1	6	1	0		49.96010	1.824000	3	6	5	3	
36.23575	2.477000	1	6	1	1		50.70311	1.799000	<1	8	2	2	
37.23163	2.413000	7	6	2	0		51.12932	1.785000	<1	8	3	0	
37.70150	2.384000	6	6	2	1		51.81434	1.763000	<1	7	5	1	

00-009-0329 Jul 7, 2020 7:50 AM (Steve Simner)

Status Deleted **Quality Mark:** Low-Precision **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca2 Si O4 ·0.3 H2 O **Empirical Formula:** Ca2 H0.6 O4.3 Si
Weight %: Ca45.12 H0.34 O38.73 Si15.81 **Atomic %:** Ca25.32 H7.59 O54.43 Si12.66
Compound Name: Calcium Silicate Hydrogen Oxide **Entry Date:** 09/01/1959 **Modification Date:** 09/01/2003
Modifications: Quality

Radiation: CuKα (1.5418 Å) **Filter:** Ni Beta **Intensity:** Visual

Molecular Weight: 177.65 g/mol

Subfiles: Inorganic

References:

Type	DOI	Reference
Primary Reference		Heller, Taylor. Crystallographic Data Ca Silicates, London, HMSO 195663.

Database Comments: Deleted Or Rejected By: Delete: Weissmann parcel December 1, 1963 (for Set 15). Sample Preparation: Synthetic product, formed hydrothermally at 200 C and above. Warning: Unindexed pattern.

d-Spacings (21) - Ca2 Si O4 ·0.3 H2 O - 00-009-0329 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
16.31056	5.430000	20					35.02221	2.560000	20					54.93501	1.670000	20				
21.65738	4.100000	10					36.19040	2.480000	40					59.59747	1.550000	40				
23.39040	3.800000	20					38.95717	2.310000	5					63.44267	1.465000	10				
26.91370	3.310000	10					42.19350	2.140000	5					64.17687	1.450000	10				
29.65467	**3.010000**	100					47.56777	**1.910000**	100					69.58105	1.350000	5				
31.47439	2.840000	20					48.10274	**1.890000**	80					74.88368	1.267000	20				
33.15224	2.700000	60					50.67295	1.800000	60					83.04155	1.162000	30				

00-012-0739 Jul 7, 2020 7:48 AM (Steve Simner)

Status Primary **Quality Mark:** Blank **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca H4 Si2 O7 **Empirical Formula:** Ca H4 O7 Si2 **Weight %:** Ca18.88 H1.90 O52.76 Si26.46
Atomic %: Ca7.14 H28.57 O50.00 Si14.29 **Compound Name:** Calcium Hydrogen Silicate **Entry Date:** 09/01/1962

Radiation: CuKα (1.5418 Å) **Filter:** Ni Beta

Crystal System: Orthorhombic
Author's Unit Cell [a: 10.04 Å **b:** 3.82 Å **c:** 8.32 Å **Volume:** 319.10 Å³ **Z:** 2.00 **MolVol:** 159.55
c/a: 0.829 **a/b:** 2.628 **c/b:** 2.178 **]** **Calculated Density:** 2.209 g/cm³ **Measured Density:** 2.23 g/cm³
SS/FOM: F(14) = 0.8(0.1391, 124)

Molecular Weight: 212.27 g/mol
Crystal Data [a: 8.320 Å **b:** 10.040 Å **c:** 3.820 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **XtlCell Vol:** 319.10 Å³
XtlCell Z: 2.00 **c/a:** 0.459 **a/b:** 0.829 **c/b:** 0.380 **]**
Reduced Cell [a: 3.820 Å **b:** 8.320 Å **c:** 10.040 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **RedCell Vol:** 319.10 Å³ **]**

Subfiles: Cement and Hydration Product, Inorganic **Pearson Symbol:** o?28.00 **Pearson Symbol w/o H:** o?20

References:

Type	DOI	Reference
Primary Reference		Funk, Thilo. Z. Anorg. Allg. Chem. 1955, 278, 245.

Database Comments: Warning: One or more of the 4th-8th strongest lines are unindexed. Unit Cell Data Source: Powder Diffraction.

d-Spacings (15) - Ca H4 Si2 O7 - 00-012-0739 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
10.78023	**8.200000**	60	0	0	1		43.03680	2.100000	10	4	1	0	
17.44279	5.080000	10	2	0	0		47.56777	1.910000	10	0	2	0	
21.23803	4.180000	40	0	0	2		50.07746	**1.820000**	100	0	1	4	
23.32814	3.810000	10	0	1	0		52.22818	1.750000	10	2	2	1	
26.26706	3.390000	10	3	0	0		60.45716	1.530000	10	6	1	0	
29.25702	**3.050000**	100	2	1	0		70.78267	1.330000	10	4	2	3	
35.45118	2.530000	10	3	1	0		75.37195	1.260000	10	0	3	1	
39.49092	2.280000	10											

00-015-0641 Jul 7, 2020 7:51 AM (Steve Simner)

Status Primary **Quality Mark:** Low-Precision **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca2 Si O4 ·0.35 H2 O **Empirical Formula:** Ca2 H0.7 O4.35 Si
Weight %: Ca44.90 H0.40 O38.98 Si15.73 **Atomic %:** Ca24.84 H8.70 O54.04 Si12.42
Compound Name: Calcium Silicate Hydrate **Entry Date:** 09/01/1965 **Modification Date:** 09/01/2006
Modifications: CellParamSource

Radiation: CuKα (1.5418 Å) **Filter:** Ni Beta **Intensity:** Visual

Molecular Weight: 178.55 g/mol

Subfiles: Cement and Hydration Product, Inorganic

References:

Type	DOI	Reference
Primary Reference		Funk. Z. Anorg. Allg. Chem. 1958, 297, 103.

Database Comments: General Comments: Low temperature form. Warning: Unindexed pattern. Unit Cell Data Source: Powder Diffraction.

d-Spacings (30) - Ca2 Si O4 ·0.35 H2 O - 00-015-0641 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
16.52505	5.360000	20					47.72697	**1.904000**	100				
21.13573	4.200000	2					50.52270	1.805000	80				
23.57919	3.770000	10					52.71433	1.735000	10				
25.28059	3.520000	10					54.23116	1.690000	80				
26.58641	3.350000	80					55.33035	1.659000	80				
29.35541	**3.040000**	100					59.63984	1.549000	50				
30.91611	**2.890000**	100					62.25797	1.490000	2				
32.29122	2.770000	10					63.63670	1.461000	2				
33.53568	2.670000	20					64.92948	1.435000	2				
34.74204	2.580000	20					66.76147	1.400000	2				
36.49495	2.460000	20					74.74546	1.269000	20				
38.26802	2.350000	10					76.88002	1.239000	2				
40.03982	2.250000	2					82.00863	1.174000	80				
42.99381	2.102000	20					98.99804	1.013000	10				
46.30760	1.959000	2					103.33029	0.982000	10				

00-021-0132 Jun 1, 2020 6:21 PM (Steve Simner)

Status Primary **Quality Mark:** Indexed **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca Al2 Si7 O18 ·1.7 H2 O **Empirical Formula:** Al2 Ca H3.4 O19.7 Si7
Weight %: Al8.86 Ca6.58 H0.56 O51.73 Si32.27 **Atomic %:** Al6.04 Ca3.02 H10.27 O59.52 Si21.15
Compound Name: Calcium Aluminum Silicate Hydrate **Entry Date:** 09/01/1971 **Modification Date:** 09/01/2003
Modifications: Quality

Intensity: Visual

Crystal System: Orthorhombic
Author's Unit Cell [a: 15.2 Å **b:** 16.6 Å **c:** 7.26 Å **Volume:** 1831.84 Å³ **Z:** 4.00 **MolVol:** 457.96
c/a: 0.478 **a/b:** 0.916 **c/b:** 0.437 **] Calculated Density:** 2.209 g/cm³ **SS/FOM:** F(18) = 2.6(0.0453, 154)

Molecular Weight: 609.25 g/mol
Crystal Data [a: 15.200 Å **b:** 16.600 Å **c:** 7.260 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **XtlCell Vol:** 1831.84 Å³
XtlCell Z: 4.00 **c/a:** 0.478 **a/b:** 0.916 **c/b:** 0.437 **]**
Reduced Cell [a: 7.260 Å **b:** 15.200 Å **c:** 16.600 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **RedCell Vol:** 1831.84 Å³ **]**

Subfiles: Cement and Hydration Product, Inorganic, Micro & Mesoporous (Zeolite), Mineral Related
Zeolite Classification: ZZ9 (Unknown structure) **Pearson Symbol:** o?132.40 **Pearson Symbol w/o H:** o?118.8

References:

Type	DOI	Reference
Primary Reference		Simonot-Grange, Watelle-Marion, Cointot. Bull. Soc. Chim. Fr. 19682747.

Database Comments: General Comments: Natural specimen, contains "Ca0.92", "Na0.20", "Mg0.04", "K0.08". Sample Preparation: Heated to 257 C at 15 torr. Warning: Lines with abs(delta 2Theta)>0.06 DEG. Unit Cell Data Source: Powder Diffraction.

d-Spacings (18) - Ca Al2 Si7 O18 ·1.7 H2 O - 00-021-0132 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
10.66286	**8.290000**	100	0	2	0		25.35381	3.510000	10	1	4	1	
13.28312	6.660000	6	0	1	1		26.50584	3.360000	6	4	0	1	
16.13111	5.490000	6	0	2	1		29.45448	3.030000	6	0	3	2	
17.03723	5.200000	25	1	3	0		30.45179	2.933000	14	3	4	1	
17.44279	5.080000	8	3	0	0		31.57706	2.831000	18	2	3	2	
17.79586	4.980000	8	2	1	1		32.30320	2.769000	35	0	6	0	
21.39336	**4.150000**	60	0	4	0		35.16402	2.550000	6	1	6	1	
23.26622	3.820000	25	2	3	1		36.49495	2.460000	1	5	4	0	
24.43407	**3.640000**	35	2	4	0		43.47156	2.080000	30	7	0	1	

00-027-1402

May 19, 2020 10:21 AM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 298.0 K **Chemical Formula:** Si
Empirical Formula: Si **Weight %:** Si100.00 **Atomic %:** Si100.00 **Compound Name:** Silicon
Mineral Name: Silicon, syn **CAS Number:** 7440-21-3 **Entry Date:** 09/01/1977 **Modification Date:** 09/01/2008
Modifications: Lambda

Radiation: CuKα1 (1.5406 Å) **Internal Standard:** W **d-Spacing:** Diffractometer **Intensity:** Diffractometer - Peak
I/Ic: 4.7

Crystal System: Cubic **SPGR:** Fd-3m (227)
Author's Unit Cell [a: 5.43088(4) Å **Volume:** 160.18 Å³ **Z:** 8.00 **MolVol:** 20.02 **]**
Calculated Density: 2.329 g/cm³ **Color:** Gray **SS/FOM:** F(11) = 408.8(0.0021, 13)

Space Group: Fd-3m (227) **Molecular Weight:** 28.09 g/mol
Crystal Data [a: 5.431 Å **b:** 5.431 Å **c:** 5.431 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **XtlCell Vol:** 160.18 Å³
XtlCell Z: 8.00 **a/b:** 1.000 **c/b:** 1.000 **]**
Reduced Cell [a: 3.840 Å **b:** 3.840 Å **c:** 3.840 Å **α:** 60.00° **β:** 60.00° **γ:** 60.00° **RedCell Vol:** 40.05 Å³ **]**

Atomic parameters are cross-referenced from PDF entry 04-012-7888 **AC Space Group:** Fd-3m (227)
AC Unit Cell [a: 5.4317(6) Å **b:** 5.4317(6) Å **c:** 5.4317(6) Å **α:** 90° **β:** 90° **γ:** 90° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	11	z,-x+1/4,-y+1/4	21	-y+1/4,z,-x+1/4	31	-x+1/4,-z+1/4,y	41	z,y,x
2	-x,-y,-z	12	-z,x+3/4,y+3/4	22	y+3/4,-z,x+3/4	32	x+3/4,z+3/4,-y	42	-z,-y,-x
3	x,-y+1/4,-z+1/4	13	-z+1/4,x,-y+1/4	23	-y+1/4,-z+1/4,x	33	y,x,z	43	z,-y+1/4,-x+1/4
4	-x,y+3/4,z+3/4	14	z+3/4,-x,y+3/4	24	y+3/4,z+3/4,-x	34	-y,-x,-z	44	-z,y+3/4,x+3/4
5	-x+1/4,y,-z+1/4	15	-z+1/4,-x+1/4,y	25	x,z,y	35	y,-x+1/4,-z+1/4	45	-z+1/4,y,-x+1/4
6	x+3/4,-y,z+3/4	16	z+3/4,x+3/4,-y	26	-x,-z,-y	36	-y,x+3/4,z+3/4	46	z+3/4,-y,x+3/4
7	-x+1/4,-y+1/4,z	17	y,z,x	27	x,-z+1/4,-y+1/4	37	-y+1/4,x,-z+1/4	47	-z+1/4,-y+1/4,x
8	x+3/4,y+3/4,-z	18	-y,-z,-x	28	-x,z+3/4,y+3/4	38	y+3/4,-x,z+3/4	48	z+3/4,y+3/4,-x
9	z,x,y	19	y,-z+1/4,-x+1/4	29	-x+1/4,z,-y+1/4	39	-y+1/4,-x+1/4,z		
10	-z,-x,-y	20	-y,z+3/4,x+3/4	30	x+3/4,-z,y+3/4	40	y+3/4,x+3/4,-z		

Origin: O2

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	IDP	AET
Si	1	8a	-43m	0.125	0.125	0.125	1.0		

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Ceramic (Semiconductor), Common Phase, Educational Pattern, Forensic, Inorganic, Metal & Alloy, Mineral Related (Mineral, Synthetic)

Mineral Classification: Diamond (supergroup), 2C-diamond (group) **Pearson Symbol:** cF8.00
Prototype Structure [Formula Order]: C **Prototype Structure [Alpha Order]:** C
LPF Prototype Structure [Formula Order]: C,cF8,227 **LPF Prototype Structure [Alpha Order]:** C,cF8,227

Cross-Ref PDF #'s: 00-005-0565 (Alternate), 00-026-1481 (Alternate), 04-001-7247 (Primary), 04-002-0118 (Alternate), 04-002-0891 (Alternate), 04-003-1456 (Alternate), 04-003-3352 (Alternate), 04-003-3353 (Alternate), 04-003-4734 (Alternate), 04-004-5099 (Alternate), 04-004-6896 (Alternate), 04-005-9699 (Alternate), 04-006-2527 (Alternate), 04-006-2591 (Alternate), 04-006-4528 (Alternate), 04-006-6436 (Alternate), 04-007-5232 (Alternate), 04-007-8736 (Alternate), 04-012-7888 (Alternate)

References:

Type	DOI	Reference
Primary Reference		Natl. Bur. Stand. (U. S.) Monogr. 25 1976, 13, 35.
Crystal Structure		Crystal Structure Source: LPF.

Database Comments: Additional Patterns: To replace 00-005-0565 and 00-026-1481. General Comments: Reflections calculated from precision measurement of a0. a0 uncorrected for refraction. Sample Source or Locality: This sample is NBS Standard Reference Material No. 640. Temperature of Data Collection: 298(1) K. Unit Cell Data Source: Powder Diffraction.

d-Spacings (11) - Si - 00-027-1402 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
28.44217	**3.135500**	100	1	1	1		88.02612	1.108600	12	4	2	2	
47.30226	**1.920100**	55	2	2	0		94.94771	1.045200	6	5	1	1	
56.12053	**1.637500**	30	3	1	1		106.71500	0.960000	3	4	4	0	
69.13014	1.357700	6	4	0	0		114.08724	0.918000	7	5	3	1	
76.37718	1.245900	11	3	3	1		127.54086	0.858700	8	6	2	0	

00-027-1402 May 19, 2020 10:21 AM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*
136.88996	0.828200	3	5	3	3	

00-029-0285

May 15, 2020 2:12 PM (Steve Simner)

Status Primary **Quality Mark:** Indexed **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca2 Al2 Si O7 ·8 H2 O **Empirical Formula:** Al2 Ca2 H16 O15 Si
Weight %: Al12.90 Ca19.16 H3.86 O57.37 Si6.71 **Atomic %:** Al5.56 Ca5.56 H44.44 O41.67 Si2.78
Compound Name: Calcium Aluminum Silicate Hydrate **Mineral Name:** Strätlingite, syn **Entry Date:** 09/01/1979

Internal Standard: Si

Crystal System: Rhombohedral **Aspect:** R* (148)
Author's Unit Cell [a: 5.747(1) Å **c:** 37.64(1) Å **Volume:** 1076.62 Å³ **Z:** 3.00 **MolVol:** 358.87 **c/a:** 6.550 **]**
Calculated Density: 1.936 g/cm³ **Measured Density:** 1.9 g/cm³ **SS/FOM:** F(25) = 58.5(0.0107, 40)

Space Group: R* **Molecular Weight:** 418.32 g/mol
Crystal Data [a: 5.747 Å **b:** 5.747 Å **c:** 37.640 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00°
XtlCell Vol: 1076.62 Å³ **XtlCell Z:** 3.00 **c/a:** 6.550 **a/b:** 1.000 **c/b:** 6.550 **]**
Reduced Cell [a: 5.747 Å **b:** 5.747 Å **c:** 12.978 Å **α:** 77.21° **β:** 77.21° **γ:** 60.00° **RedCell Vol:** 358.87 Å³ **]**

Sign: =-

Subfiles: Cement and Hydration Product, Inorganic, Mineral Related (Mineral, Synthetic) **Pearson Symbol:** hR36.00
Pearson Symbol w/o H: hR20

References:

Type	DOI	Reference
Primary Reference		Kuzel, H. Neues Jahrb. Mineral., Monatsh. 1976319.
Optical Data		Hentschel, G., Kuzel. Neues Jahrb. Mineral., Monatsh. 1976326.
Powder Data (Additonal References)		Am. Mineral. 1977, 62, 395.

Database Comments: Analysis: Chemical analysis not given. General Comments: Occurs as a mineral at Bellerberg, Mayen/Eifel, Germany (Hentschel, G., Kuzel, Neues Jahrb. Mineral., Monatsh., 326 (1976)). Sample Preparation: Prepared by slow hydration of glasses with the composition "C2 A S" or CAS in saturated lime solutions at 20 C over 120 days. Unit Cell: Powder data indexed by aid of single-crystal data. Warning: Lower quality mark was set by the editor. Unit Cell Data Source: Powder Diffraction.

d-Spacings (25) - Ca2 Al2 Si O7 ·8 H2 O - 00-029-0285 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
7.06588	**12.500000**	100	0	0	3		36.14516	2.483000	10	0	2	1	
14.11342	**6.270000**	40	0	0	6		37.32786	2.407000	10	0	2	4	
17.97783	4.930000	10	1	0	1		37.96595	2.368000	20	1	1	9	
18.43020	4.810000	5	0	1	2		42.61087	2.120000	10	1	1	12	
20.16472	4.400000	10	1	0	4		43.55960	2.076000	<5	0	2	10	
21.23803	**4.180000**	70	0	0	9		44.76169	2.023000	10	0	1	17	
24.36610	3.650000	<5	1	0	7		44.99620	2.013000	<5	2	0	11	
26.01710	3.422000	5	0	1	8		48.18406	1.887000	10	0	2	13	
31.08148	2.875000	20	1	1	0		48.40231	1.879000	5	2	1	1	
31.71501	2.819000	<5	0	1	11		49.38175	1.844000	<5	2	1	4	
31.92424	2.801000	<5	1	1	3		49.93084	1.825000	<5	1	2	5	
34.27608	2.614000	20	1	1	6		50.91529	1.792000	5	0	0	21	
35.84650	2.503000	10	1	0	13								

00-041-0224 Jun 8, 2020 10:47 AM (Steve Simner)

Status Primary **Quality Mark:** Indexed **Environment:** Ambient **Temp:** 295.0 K
Chemical Formula: Ca S O4 ·0.5 H2 O **Empirical Formula:** Ca H O4.5 S **Weight %:** Ca27.61 H0.69 O49.60 S22.09
Atomic %: Ca13.33 H13.33 O60.00 S13.33 **Compound Name:** Calcium Sulfate Hydrate **Mineral Name:** Bassanite, syn
CAS Number: 10034-76-1 **Entry Date:** 09/01/1991

Radiation: CuKα1 (1.5405 Å) **Filter:** Ni Beta **Internal Standard:** Si **d-Spacing:** Diffractometer **Cutoff:** 22.10 Å
Intensity: Visual - Peak

Crystal System: Monoclinic **SPGR:** I2 (5)
Author's Unit Cell [a: 12.028 Å **b:** 6.932 Å **c:** 12.691 Å **β:** 90.183° **Volume:** 1058.15 Å³ **Z:** 12.00
MolVol: 88.18 **c/a:** 1.055 **a/b:** 1.735 **c/b:** 1.831 **]** **Calculated Density:** 2.733 g/cm³
Measured Density: 2.7 g/cm³ **Color:** White **SS/FOM:** F(30) = 27.3(0.0087, 126)

Space Group: I2 (5) **Molecular Weight:** 145.15 g/mol
Crystal Data [a: 12.691 Å **b:** 6.932 Å **c:** 12.028 Å **α:** 90.00° **β:** 90.18° **γ:** 90.00° **XtlCell Vol:** 1058.15 Å³
XtlCell Z: 12.00 **c/a:** 0.948 **a/b:** 1.831 **c/b:** 1.735 **]**
Reduced Cell [a: 6.932 Å **b:** 9.392 Å **c:** 9.418 Å **α:** 79.51° **β:** 68.41° **γ:** 68.34° **RedCell Vol:** 529.07 Å³ **]**

nωβ: =1.558 **εγ:** =1.586 **Sign:** =+

Atomic parameters are cross-referenced from PDF entry 04-013-8392 **AC Space Group:** I121 (5)
AC Unit Cell [a: 12.0275(4) Å **b:** 6.9312(3) Å **c:** 12.6919(5) Å **α:** 90° **β:** 90.18(1)° **γ:** 90° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator
1	x,y,z	2	-x,y,-z

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	IDP	AET
Ca	1	2a	2	0.0	0.4553	0.0	1.0		
Ca	2	2b	2	0.0	0.4553	0.5	1.0		
Ca	3	4c	1	0.2724	0.2724	0.1667	1.0		
Ca	4	4c	1	0.7276	0.2724	0.3333	1.0		
S	5	4c	1	0.7248	0.2752	0.0833	1.0		
S	6	4c	1	0.0	0.4496	0.25	1.0		
S	7	4c	1	0.2752	0.2752	0.4167	1.0		
O	8	4c	1	0.1291	0.726	0.0173	1.0		
O	9	4c	1	0.1291	0.726	0.5173	1.0		
O	10	4c	1	0.2985	0.4435	0.8507	1.0		
O	11	4c	1	0.2985	0.4435	0.3507	1.0		
O	12	4c	1	0.0725	0.3306	0.184	1.0		
O	13	4c	1	0.0725	0.3306	0.684	1.0		
O	14	4c	1	0.2538	0.6185	0.1556	1.0		
O	15	4c	1	0.2538	0.6185	0.6556	1.0		
O	16	4c	1	0.1824	0.3102	0.9889	1.0		
O	17	4c	1	0.1824	0.3102	0.4889	1.0		
O	18	4c	1	0.0639	0.5714	0.8222	1.0		
O	19	4c	1	0.0639	0.5714	0.3222	1.0		
O	20	2b	2	0.5	0.5985	0.0	1.0		
O	21	4c	1	0.5493	0.4508	0.3333	1.0		
H	22	4c	1	0.428	0.671	0.0	1.0		
H	23	4c	1	0.55	0.307	0.333	1.0		
H	24	4c	1	0.621	0.523	0.333	1.0		

Crystal (Symmetry Allowed): Non-centrosymmetric - Enantiomorphic, Optical Activity, Pyro / Piezo (p), Piezo (2nd Harm.)

Subfiles: Cement and Hydration Product, Ceramic (Bioceramic), Common Phase, Inorganic, Mineral Related (Mineral, Synthetic)
Pearson Symbol: mC90.00 **Pearson Symbol w/o H:** mC78 **ANX:** A2B2X9

Cross-Ref PDF #'s: 00-033-0310 (Deleted), 00-036-0617 (Alternate), 04-011-1765 (Primary), 04-013-8392 (Alternate), 04-014-0553 (Primary)

References:

Type	DOI	Reference
Primary Reference		Poellmann, H., Kuzel, H.-J., Mineralogical Inst. of University, Erlangen, Germany. ICDD Grant-in-Aid 1989.
Crystal Structure		Crystal Structure Source: LPF.
Optical Data		Dana's System of Mineralogy, 7th Ed. 1951, II, 476.
Unit Cell		Kuzel, H.-J., Hauner, M. Zem.-Kalk-Gips 1987, 40, 628.

00-041-0224 Jun 8, 2020 10:47 AM (Steve Simner)

Database Comments: Additional Patterns: To replace 00-033-0310 and 00-036-0617. ANX: A2B2X9. Analysis: Analysis (wt.%): CaO 38.5, "S O3" 55.0, "H2 O" 6.5: "Ca S O4 ·0.53 H2 O". Footnotes for HKL and Superlattice D-spacings: *Not permitted by space group. Sample Preparation: Dehydration of gypsum in "H N O3" (60%). Rehydration at pressure of water/pressure of oxygen=0.35 at 22 C. Temperature of Data Collection: 295 K. Warning: One or more lines are unindexed.

d-Spacings (57) - Ca S O4 ·0.5 H2 O - 00-041-0224 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
14.72047	**6.012770**	80	2	0	0		52.70190	1.735380	10	6	2	0	
20.30067	4.370840	5	-2	0	2		52.78183	1.732940	10	0	4	0	
20.74066	4.279100	5	-2	1	1		53.90202	1.699540	5	-1	4	1	
22.24075	3.993760	5	-1	0	3		54.14174	1.692580	20	6	0	4	
23.23077	3.825750	5	-3	0	1		55.10177	1.665340	20	2	4	0	
24.63080	3.611370	5	0	1	3		57.13196	1.610890	5	-2	4	2	
25.67082	3.467370	50	0	2	0		58.42218	1.578350	5	-3	4	1	
27.68088	3.219980	5	-1	2	1		59.77202	1.545890	5	4	2	6	
29.33095	3.042480	10	0	2	2		60.33218	1.532870	5	1	1	8	
29.69099	**3.006400**	100	4	0	0		60.86238	1.520780	5	6	2	4	
31.90109	**2.802980**	90	2	0	4		61.74235	1.501200	5	4	4	0	
32.96109	2.715220	10	4	0	2		62.82211	1.477970	5	-7	1	4	
34.26108	2.615110	5	1	2	3		63.01211	1.473970	10	2	4	4	
34.89122	2.569310	5	3	2	1		63.92226	1.455160	5	-3	3	6	
38.42124	2.340980	5	0	2	4		64.46216	1.444270	5	-3	1	8	
39.65142	2.271140	5	4	2	0		64.61210	1.441280	5	3	1	8	
40.47133	2.227000	5	3	2	3		65.34226	1.426930	5	-6	3	3	
41.34137	2.182120	5	-2	2	4		66.87223	1.397950	5	-5	4	1	
42.25143	2.137200	10	4	2	2		68.96253	1.360590	5	-8	0	4	
42.71146	2.115240	10	0	0	6		71.97260	1.310910	10	6	4	0	
44.64143	2.028170	5	0	3	3		72.73287	1.299070	10	4	2	8	
45.26163	2.001810	5	3	3	0		75.11299	1.263700	10	8	2	4	
46.45164	1.953260	5	-5	2	1		76.61311	1.242650	10	3	3	8	
47.61171	1.908340	10	6	0	2		78.76313	1.214030	5	7	4	1	
49.24159	1.848920	20	-4	2	4		79.99302	1.198430	5	4	3	8	*
49.36175	1.844700	30	4	2	4		81.42360	1.180950	5	10	0	2	
50.29164	1.812750	5	4	3	1		82.43287	1.169030	5	6	2	8	
51.01168	1.788840	5	5	2	3		83.48339	1.156970	5	9	3	0	
52.19194	1.751130	5	6	1	3								

00-044-0448 Jun 1, 2020 6:19 PM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 298.0 K
Chemical Formula: Ca4 Fe2 O6 (S O3) ·12 H2 O **Structural Formula:** 3 Ca O · Fe2 O3 · Ca S O3 ·12 H2 O
Empirical Formula: Ca4 Fe2 H24 O21 S **Weight %:** Ca24.14 Fe16.82 H3.64 O50.58 S4.83
Atomic %: Ca7.69 Fe3.85 H46.15 O40.38 S1.92 **Compound Name:** Calcium Iron Oxide Sulfite Hydrate
Entry Date: 09/01/1994

Radiation: CuKα1 (1.5405 Å) **Filter:** Ni Beta **Internal Standard:** Si **d-Spacing:** Diffractometer **Cutoff:** 9.80 Å
Intensity: Diffractometer - Peak **I/Ic:** 1.2

Crystal System: Rhombohedral **SPGR:** R-3c (167)
Author's Unit Cell [a: 5.903 Å **c:** 50.531 Å **Volume:** 1524.87 Å³ **Z:** 3.00 **MolVol:** 508.29 **c/a:** 8.560 **]**
Calculated Density: 2.17 g/cm³ **Color:** Colorless **SS/FOM:** F(25) = 42.6(0.0100, 59)

Space Group: R-3c (167) **Molecular Weight:** 664.24 g/mol
Crystal Data [a: 5.903 Å **b:** 5.903 Å **c:** 50.531 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00°
XtlCell Vol: 1524.87 Å³ **XtlCell Z:** 3.00 **c/a:** 8.560 **a/b:** 1.000 **c/b:** 8.560 **]**
Reduced Cell [a: 5.903 Å **b:** 5.903 Å **c:** 17.185 Å **α:** 80.11° **β:** 80.11° **γ:** 60.00° **RedCell Vol:** 508.29 Å³ **]**

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Inorganic **Pearson Symbol:** hR52.00 **Pearson Symbol w/o H:** hR28

References:

Type	DOI	Reference
Primary Reference		Poellmann, H., Ecker, M., Friedrich-Alexander Univ., Erlangen-Nurnberg, Erlangen, Germany. ICDD Grant-in-Aid 1993.

Database Comments: Analysis: Chemical analysis (wt.%): CaO 33.52, "Fe2 O3" 23.80, "S O2" 9.51, "H2 O" 32.15. General Comments: 35% relative humidity. Sample Preparation: Prepared by mixing "Fe (O H)3", CaO and "Na2 S O3" in stoichiometric ratio with excess water at 60 C. Temperature of Data Collection: 298 K.

d-Spacings (25) - Ca4 Fe2 O6 (S O3) ·12 H2 O - 00-044-0448 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
10.51034	**8.409950**	100	0	0	6		44.50155	2.034220	15	1	1	18	
21.09067	**4.208870**	45	0	0	12		49.29163	1.847160	4	1	2	8	
22.36073	**3.972600**	27	0	1	8		50.19184	1.816120	3	0	1	26	
30.26096	2.951060	27	1	1	0		50.75175	1.797390	10	2	0	20	
32.10111	2.785970	17	1	1	6		53.41203	1.713970	2	1	1	24	
34.28108	2.613630	6	1	1	9		53.73186	1.704520	17	3	0	0	
35.82119	2.504710	8	0	2	4		54.92182	1.670370	12	3	0	6	
37.18131	2.416150	26	1	1	12		58.32210	1.580820	5	1	1	27	
37.96129	2.368280	9	2	0	8		60.23199	1.535180	4	1	2	20	
39.76142	2.265110	14	0	1	20		61.40211	1.508700	3	0	1	32	
40.61144	2.219640	2	1	1	15		62.94214	1.475440	5	2	2	0	
43.14141	2.095150	3	1	0	22		63.98227	1.453940	4	2	2	6	
43.33146	2.086400	3	2	0	14								

00-044-1481

May 15, 2020 3:20 PM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca (O H)2 **Empirical Formula:** Ca H2 O2 **Weight %:** Ca54.09 H2.72 O43.19
Atomic %: Ca20.00 H40.00 O40.00 **Compound Name:** Calcium Hydroxide **Mineral Name:** Portlandite, syn
CAS Number: 1305-62-0 **Entry Date:** 09/01/1994

Radiation: CuKα1 (1.5406 Å) **Filter:** Graph Mono **Internal Standard:** Si **d-Spacing:** Diffractometer **Cutoff:** 15.00 Å
Intensity: Diffractometer - Peak **I/Ic:** 2.9

Crystal System: Hexagonal **SPGR:** P-3m1 (164)
Author's Unit Cell [a: 3.5899(4) Å **c:** 4.916(3) Å **Volume:** 54.87 Å³ **Z:** 1.00 **MolVol:** 54.87 **c/a:** 1.369 **]**
Calculated Density: 2.242 g/cm³ **Color:** White **SS/FOM:** F(25) = 51.8(0.0167, 29)

Space Group: P-3m1 (164) **Molecular Weight:** 74.09 g/mol
Crystal Data [a: 3.590 Å **b:** 3.590 Å **c:** 4.916 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **XtlCell Vol:** 54.87 Å³
XtlCell Z: 1.00 **c/a:** 1.369 **a/b:** 1.000 **c/b:** 1.369 **]**
Reduced Cell [a: 3.590 Å **b:** 3.590 Å **c:** 4.916 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **RedCell Vol:** 54.87 Å³ **]**

Atomic parameters are cross-referenced from PDF entry 04-006-9147 **AC Space Group:** P-3m1 (164)
AC Unit Cell [a: 3.5918(66) Å **b:** 3.5918(66) Å **c:** 4.9063(96) Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	3	-y,x-y,z	5	-x+y,-x,z	7	-y,-x,z	9	x,x-y,z	11	-x+y,y,z
2	-x,-y,-z	4	y,-x+y,-z	6	x-y,x,-z	8	y,x,-z	10	-x,-x+y,-z	12	x-y,-y,-z

ADP Type: U

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Uiso	AET
Ca	1	1a	-3m.	0.0	0.0	0.0	1.0	0.01194	
O	2	2d	3m.	0.33333	0.66666	0.234	1.0	0.01203	
H	3	2d	3m.	0.33333	0.66666	0.4256	1.0	0.04203	

Anisotropic Displacement Parameters:

Atom	Num	Uani11	Uani22	Uani33	Uani12	Uani13	Uani23
Ca	1	0.0083	0.0083	0.0193	0.0042	0.0	0.0
O	2	0.0106	0.0106	0.0149	0.0053	0.0	0.0
H	3	0.0528	0.0528	0.0205	0.0264	0.0	0.0

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Cement and Hydration Product, Common Phase, Forensic, Inorganic, Mineral Related (Mineral, Synthetic), Pharmaceutical (Excipient)

Mineral Classification: Brucite (group), hydroxide (subgroup) **Pearson Symbol:** hP5.00 **Pearson Symbol w/o H:** hP3

Cross-Ref PDF #'s: 04-006-9147 (Alternate), 04-006-9148 (Alternate), 04-006-9149 (Alternate), 04-006-9150 (Alternate), 04-006-9151 (Alternate), 04-006-9152 (Alternate), 04-007-5231 (Alternate), 04-008-0220 (Alternate), 04-010-3117 (Primary)

References:

Type	DOI	Reference
Primary Reference		Martin, K., McCarthy, G., North Dakota State University, Fargo, North Dakota, USA. ICDD Grant-in-Aid 1992.
Crystal Structure		Crystal Structure Source: LPF.
Optical Data		Winchell, A., Winchell, H. Microscopic Character of Artificial Inorg. Solid Sub. 196469.

Database Comments: Additional Patterns: Validated by a calculated pattern. General Comments: Average relative standard deviation in intensity of the ten strongest reflections for three specimen mounts = 2.2%. Astringent. Sample Source or Locality: Sample obtained from Sigma Chemical Co. Unit Cell Data Source: Powder Diffraction.

d-Spacings (28) - Ca (O H)2 - 00-044-1481 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
18.00730	**4.922000**	72	0	0	1		62.63192	1.482000	9	2	0	1	
28.67094	3.111000	27	1	0	0		64.23143	1.448900	7m	1	0	3	
34.10125	**2.627000**	100	1	0	1		64.23143	1.448900	7m	1	1	2	
36.52569	2.458000	1	0	0	2		71.80861	1.313500	6	2	0	2	
47.12003	1.927100	30	1	0	2		77.65202	1.228600	1	0	0	4	
50.81200	**1.795400**	31	1	1	0		79.09241	1.209800	2	1	1	3	
54.35646	1.686400	14	1	1	1		81.90694	1.175200	2	2	1	0	
56.09070	1.638300	1	0	0	3		84.74839	1.142900	5m	1	0	4	
59.42442	1.554100	3	2	0	0		84.74839	1.142900	5m	2	1	1	

00-044-1481 May 15, 2020 3:20 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
86.19403	1.127400	2	2	0	3		103.13890	0.983300	<1	0	0	5	
93.20601	1.060100	3	2	1	2		106.06233	0.964100	1	2	0	4	
96.02628	1.036300	2	3	0	0		107.57460	0.954700	2	2	1	3	
98.87902	1.013900	2m	1	1	4		110.51638	0.937390	<1	1	0	5	
98.87902	1.013900	2m	3	0	1		118.26283	0.897400	1	2	2	0	

00-045-0572 May 15, 2020 2:58 PM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 298.0 K
Chemical Formula: Ca8 Al2 Fe2 O12 C O3 (O H)2 ·22 H2 O
Structural Formula: 6 Ca O · Al2 O3 · Fe2 O3 · Ca C O3 · Ca (O H)2 ·22 H2 O
Empirical Formula: Al2 C Ca8 Fe2 H46 O39 **Weight %:** Al4.62 C1.03 Ca27.44 Fe9.56 H3.97 O53.39
Atomic %: Al2.04 C1.02 Ca8.16 Fe2.04 H46.94 O39.80
Compound Name: Calcium Aluminum Iron Oxide Carbonate Hydroxide Hydrate **Entry Date:** 09/01/1995

Radiation: CuKα1 (1.5405 Å) **Filter:** Ni Beta **Internal Standard:** Si **Cutoff:** 17.70 Å **Intensity:** Integrated
I/Ic: 1.5

Crystal System: Rhombohedral **SPGR:** R3c (161)
Author's Unit Cell [a: 5.882 Å **c:** 47.934 Å **Volume:** 1436.23 Å³ **Z:** 1.50 **MolVol:** 957.49 **c/a:** 8.149 **]**
Calculated Density: 2.027 g/cm³ **Color:** Colorless **SS/FOM:** F(21) = 63.9(0.0059, 56)

Space Group: R3c (161) **Molecular Weight:** 1168.61 g/mol
Crystal Data [a: 5.882 Å **b:** 5.882 Å **c:** 47.934 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00°
XtlCell Vol: 1436.23 Å³ **XtlCell Z:** 1.50 **c/a:** 8.149 **a/b:** 1.000 **c/b:** 8.149 **]**
Reduced Cell [a: 5.882 Å **b:** 5.882 Å **c:** 16.335 Å **α:** 79.63° **β:** 79.63° **γ:** 60.00° **RedCell Vol:** 478.74 Å³ **]**

Crystal (Symmetry Allowed): Non-centrosymmetric - Pyro / Piezo (p), Piezo (2nd Harm.)

Subfiles: Inorganic **Pearson Symbol:** hR49.00 **Pearson Symbol w/o H:** hR26

References:

Type	DOI	Reference
Primary Reference		Ecker, M., Poellmann, H., Friedrich-Alexander-Univ. Erlangen-Nurnberg, Germany. ICDD Grant-in-Aid 1994.

Database Comments: Analysis: Chemical analysis (wt.%): CaO 37.8, "Fe2 O3" 13.4, "Al2 O3" 8.6, "C O2" + "H2 O" 40.1. General Comments: 35% relative humidity. Sample Preparation: Prepared by mixing "Fe (O H)3", "Al (O H)3", CaO and "Na2 C O3" in molar equation with excess water at 25 C. Temperature of Data Collection: 298 K.

d-Spacings (21) - Ca8 Al2 Fe2 O12 C O3 (O H)2 ·22 H2 O - 00-045-0572 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
11.06036	**7.992940**	100	0	0	6		45.93154	1.974160	6	1	1	18	
22.23071	**3.995540**	30	0	0	12		52.37175	1.745540	4	2	0	20	
22.90075	**3.880130**	12	0	1	8		52.77199	1.733240	3	0	1	26	
30.37098	2.940620	6	1	1	0		53.95178	1.698090	4	3	0	0	
32.41108	2.760030	4	1	1	6		55.25189	1.661170	5	3	0	6	
34.81110	2.575040	5	1	1	9		59.06195	1.562770	2	3	0	12	
36.02120	2.491260	3	0	2	4		61.75239	1.500980	3	1	2	20	
37.95130	2.368880	7	1	1	12		62.11203	1.493150	3	2	0	26	
38.37133	2.343910	4	2	0	8		64.38222	1.445870	2	2	2	6	
41.61143	2.168580	8	0	1	20		64.81235	1.437310	2	0	1	32	
44.29146	2.043380	2	2	0	14								

00-046-1045

May 15, 2020 1:20 PM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 296.0 K **Chemical Formula:** Si O2
Empirical Formula: O2 Si **Weight %:** O53.26 Si46.74 **Atomic %:** O66.67 Si33.33 **Compound Name:** Silicon Oxide
Mineral Name: Quartz, syn **Entry Date:** 09/01/1996

Radiation: CuKα1 (1.5406 Å) **Filter:** Ge Mono **Internal Standard:** Si **d-Spacing:** Diffractometer
Intensity: Diffractometer - Integrated **I/Ic:** 3.41

Crystal System: Hexagonal **SPGR:** P3221 (154)
Author's Unit Cell [a: 4.91344(4) Å **c:** 5.40524(8) Å **Volume:** 113.01 Å³ **Z:** 3.00 **MolVol:** 37.67 **c/a:** 1.100 **] Calculated Density:** 2.649 g/cm³ **Measured Density:** 2.66 g/cm³ **Color:** White
SS/FOM: F(30) = 538.7(0.0018, 31)

Space Group: P3221 (154) **Molecular Weight:** 60.08 g/mol
Crystal Data [a: 4.913 Å **b:** 4.913 Å **c:** 5.405 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **XtlCell Vol:** 113.01 Å³
XtlCell Z: 3.00 **c/a:** 1.100 **a/b:** 1.000 **c/b:** 1.100 **]**
Reduced Cell [a: 4.913 Å **b:** 4.913 Å **c:** 5.405 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **RedCell Vol:** 113.01 Å³ **]**

nωβ: =1.544 **εγ:** =1.553 **Sign:** =+

Atomic parameters are cross-referenced from PDF entry 04-012-0490 **AC Space Group:** P3221 (154)
AC Unit Cell [a: 4.91427(12) Å **b:** 4.91427(12) Å **c:** 5.4058(2) Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	3	-x+y,-x,z+1/3	5	-x,-x+y,-z+2/3
2	-y,x-y,z+2/3	4	y,x,-z	6	x-y,-y,-z+1/3

ADP Type: U

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Uiso	AET
Si	1	3a	.2.	0.4723	0.0	0.66666	1.0	0.00754	
O	2	6c	1	0.416	0.2658	0.7881	1.0	0.01747	

Anisotropic Displacement Parameters:

Atom	Num	Uani11	Uani22	Uani33	Uani12	Uani13	Uani23
Si	1	0.0075	0.0095	0.0063	0.00475	-0.002	-4.0E-5
O	2	0.026	0.02	0.0124	0.016	-0.003	-5.0E-5

Crystal (Symmetry Allowed): Non-centrosymmetric

Subfiles: Cement and Hydration Product, Common Phase, Forensic, Inorganic, Metal & Alloy, Mineral Related (Mineral, Synthetic), Pharmaceutical (Excipient)

Mineral Classification: Quartz (supergroup), Class Member **Pearson Symbol:** hP9.00
Prototype Structure [Formula Order]: Si O2 **Prototype Structure [Alpha Order]:** O2 Si
LPF Prototype Structure [Formula Order]: Si O2,hP9,152 **LPF Prototype Structure [Alpha Order]:** O2 Si,hP9,152

Cross-Ref PDF #'s: 00-033-1161 (Alternate), 01-085-0335 (Alternate), 04-005-4494 (Alternate), 04-012-0490 (Alternate)

References:

Type	DOI	Reference
Primary Reference		Kern, A., Eysel, W., Mineralogisch-Petrograph. Inst., Univ. Heidelberg, Germany. ICDD Grant-in-Aid 1993.
Crystal Structure		Crystal Structure Source: LPF.
Optical Data		Swanson, Fuyat. Natl. Bur. Stand. (U. S.), Circ. 539 1954, 3, 24.
Structure		Z. Kristallogr. 1992, 198, 177.

Database Comments: Additional Patterns: To replace 00-033-1161. See PDF 01-085-0335. General Comments: Low temperature quartz. 2θ determination based on profile fit method. Temperature of Data Collection: 296(1) K. Unit Cell Data Source: Powder Diffraction.

d-Spacings (58) - Si O2 - 00-046-1045 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
20.85950	**4.254990**	16	1	0	0		50.62056	1.801740	<1	0	0	3	
26.63929	**3.343470**	100	1	0	1		54.87338	1.671730	4	2	0	2	
36.54309	2.456870	9	1	1	0		55.32347	1.659190	2	1	0	3	
39.46405	2.281490	8	1	0	2		57.23362	1.608270	<1	2	1	0	
40.29891	2.236130	4	1	1	1		59.95837	1.541530	9	2	1	1	
42.44898	2.127710	6	2	0	0		64.03401	1.452890	2	1	1	3	
45.79177	1.979860	4	2	0	1		65.78421	1.418410	<1	3	0	0	
50.13753	**1.817960**	13	1	1	2		67.74212	1.382100	6	2	1	2	

00-046-1045 May 15, 2020 1:20 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
68.14202	1.374960	7	2	0	3		104.19936	0.976174	<1	3	2	0	
68.31610	1.371880	5	3	0	1		106.58922	0.960785	<1	3	2	1	
73.46578	1.287910	2	1	0	4		112.10978	0.928526	<1	4	1	0	
75.65768	1.255950	3	3	0	2		114.05664	0.918159	<1	3	2	2	
77.67304	1.228320	1	2	2	0		114.46250	0.916060	2	4	0	3	
79.88168	1.199820	2	2	1	3		114.63466	0.915176	2	4	1	1	
80.04440	1.197790	<1	2	2	1		115.88056	0.908890	<1	2	2	4	
81.17066	1.183990	2	1	1	4		117.53232	0.900851	<1	0	0	6	
81.48879	1.180170	2	3	1	0		118.30814	0.897188	<1	2	1	5	
83.83772	1.152980	1	3	1	1		120.11906	0.888910	1	3	1	4	
84.95477	1.140650	<1	2	0	4		121.84790	0.881352	<1	1	0	6	
87.43656	1.114550	<1	3	0	3		122.59978	0.878167	<1	4	1	2	
90.82841	1.081550	2	3	1	2		127.24516	0.859796	<1	3	0	5	
92.78531	1.063800	<1	4	0	0		131.19674	0.845837	<1	1	1	6	
94.64763	1.047720	1	1	0	5		132.74954	0.840746	<1	5	0	1	
95.11542	1.043800	<1	4	0	1		134.28623	0.835918	<1	4	0	4	
96.23447	1.034610	1	2	1	4		136.41685	0.829560	1	2	0	6	
98.74719	1.014900	1	2	2	3		137.88751	0.825393	2	4	1	3	
102.22753	0.989576	<1	1	1	5		140.31020	0.818911	<1	3	3	0	
102.56347	0.987246	<1	3	1	3		143.24225	0.811682	3	5	0	2	
103.87331	0.978345	<1	3	0	4		144.11007	0.809668	<1	3	3	1	

00-047-1144

May 18, 2020 4:08 PM (Steve Simner)

Status Primary **Quality Mark:** Indexed **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Phase: β **Chemical Formula:** Si O2 **Empirical Formula:** O2 Si **Weight %:** O53.26 Si46.74
Atomic %: O66.67 Si33.33 **Compound Name:** Silicon Oxide **Alternate Name:** β-quartz **Entry Date:** 09/01/1997
Modification Date: 09/01/2003 **Modifications:** Quality

Internal Standard: SiO2 **d-Spacing:** Diffractometer **Intensity:** Diffractometer - Peak

Crystal System: Hexagonal **SPGR:** P6222 (180)
Author's Unit Cell [a: 4.996 Å **c:** 5.453 Å **Volume:** 117.87 Å³ **Z:** 3.00 **MolVol:** 39.29 **c/a:** 1.091 **]**
Calculated Density: 2.539 g/cm³ **SS/FOM:** F(11) = 29.5(0.0286, 13)

Space Group: P6222 (180) **Molecular Weight:** 60.08 g/mol
Crystal Data [a: 4.996 Å **b:** 4.996 Å **c:** 5.453 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **XtlCell Vol:** 117.87 Å³
XtlCell Z: 3.00 **c/a:** 1.091 **a/b:** 1.000 **c/b:** 1.091 **]**
Reduced Cell [a: 4.996 Å **b:** 4.996 Å **c:** 5.453 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **RedCell Vol:** 117.87 Å³ **]**

Atomic parameters are cross-referenced from PDF entry 04-007-1808 **AC Space Group:** P6222 (180)
AC Unit Cell [a: 4.9977 Å **b:** 4.9977 Å **c:** 5.4601 Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	4	-x,-y,z	7	y,x,-z+2/3	10	-y,-x,-z+2/3
2	-y,x-y,z+2/3	5	y,-x+y,z+2/3	8	-x,-x+y,-z+1/3	11	x,x-y,-z+1/3
3	-x+y,-x,z+1/3	6	x-y,x,z+1/3	9	x-y,-y,-z	12	-x+y,y,-z

ADP Type: U

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Uiso	AET
Si	1	3c	222	0.5	0.0	0.0	1.0	0.01974	
O	2	12k	1	0.4164	0.2381	0.1419	0.5	0.03959	

Anisotropic Displacement Parameters:

Atom	Num	Uani11	Uani22	Uani33	Uani12	Uani13	Uani23
Si	1	0.0227	0.0184	0.0167	0.0092	0.0	0.0
O	2	0.0529	0.0374	0.0381	0.0298	-0.0064	-0.0174

Crystal (Symmetry Allowed): Non-centrosymmetric - Enantiomorphic, Optical Activity, Piezo (2nd Harm.)

Subfiles: Inorganic, Metal & Alloy, Pharmaceutical (Excipient) **Pearson Symbol:** hP9.00
Prototype Structure [Formula Order]: Si O2 **Prototype Structure [Alpha Order]:** O2 Si
LPF Prototype Structure [Formula Order]: Si O2,hP9,180 **LPF Prototype Structure [Alpha Order]:** O2 Si,hP9,180

Cross-Ref PDF #'s: 04-007-1808 (Alternate)

References:

Type	DOI	Reference
Primary Reference		Liu, X., Su, W., Zhao, X., Wang, Y. Mater.Lett. 1993, 18, 234.
Crystal Structure		Crystal Structure Source: LPF.

Database Comments: Sample Preparation: ZSM-5 zeolites, heated for 5 hours at 400 C to remove water and organics, were put into the high-pressure chamber and the pressure was brought to 2.50 GPa. Then the temperature was increased to 800 C. After being held at that temperature for 20 minutes, the specimens were quenched to room temperature under pressure and the pressure released. Warning: Not enough reflections above the intensity cut off to meet higher quality mark criteria. Unit Cell Data Source: Powder Diffraction.

d-Spacings (11) - Si O2 - 00-047-1144 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
20.50857	**4.327000**	20	1	0	0		49.41033	1.843000	11	1	1	2	
26.24342	**3.393000**	100	0	1	1		54.02367	1.696000	8	0	2	2	
35.90583	2.499000	18	1	1	0		54.68650	1.677000	7	1	0	3	
38.99230	2.308000	16	1	0	2		56.17654	1.636000	6	2	1	0	
41.68344	2.165000	14	2	0	0		58.92810	**1.566000**	19	2	1	1	
44.99620	2.013000	12	0	2	1								

00-055-0738

Jun 29, 2020 3:05 PM (Steve Simner)

Status Primary **Quality Mark:** Indexed **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca3 Si O5 **Empirical Formula:** Ca3 O5 Si **Weight %:** Ca52.66 O35.04 Si12.30
Atomic %: Ca33.33 O55.56 Si11.11 **Compound Name:** Calcium Silicate **Alternate Name:** Alite M1; C3S
Entry Date: 09/01/2005 **Modification Date:** 09/01/2017 **Modifications:** FQM

Radiation: CuKα1 (1.5405 Å) **d-Spacing:** Calculated **Intensity:** Calculated

Crystal System: Monoclinic **SPGR:** Pc (7)
Author's Unit Cell [a: 27.8736(2) Å **b:** 7.0590(5) Å **c:** 12.2575(8) Å **β:** 116.030(6)° **Volume:** 2167.14 Å³
Z: 18.00 **MolVol:** 120.40 **c/a:** 0.440 **a/b:** 3.949 **c/b:** 1.736 **]** **Calculated Density:** 3.149 g/cm³
SS/FOM: F(30) = 478.4(0.0005, 121)

Space Group: Pc (7) **Molecular Weight:** 228.31 g/mol
Crystal Data [a: 25.046 Å **b:** 7.059 Å **c:** 12.257 Å **α:** 90.00° **β:** 90.06° **γ:** 90.00°
XtlCell Vol: 2167.14 Å³ **XtlCell Z:** 18.00 **c/a:** 0.489 **a/b:** 3.548 **c/b:** 1.736 **]**
Reduced Cell [a: 7.059 Å **b:** 12.257 Å **c:** 25.046 Å **α:** 90.06° **β:** 90.00° **γ:** 90.00° **RedCell Vol:** 2167.14 Å³ **]**

Crystal (Symmetry Allowed): Non-centrosymmetric - Optical Activity, Pyro / Piezo (p), Piezo (2nd Harm.)

Subfiles: Cement and Hydration Product, Ceramic, Inorganic **Pearson Symbol:** mP162.00

Cross-Ref PDF #'s: 00-049-0442 (Primary), 00-055-0739 (Primary), 00-055-0740 (Primary), 04-018-9701 (Primary)

References:

Type	DOI	Reference
Primary Reference		de Noirfontaine, M.-N., CNRS-CECM, Vitry-sur-Seine, France. Private Communication 2003.
Unit Cell		de Noirfontaine, M.-N., Dunstetter, F., Courtial, M., Gasecki, G., Signes-Frehel, M. "Polymorphism of tricalcium silicate in portland cement: a fast visual identification of structure and superstructure." Powder Diffr. 2003, 18, 7.

Database Comments: Additional Patterns: See 00-049-0442, 00-055-0739 and 00-055-0740. Processing Information: Rietveld refinement. Warning: One or more lines are unindexed.

d-Spacings (112) - Ca3 Si O5 - 00-055-0738 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
14.86534	5.954500	13	1	0	-2		35.68290	2.514100	10	-10	1	3	
14.87237	5.951700	24	3	0	-2		36.31920	2.471500	9	2	0	4	
14.89351	5.943300	46	0	1	1		36.35573	2.469100	8	-10	0	4	
16.10837	5.497700	12	1	1	1		36.46426	2.462000	12	6	2	1	
16.11456	5.495600	8	3	1	-1		36.64300	2.450400	29	9	1	-4	
22.89854	3.880500	15	3	0	2		37.67198	2.385800	8	7	1	2	
22.92189	3.876600	17	7	0	-2		37.69821	2.384200	11	-11	1	2	
22.93388	3.874600	50	6	1	-1		38.21564	2.353100	12	0	3	0	
23.89750	3.720500	14	6	1	-2		38.39022	2.342800	8	1	3	0	
25.16287	3.536200	37	3	1	-3		38.63187	2.328700	70	3	0	4	
25.21070	3.529600	11	0	2	0		38.70618	2.324400	129	9	2	-2	
25.41270	3.502000	29	2	1	-3		38.82255	2.317700	22	-12	0	2	
25.41934	3.501100	23	4	1	-3		39.03101	2.305800	8	6	1	-5	
27.21868	3.273600	8	3	2	-1		39.05745	2.304300	18	3	2	-4	
27.35753	3.257300	8	0	1	3		39.11220	2.301200	11	0	3	1	
27.41158	3.251000	11	3	2	0		39.11751	2.300900	14	7	2	1	
27.93289	3.191500	8	7	1	0		39.53426	2.277600	22	3	1	-5	
29.33470	3.042100	284	3	0	-4		39.76526	2.264900	12	3	3	0	
29.39595	3.035900	590	3	2	-2		40.03054	2.250500	14	3	2	3	
30.00241	2.975900	75	6	0	-4		40.40695	2.230400	8	1	2	-4	
30.04684	2.971600	155	0	2	2		41.20748	2.188900	84	-12	0	4	
32.06293	2.789200	8	6	0	2		41.21733	2.188400	272	6	2	2	
32.09602	2.786400	12	9	1	-1		41.22718	2.187900	44	6	1	3	
32.13629	**2.783000**	1000	9	0	0		41.23900	2.187300	21	-10	2	2	
32.48885	2.753600	405	0	0	4		41.26464	2.186000	50	-12	1	3	
32.51555	2.751400	24	8	0	-4		41.27649	2.185400	43	9	2	0	
32.54716	2.748800	8	2	2	2		41.52687	2.172800	11	1	1	-5	
32.55933	**2.747800**	673	6	2	-2		41.55488	2.171400	22	9	1	-5	
33.15982	2.699400	18	3	1	3		41.56089	2.171100	71	0	2	4	
33.18891	2.697100	13	9	1	-3		41.62107	2.168100	53	3	3	1	
33.21172	2.695300	28	6	2	0		42.52252	2.124200	9	5	3	-2	
33.72033	2.655800	8	4	2	-3		42.97450	2.102900	18	0	1	5	
34.29096	2.612900	311	9	0	-4		43.03465	2.100100	21	9	2	-4	
34.30449	2.611900	19	5	2	-3		43.07556	2.098200	14	6	3	-1	
34.31533	**2.611100**	627	3	2	2		43.63472	2.072600	12	6	3	-2	
34.33159	2.609900	32	7	2	-2		44.38937	2.039100	14	3	3	-3	

00-055-0738 Jun 29, 2020 3:05 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
45.58884	1.988200	8	3	2	-5		59.83557	1.544400	48	-18	0	4	
45.66892	1.984900	24	3	0	-6		59.85265	1.544000	9	-16	1	6	
45.69080	1.984000	16	9	0	-6		60.61031	1.526500	19	3	4	-4	
45.76148	1.981100	15	0	3	3		60.98570	1.518000	10	6	4	-4	
45.77125	1.980700	54	6	3	-3		62.11896	1.493000	114	3	2	6	
46.78031	1.940300	51	6	0	4		62.17449	1.491800	123	-15	2	6	
46.85451	1.937400	117	-12	2	2		62.23940	1.490400	126	9	4	0	
49.61718	1.835800	14	0	0	6		62.35101	1.488000	40	-12	0	8	
49.73288	1.831800	34	9	3	-3		62.45370	1.485800	83	0	4	4	
49.87537	1.826900	23	9	2	2		63.41367	1.465600	38	3	0	-8	
49.87828	1.826800	13	-15	0	4		63.56379	1.462500	81	9	4	-4	
51.65384	1.768100	378	6	2	-6		63.77327	1.458200	8	-15	3	3	
51.75758	1.764800	211	0	4	0		66.41288	1.406500	8	3	4	4	
55.80163	1.646100	10	-15	0	6		67.12493	1.393300	12	-18	2	2	
55.83483	1.645200	26	6	3	3		67.22330	1.391500	51	18	0	0	
55.86067	1.644500	8	-12	3	3		68.03848	1.376800	15	0	0	8	
56.14665	1.636800	8	6	4	-2		68.20182	1.373900	20	-12	4	4	
56.33401	1.631800	113	9	0	4		70.92376	1.327700	14	15	2	2	
56.40932	1.629800	161	-15	2	2		72.31141	1.305600	13	6	4	4	
59.82703	1.544600	125	12	2	2		79.04552	1.210400	14	-8	5	5	

00-060-0312

Jun 1, 2020 6:23 PM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: 3 Ca O · Al2 O3 ·0.17 Ca S O4 ·0.5 Ca (O H)2 ·0.33 Ca C O3 ·x H2 O
Empirical Formula: Al2 C0.33 Ca4 H3 O9.67 S0.17 **Weight %:** Al14.15 C1.04 Ca42.03 H0.79 O40.56 S1.43
Atomic %: Al10.43 C1.72 Ca20.87 H15.65 O50.44 S0.89
Compound Name: Calcium Aluminum Oxide Carbonate Sulfate Hydroxide Hydrate **Entry Date:** 09/01/2010

Internal Standard: Si **d-Spacing:** Diffractometer **Intensity:** Diffractometer - Peak

Crystal System: Rhombohedral **Aspect:** R* (148)
Author's Unit Cell [a: 5.771 Å **c:** 49.52 Å **Volume:** 1428.28 Å³ **c/a:** 8.581 **] SS/FOM:** F(23) = 20.1(0.0155, 74)

Space Group: R* **Molecular Weight:** 381.42 g/mol
Crystal Data [a: 5.771 Å **b:** 5.771 Å **c:** 49.520 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00°
XtlCell Vol: 1428.28 Å³ **c/a:** 8.581 **a/b:** 1.000 **c/b:** 8.581 **]**
Reduced Cell [a: 5.771 Å **b:** 5.771 Å **c:** 16.839 Å **α:** 80.13° **β:** 80.13° **γ:** 60.00° **RedCell Vol:** 476.09 Å³ **]**

Subfiles: Cement and Hydration Product, Inorganic **Pearson Symbol:** hR?

References:

Type	DOI	Reference
Primary Reference		Poellmann, H. "Syntheses, properties and solid solution of ternary lamellar calcium aluminate hydroxi salts (AFm-phases) containing \S O4\2-, \C O3\2- and \O H\-." Neues Jahrb. Mineral., Abh. 2006, 182, 173.

Database Comments: Analysis: Chemical analysis (wt.%): "Ca O" 39.1, "Al2 O3" 18.2, "S O3" 2.0, "C O2" 2.4, "H2 O" 40.1. Sample Preparation: Prepared from a mixture of tricalciumaluminate, gypsum, lime and calcite. Unit Cell Data Source: Powder Diffraction.

d-Spacings (23) - 3 Ca O · Al2 O3 ·0.17 Ca S O4 ·0.5 Ca (O H)2 ·0.33 Ca C O3 ·x H2 O - 00-060-0312 (Stick, Fixed Slit Intensity) - Cu

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
10.70170	**8.260000**	100	0	0	6		40.39561	2.231000	10	0	2	10	
21.49818	**4.130000**	80	0	0	12		40.60456	2.220000	20	0	1	20	
22.84186	3.890000	30	0	1	8		43.80360	2.065000	15	0	0	24	
25.28789	3.519000	5	1	0	10		44.05043	2.054000	20	1	0	22	
30.97104	**2.885000**	60	1	1	0		45.49699	1.992000	20	1	1	18	
32.49613	2.753000	25	0	0	18		46.66056	1.945000	10	0	2	16	
32.87665	2.722000	10	1	1	6		51.94095	1.759000	10	2	0	20	
34.02118	2.633000	10	1	0	16		55.07809	1.666000	10	3	0	0	
35.07879	2.556000	30	1	1	9		55.58504	1.652000	10	0	0	30	
36.66469	2.449000	20	0	2	4		56.28892	1.633000	5	3	0	6	
38.01595	2.365000	25	1	1	12		59.80996	1.545000	5	3	0	12	
38.81732	2.318000	15	2	0	8								

01-074-8549

May 15, 2020 1:25 PM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Al2 (Al2.588 Si1.412) O9.706 **Empirical Formula:** Al4.588 O9.706 Si1.412
Weight %: Al38.84 O48.72 Si12.44 **Atomic %:** Al29.21 O61.80 Si8.99 **Compound Name:** Aluminum Silicon Oxide
Entry Date: 09/01/2008 **Modification Date:** 09/01/2016 **Modifications:** Quality

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 0.8 **I/Ic - CW ND:** 0.19

Crystal System: Orthorhombic **SPGR:** Pbam (55)
Author's Unit Cell [a: 7.5741(2) Å **b:** 7.6856(2) Å **c:** 2.88582(7) Å **Volume:** 167.99 Å³ **Z:** 1.00
MolVol: 167.99 **c/a:** 0.381 **a/b:** 0.985 **c/b:** 0.375 **] Calculated Density:** 3.151 g/cm³
Structural Density: 3.15 g/cm³ **SS/FOM:** F(30) = 999.9(0.0001, 32) **R-factor:** 0.069

Space Group: Pbam (55) **Molecular Weight:** 318.74 g/mol
Crystal Data [a: 7.574 Å **b:** 7.686 Å **c:** 2.886 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **XtlCell Vol:** 167.99 Å³
XtlCell Z: 1.00 **c/a:** 0.381 **a/b:** 0.985 **c/b:** 0.375 **]**
Reduced Cell [a: 2.886 Å **b:** 7.574 Å **c:** 7.686 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **RedCell Vol:** 167.99 Å³ **]**

AC Space Group: PBAM (55)
AC Unit Cell [a: 7.5741(2) Å **b:** 7.6856(2) Å **c:** 2.88582(7) Å **α:** 90° **β:** 90° **γ:** 90° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	3	-x,-y,z	5	x+1/2,-y+1/2,-z	7	-x+1/2,y+1/2,-z
2	-x,-y,-z	4	x,y,-z	6	-x+1/2,y+1/2,z	8	x+1/2,-y+1/2,z

ADP Type: B

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Biso	AET
Al	1	2a		0.0	0.0	0.0	1.0	1.91	
Al	2	4h		0.15103	0.3415	0.5	0.5	1.91	
Si	3	4h		0.15103	0.3415	0.5	0.353	1.91	
Al	4	4h		0.264	0.21	0.5	0.147	1.91	
O	5	4h		0.3501	0.4256	0.5	1.0	1.91	
O	6	4g		0.1289	0.2212	0.0	1.0	1.91	
O	7	2d		0.5	0.0	0.5	0.559	1.91	
O	8	4h		0.429	0.072	0.5	0.147	1.91	

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Inorganic **Pearson Symbol:** oP15.71 **Prototype Structure [Formula Order]:** Al4.8 Si1.2 O9
Prototype Structure [Alpha Order]: Al4.8 O9 Si1.2 **ANX:** A3X5

Cross-Ref PDF #'s: 04-014-4567 (Related Phase)

References:

Type	DOI	Reference
Primary Reference		Calculated from ICSD using POWD-12++.
Structure	10.1016/j.jeurceramsoc.2004.02.015	Tkalcec, E., Kurajica, S., Ivankovic, H. "Diphasic aluminosilicate gels with two stage mullitization in temperature range of 1200 - 1300 C". J. Eur. Ceram. Soc. 2005, 25, 613.

Database Comments: ANX: A3X5. Analysis: Al4.588 O9.706 Si1.412. Formula from original source: Al2 (Al2.588 Si1.412) O9.706. ICSD Collection Code: 152977. Calculated Pattern Original Remarks: Not leached sample heat treated at 1290 K. Wyckoff Sequence: h4 g d a(PBAM). Unit Cell Data Source: Powder Diffraction.

d-Spacings (183) - Al2 (Al2.588 Si1.412) O9.706 - 01-074-8549 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
16.41805	5.394690	507	1	1	0		42.82168	2.110050	16	3	2	0	
23.12627	3.842800	1	0	2	0		46.02002	1.970570	13	2	2	1	
23.47152	3.787050	5	2	0	0		47.26830	1.921400	11	0	4	0	
25.97878	**3.426960**	678	1	2	0		48.00742	1.893530	57	4	0	0	
26.21165	**3.397040**	1000	2	1	0		48.86150	1.862410	8	1	4	0	
30.96201	2.885820	188	0	0	1		49.00336	1.857350	3	1	3	1	
33.18587	2.697340	363	2	2	0		49.36404	1.844620	87	3	1	1	
35.24094	2.544610	489	1	1	1		49.53797	1.838550	18	4	1	0	
37.01220	2.426800	155	1	3	0		50.72636	1.798230	16	3	3	0	
37.46344	2.398600	13	3	1	0		53.42851	1.713480	56	2	4	0	
38.99968	2.307580	11	0	2	1		53.77344	1.703300	48	3	2	1	
39.21614	2.295340	210	2	0	1		53.93702	1.698520	97	4	2	0	
40.84645	**2.207410**	574	1	2	1		57.58300	1.599340	135	0	4	1	
42.57002	2.121940	194	2	3	0		58.22801	1.583150	47	4	0	1	

01-074-8549 May 15, 2020 1:25 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
58.97651	1.564830	11	1	4	1		104.29507	0.975540	1	6	5	0	
59.57208	1.550600	8	4	1	1		104.97366	0.971088	6	3	5	2	
60.62435	1.526180	347	3	3	1		105.53989	0.967430	4	5	3	2	
61.50557	1.506410	6	1	5	0		106.40487	0.961940	2	0	0	3	
62.43360	1.486230	5	5	1	0		107.04921	0.957926	4	0	6	2	
63.04214	1.473340	5	2	4	1		107.84367	0.953064	5	1	8	0	
63.50074	1.463800	73	4	2	1		108.33926	0.950079	16m	1	6	2	
64.53027	1.442910	155	0	0	2		108.33926	0.950079	16m	6	0	2	
65.47956	1.424270	32	2	5	0		108.85638	0.947003	11m	1	1	3	
66.26499	1.409280	51	5	2	0		108.85638	0.947003	11m	8	0	0	
67.09166	1.393910	8	1	1	2		109.55625	0.942902	8m	6	1	2	
69.51860	1.351060	14m	0	2	2		109.55625	0.942902	8m	7	4	0	
69.51860	1.351060	14m	3	4	1		110.11821	0.939660	3	8	1	0	
69.65958	1.348670	29m	2	0	2		111.41773	0.932333	4	2	0	3	
69.65958	1.348670	29m	4	4	0		111.62173	0.931204	3	2	8	0	
70.45259	1.335420	81	1	5	1		112.08210	0.928677	2	2	6	2	
70.79247	1.329840	38	1	2	2		112.54869	0.926145	10m	1	2	3	
70.90102	1.328070	51	2	1	2		112.54869	0.926145	10m	2	1	3	
71.32023	1.321290	19	5	1	1		112.91773	0.924163	7	6	5	1	
71.84463	1.312930	13	3	5	0		113.26600	0.922309	3	6	2	2	
72.41868	1.303930	10	5	3	0		113.77672	0.919619	4m	4	5	2	
73.93248	1.280930	10	0	6	0		113.77672	0.919619	4m	8	2	0	
74.18524	1.277190	98	2	5	1		114.11286	0.917867	1	5	4	2	
74.51792	1.272310	35	2	2	2		115.35499	0.911518	4	0	8	1	
74.92942	1.266340	89	5	2	1		116.45677	0.906048	1	2	2	3	
75.20725	1.262350	34m	1	6	0		116.67402	0.904987	1	1	8	1	
75.20725	1.262350	34m	6	0	0		117.24848	0.902209	1	4	7	1	
76.39454	1.245660	14	6	1	0		117.89834	0.899114	5m	6	6	0	
76.78838	1.240250	25	1	3	2		117.89834	0.899114	5m	8	0	1	
77.06920	1.236430	7	3	1	2		118.15807	0.897891	1	3	8	0	
78.16392	1.221830	13	4	4	1		118.64361	0.895626	1m	3	6	2	
78.81198	1.213400	4	2	6	0		118.64361	0.895626	1m	7	4	1	
79.92329	1.199300	5	6	2	0		119.10762	0.893487	3	8	1	1	
80.41670	1.193180	22m	2	3	2		119.25408	0.892817	2	3	1	3	
80.41670	1.193180	22m	4	5	0		119.69938	0.890795	2	6	3	2	
80.58922	1.191060	4	3	2	2		120.10116	0.888990	2	5	7	0	
80.81837	1.188260	16	5	3	1		120.30976	0.888060	1	8	3	0	
82.28292	1.170780	1	0	6	1		120.72848	0.886208	1	2	8	1	
83.52141	1.156540	3m	1	6	1		121.05278	0.884787	1	7	5	0	
83.52141	1.156540	3m	6	0	1		123.29496	0.875276	2	3	2	3	
83.76551	1.153790	1	0	4	2		125.10249	0.867996	2	1	7	2	
84.31464	1.147670	7	4	0	2		126.11032	0.864082	1	5	5	2	
84.80148	1.142320	1	3	6	0		127.14666	0.860163	8m	0	4	3	
84.95661	1.140630	1	1	4	2		127.14666	0.860163	8m	7	1	2	
85.46977	1.135090	3	4	1	2		127.83506	0.857618	3m	3	8	1	
85.72621	1.132350	4	6	3	0		127.83506	0.857618	3m	4	0	3	
86.38473	1.125400	4	3	3	2		128.07575	0.856739	3	4	8	0	
87.04534	1.118550	8	2	6	1		128.62053	0.854770	2m	1	4	3	
88.13913	1.107470	3	6	2	1		128.62053	0.854770	2m	4	6	2	
88.51865	1.103700	15	2	4	2		129.30731	0.852328	1	4	1	3	
88.60779	1.102820	14	4	5	1		129.57592	0.851385	2m	2	7	2	
88.92757	1.099680	27m	4	2	2		129.57592	0.851385	2m	6	4	2	
88.92757	1.099680	27m	5	4	1		130.09204	0.849592	7m	5	7	1	
90.29043	1.086590	2	1	7	0		130.09204	0.849592	7m	8	4	0	
91.11000	1.078940	1	5	5	0		130.49626	0.848205	22m	1	9	0	
91.92972	1.071450	2	7	1	0		130.49626	0.848205	22m	3	3	3	
92.97463	1.062130	2	3	6	1		131.17166	0.845921	12	7	5	1	
93.10668	1.060970	2	4	6	0		131.59880	0.844498	2	7	2	2	
93.84920	1.054520	2m	2	7	0		133.36136	0.838799	1	2	4	3	
93.84920	1.054520	2m	6	3	1		133.92770	0.837026	4m	4	2	3	
94.44885	1.049400	1	3	4	2		133.92770	0.837026	4m	9	1	0	
94.68799	1.047380	1	4	3	2		135.23506	0.833039	2	2	9	0	
95.39010	1.041520	3m	1	5	2		136.80845	0.828433	1	6	7	0	
95.39010	1.041520	3m	7	2	0		137.77626	0.825702	18	3	7	2	
96.15304	1.035270	2	5	1	2		139.10233	0.822084	2	9	2	0	
98.48615	1.016890	13	1	7	1		139.85011	0.820106	7	7	3	2	
98.91337	1.013640	12	2	5	2		141.98001	0.814713	4	8	4	1	
99.31585	1.010610	5	5	5	1		142.22632	0.814112	3m	1	9	1	
99.64024	1.008190	20	5	2	2		142.22632	0.814112	3m	3	4	3	
99.82101	1.006850	22	3	7	0		143.64383	0.810743	8	1	5	3	
100.14683	1.004450	8	7	1	1		144.12799	0.809627	1	5	6	2	
101.20935	0.996758	9	7	3	0		144.43209	0.808935	1	3	9	0	
101.34312	0.995804	13	4	6	1		144.77406	0.808165	1	6	5	2	
102.04045	0.990882	19m	2	7	1		145.04955	0.807551	2	5	1	3	
102.04045	0.990882	19m	6	4	1		145.70112	0.806121	3	8	5	0	
102.84812	0.985287	12	4	4	2		146.94020	0.803487	2	9	1	1	
103.67635	0.979665	2	7	2	1		148.48383	0.800360	4	2	9	1	
103.90788	0.978114	1	5	6	0								

01-078-4614

May 15, 2020 1:18 PM (Steve Simner)

Status Alternate **Quality Mark:** Star **Environment:** Ambient **Temp:** 293.0 K **Chemical Formula:** Ca (C O3)
Empirical Formula: C Ca O3 **Weight %:** C12.00 Ca40.04 O47.95 **Atomic %:** C20.00 Ca20.00 O60.00
Compound Name: Calcium Carbonate **Mineral Name:** Calcite, syn **Entry Date:** 09/01/2011

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 3.13 **I/Ic - CW ND:** 0.87

Crystal System: Rhombohedral **SPGR:** R-3c (167)
Author's Unit Cell [a: 4.99029(14) Å **c:** 17.0687(6) Å **Volume:** 368.11 Å³ **Z:** 6.00 **MolVol:** 61.35 **c/a:** 3.420 **] Calculated Density:** 2.709 g/cm³ **Structural Density:** 2.71 g/cm³ **SS/FOM:** F(30) = 999.9(0.0001, 30)
R-factor: 0.029

Space Group: R-3c (167) **Molecular Weight:** 100.09 g/mol
Crystal Data [a: 4.990 Å **b:** 4.990 Å **c:** 17.069 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **XtlCell Vol:** 368.11 Å³
XtlCell Z: 6.00 **c/a:** 3.421 **a/b:** 1.000 **c/b:** 3.421 **]**
Reduced Cell [a: 4.990 Å **b:** 4.990 Å **c:** 6.377 Å **α:** 66.97° **β:** 66.97° **γ:** 60.00° **RedCell Vol:** 122.70 Å³ **]**

Atomic parameters are cross-referenced from PDF entry 04-007-8659 **AC Space Group:** R-3cH (167)
AC Unit Cell [a: 4.989 Å **b:** 4.989 Å **c:** 17.062 Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	4	y,-x+y,-z	7	-y,-x,z+1/2	10	-x,-x+y,-z+1/2
2	-x,-y,-z	5	-x+y,-x,z	8	y,x,-z+1/2	11	-x+y,y,z+1/2
3	-y,x-y,z	6	x-y,x,-z	9	x,x-y,z+1/2	12	x-y,-y,-z+1/2

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	IDP	AET
Ca	1	6b	-3.	0.0	0.0	0.0	1.0		6-a
C	2	6a	32	0.0	0.0	0.25	1.0		3#b
O	3	18e	.2	0.2593	0.0	0.25	1.0		1#a

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Cement and Hydration Product, Ceramic (Bioceramic), Common Phase, Forensic, Inorganic, Mineral Related (Mineral, Synthetic), Pharmaceutical (Excipient), Superconducting Material

Mineral Classification: Calcite (supergroup), calcite (group) **Pearson Symbol:** hR10.00
Prototype Structure [Formula Order]: Ca C O3 **Prototype Structure [Alpha Order]:** C Ca O3 **ANX:** ABX3

Cross-Ref PDF #'s: 00-001-0837 (Deleted), 00-002-0623 (Deleted), 00-002-0629 (Deleted), 00-003-0569 (Deleted), 00-003-0596 (Deleted), 00-003-0670 (Deleted), 00-004-0636 (Deleted), 00-004-0637 (Deleted), 00-005-0586 (Alternate), 00-024-0027 (Deleted), 00-047-1743 (Primary), 00-066-0867 (Primary), 01-071-3699 (Primary), 01-072-1937 (Alternate), 01-072-4582 (Alternate), 01-075-6049 (Alternate), 01-078-3262 (Alternate), 01-078-4615 (Alternate), 01-080-2791 (Alternate), 01-080-2792 (Alternate), 01-080-2793 (Alternate), 01-080-2794 (Alternate), 01-080-2795 (Alternate), 01-080-2796 (Alternate), 01-080-2797 (Alternate), 01-080-2798 (Alternate), 01-080-2799 (Alternate), 01-080-2800 (Alternate), 01-080-2801 (Alternate), 01-080-2802 (Alternate), 01-080-2803 (Alternate), 01-080-2804 (Alternate), 01-080-2805 (Alternate), 01-080-2806 (Alternate), 01-080-2807 (Alternate), 01-080-2808 (Alternate), 01-080-2809 (Alternate), 01-080-2810 (Alternate), 01-080-2811 (Alternate), 01-080-9775 (Alternate), 01-080-9776 (Alternate), 01-083-0577 (Alternate), 01-083-0578 (Alternate), 01-083-3288 (Alternate), 01-083-3289 (Alternate), 01-083-4601 (Alternate), 01-083-4602 (Alternate), 01-083-4603 (Alternate), 01-083-4604 (Alternate), 01-083-4605 (Alternate), 01-083-4606 (Alternate), 01-083-4607 (Alternate), 01-083-4608 (Alternate), 01-083-4609 (Alternate), 01-083-4610 (Alternate), 01-083-4611 (Alternate), 01-083-4612 (Alternate), 01-083-4613 (Alternate), 01-083-4614 (Alternate), 01-083-4615 (Alternate), 01-083-4616 (Alternate), 01-083-4617 (Alternate), 01-083-4618 (Alternate), 01-083-4619 (Alternate), 01-083-4620 (Alternate), 01-083-4621 (Alternate), 01-083-4622 (Alternate), 01-083-4623 (Alternate), 01-083-4624 (Alternate), 01-083-4625 (Alternate), 01-083-4626 (Alternate), 01-083-4627 (Alternate), 01-083-4628 (Alternate), 01-083-4629 (Alternate), 01-085-0849 (Alternate), 01-086-2334 (Alternate), 01-086-2339 (Alternate), 01-086-2340 (Alternate), 01-086-2341 (Alternate), 01-086-2342 (Alternate), 01-086-2343 (Alternate), 04-001-7249 (Alternate), 04-002-9082 (Alternate), 04-006-6528 (Alternate), 04-007-0049 (Alternate), 04-007-2808 (Alternate), 04-007-4388 (Alternate), 04-007-8659 (Primary), 04-008-0198 (Alternate), 04-008-0212 (Alternate), 04-008-0213 (Alternate), 04-008-0788 (Alternate), 04-012-8072 (Alternate), 04-016-9713 (Alternate)

References:

Type	DOI	Reference
Primary Reference		Calculated from ICSD using POWD-12++.
Crystal Structure		Crystal Structure Source: LPF.
Structure	10.1154/1.3257906	Sitepu, H. "Texture and structural refinement using neutron diffraction data from molybdite (Mo O3) and calcite (Ca C O3) powders and a Ni-rich Ni50.7 Ti49.30 alloy". Powder Diffr. 2009, 24, 315.

01-078-4614 May 15, 2020 1:18 PM (Steve Simner)

Database Comments: ANX: ABX3. Analysis: C1 Ca1 O3. Formula from original source: Ca (C O3). ICSD Collection Code: 166364. Calculated Pattern Original Remarks: Refinement using the March-dollase model for preferred orientation. A refinement using the generalized-spherical-harmonic model is given in ICSD 166365. Sample Source or Locality: synthetic. Temperature of Data Collection: 293 K. Wyckoff Sequence: e b a (R3-CH). Unit Cell Data Source: Powder Diffraction.

d-Spacings (78) - Ca (C O3) - 01-078-4614 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
23.04868	3.855560	93	0	1	2		96.09884	1.035710	10m	1	1	15	
29.39051	**3.036450**	1000	1	0	4		97.64909	1.023360	2	2	1	13	
31.42014	2.844780	20	0	0	6		99.11078	1.012150	19	0	3	12	
35.96312	2.495150	141	1	1	0		102.19509	0.989802	3	3	2	1	
39.39967	**2.285070**	183	1	1	3		102.91247	0.984846	9	2	3	2	
43.14984	2.094760	147	2	0	2		103.48954	0.980923	2	1	3	10	
47.10240	1.927780	63	0	2	4		104.06823	0.977045	7	1	2	14	
47.48490	1.913140	181	0	1	8		105.80340	0.965745	7	3	2	4	
48.48909	**1.875840**	190	1	1	6		106.09567	0.963889	14	0	4	8	
56.55185	1.626030	32	2	1	1		107.26889	0.956572	3	0	2	16	
57.38722	1.604330	86	1	2	2		107.99875	0.952126	2	2	3	5	
58.05162	1.587540	9	1	0	10		108.59061	0.948579	1m	0	0	18	
60.65378	1.525510	48	2	1	4		108.59061	0.948579	1m	3	1	11	
60.97592	1.518220	21	2	0	8		109.52631	0.943076	14	4	1	0	
61.34894	1.509880	23	1	1	9		110.42307	0.937920	6	2	2	12	
63.03642	1.473460	19	1	2	5		111.77091	0.930382	1	1	4	3	
64.64782	1.440570	57	3	0	0		114.00088	0.918449	2	3	2	7	
65.57697	1.422390	29	0	0	12		115.07936	0.912910	1	4	0	10	
69.16563	1.357090	10	2	1	7		117.89474	0.899131	5	2	3	8	
70.21173	1.339410	17	0	2	10		118.74229	0.895169	4	1	4	6	
72.86825	1.296990	24	1	2	8		119.17401	0.893183	5	2	1	16	
73.64687	1.285190	5	3	0	6		120.68350	0.886406	5	1	1	18	
76.25657	1.247570	10	2	2	0		127.20514	0.859945	1	5	0	2	
77.12239	1.235710	16	1	1	12		127.91402	0.857329	5	3	2	10	
78.40928	1.218620	1	2	2	3		128.45313	0.855372	1	1	2	17	
80.21432	1.195680	1	1	3	1		128.63001	0.854736	2	3	1	14	
80.92361	1.186980	4	3	1	2		130.80966	0.847140	5	0	5	4	
81.49213	1.180130	19	2	1	10		131.61813	0.844434	1	1	4	9	
82.06043	1.173390	2	0	1	14		132.75546	0.840727	1	2	2	15	
83.74948	1.153970	35	1	3	4		133.85052	0.837266	4	0	1	20	
84.78225	1.142530	16	2	2	6		134.42830	0.835482	1	2	3	11	
85.85887	1.130940	1	3	1	5		135.68013	0.831715	3	3	3	0	
86.42203	1.125010	4	1	2	11		138.77348	0.822968	1	3	3	3	
91.46354	1.075690	1	1	3	7		141.54130	0.815795	1	2	4	1	
91.88329	1.071870	1	0	4	2		142.68132	0.813014	5	4	2	2	
93.00760	1.061840	6	2	0	14		144.57684	0.808608	1	0	4	14	
94.68799	1.047380	21	4	0	4		147.58162	0.802167	5	2	4	4	
94.97043	1.045010	23	3	1	8		148.10935	0.801103	4	5	0	8	
96.09884	1.035710	10m	1	0	16		149.55089	0.798297	5	3	3	6	

01-080-8935 Mav 15. 2020 1:31 PM (Steve Simner)

Status Alternate **Quality Mark:** Star **Environment:** Ambient **Temp:** 293.0 K **Phase:** β
Chemical Formula: Ca2 (Si O4) **Empirical Formula:** Ca2 O4 Si **Weight %:** Ca46.54 O37.15 Si16.31
Atomic %: Ca28.57 O57.14 Si14.29 **Compound Name:** Calcium Silicate **Mineral Name:** Larnite
Alternate Name: c2S, β-Ca2 (Si O4) **Entry Date:** 09/01/2013

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 0.75 **I/Ic - CW ND:** 0.15

Crystal System: Monoclinic **SPGR:** P21/n (14)
Author's Unit Cell [a: 5.5051(3) Å **b:** 6.7551(3) Å **c:** 9.3108(5) Å **β:** 94.513(4)° **Volume:** 345.17 Å³
Z: 4.00 **MolVol:** 86.29 **c/a:** 1.691 **a/b:** 0.815 **c/b:** 1.378 **]** **Calculated Density:** 3.314 g/cm³
Structural Density: 3.31 g/cm³ **SS/FOM:** F(30) = 491.8(0.0019, 32) **R-factor:** 0.053

Space Group: P21/n (14) **Molecular Weight:** 172.24 g/mol
Crystal Data [a: 9.311 Å **b:** 6.755 Å **c:** 5.505 Å **α:** 90.00° **β:** 94.51° **γ:** 90.00° **XtlCell Vol:** 345.17 Å³
XtlCell Z: 4.00 **c/a:** 0.591 **a/b:** 1.378 **c/b:** 0.815 **]**
Reduced Cell [a: 5.505 Å **b:** 6.755 Å **c:** 9.311 Å **α:** 90.00° **β:** 94.51° **γ:** 90.00° **RedCell Vol:** 345.17 Å³ **]**

Atomic parameters are cross-referenced from PDF entry 04-007-8540 **AC Space Group:** P121/n1 (14)
AC Unit Cell [a: 5.48(2) Å **b:** 6.76(2) Å **c:** 9.28(2) Å **α:** 90° **β:** 94.55° **γ:** 90° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	2	-x,-y,-z	3	x+1/2,-y+1/2,z+1/2	4	-x+1/2,y+1/2,-z+1/2

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	IDP	AET
Ca	1	4e	1	0.26	0.338	0.429	1.0		6-b
Ca	2	4e	1	0.284	-0.002	-0.299	1.0		8-b
Si	3	4e	1	0.26	-0.224	0.421	1.0		4-a
O	4	4e	1	0.32	0.002	0.434	1.0		1#a
O	5	4e	1	0.033	-0.253	0.308	1.0		1#a
O	6	4e	1	0.483	-0.36	0.366	1.0		1#a
O	7	4e	1	0.178	-0.325	-0.421	1.0		1#a

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Cement and Hydration Product, Ceramic, Common Phase, Forensic, Inorganic, Mineral Related (Mineral, Natural), Pharmaceutical (Excipient)

Mineral Classification: Olivine (group), related structures **Pearson Symbol:** mP28.00
Prototype Structure [Formula Order]: Ca2 Si O4 **ANX:** AB2X4

Cross-Ref PDF #'s: 00-009-0351 (Alternate), 00-029-0371 (Deleted), 00-033-0302 (Primary), 00-049-1673 (Primary), 01-077-0388 (Alternate), 01-083-0460 (Alternate), 01-083-0461 (Alternate), 01-083-0462 (Alternate), 01-083-0463 (Alternate), 01-083-0464 (Alternate), 01-083-0465 (Alternate), 01-084-7116 (Alternate), 04-007-2687 (Alternate), 04-007-8540 (Primary), 04-007-9746 (Alternate), 04-008-1786 (Alternate), 04-008-8073 (Alternate), 04-013-6288 (Alternate), 04-013-6289 (Alternate), 04-013-6290 (Alternate), 04-013-6291 (Alternate), 04-013-6292 (Alternate), 04-013-6293 (Alternate), 04-013-6294 (Alternate)

References:

Type	DOI	Reference
Primary Reference		Calculated from ICSD using POWD-12++.
Crystal Structure		Crystal Structure Source: LPF.
Structure		Yamnova, N.A., Zubkova, N.V., Eremin, N.N., Zadov, A.E., Gazeev, V.M. "Crystal structure of larnite - Ca2 Si O4 and specific features of polymorphic transitions in dicalcium orthosilicate". Kristallografiya 2011, 56, 235.
Structure		Yamnova, N.A., Zubkova, N.V., Eremin, N.N., Zadov, A.E., Gazeev, V.M. Crystallogr. Rep. 2011, 56, 210.

Database Comments: ANX: AB2X4. Analysis: Ca2 O4 Si1. Formula from original source: Ca2 (Si O4). ICSD Collection Code: 421708. Calculated Pattern Original Remarks: For description of atomic environment (AE) see isotypic compound in CCode 24640. Sample Source or Locality: near Gazeev, Lakargi Mountain, Kabardino-Balkaria, Russia. Temperature of Data Collection: 293 K. Wyckoff Sequence: e7 (P121/N1). Unit Cell Data Source: Single Crystal.

d-Spacings (198) - Ca2 (Si O4) - 01-080-8935 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
16.21495	5.461800	1	0	1	1		20.83721	4.259490	8	1	1	0	
18.10428	4.895850	36	-1	0	1		22.40884	3.964180	2	-1	1	1	
19.10763	4.640970	89	0	0	2		23.23421	3.825190	54	0	1	2	

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2θ (°)	d (Å)	I	h	k	l	*
23.48637	3.784690	45	1	1	1	
26.36562	3.377550	64	0	2	0	
27.52215	3.238190	99	-1	1	2	
28.09050	3.173950	61	0	2	1	
29.28843	3.046800	133	1	1	2	
31.06541	2.876450	263	1	2	0	
31.78491	2.812960	159	0	1	3	
32.04464	**2.790750**	1000	-1	0	3	
32.17013	**2.780150**	837	-1	2	1	
32.60544	**2.744020**	862	2	0	0	
32.76648	2.730900	324	0	2	2	
32.95073	2.716050	337	1	2	1	
34.34666	2.608790	629	1	0	3	
34.75177	2.579300	9	-1	1	3	
35.27444	2.542270	139	2	1	0	
35.90865	2.498810	9	-2	1	1	
36.01955	2.491370	5	-1	2	2	
36.68129	2.447930	146	-2	0	2	
36.90489	2.433610	73	1	1	3	
37.31741	2.407650	138	2	1	1	
37.42476	2.400990	173	1	2	2	
38.77418	2.320480	27	0	0	4	
39.10743	2.301470	31	-2	1	2	
39.46477	2.281450	289m	0	2	3	
39.46477	2.281450	289m	2	0	2	
41.09542	2.194610	198	0	1	4	
41.22067	2.188230	504	0	3	1	
41.70017	2.164170	177	2	1	2	
41.95995	2.151370	25	-1	2	3	
42.40656	2.129740	76	2	2	0	
42.95092	2.104000	20	-2	2	1	
43.20746	2.092100	82	-1	1	4	
43.40183	2.083180	48	1	3	0	
43.81186	2.064630	6	1	2	3	
44.17015	2.048710	163m	2	2	1	
44.17015	2.048710	163m	-1	3	1	
44.40359	2.038480	100	-2	1	3	
44.69531	2.025850	112	0	3	2	
44.83739	2.019760	141	1	3	1	
45.60895	1.987370	187	1	1	4	
45.73733	1.982090	292	-2	2	2	
47.49939	1.912590	83	0	2	4	
47.89934	1.897550	81	2	1	3	
48.03924	1.892350	144	2	2	2	
48.38478	1.879640	8	1	3	2	
49.35662	1.844880	54m	-1	2	4	
49.35662	1.844880	54m	-2	0	4	
50.05982	1.820600	35m	0	3	3	
50.05982	1.820600	35m	-3	0	1	
50.47512	1.806590	111	-2	2	3	
50.61064	1.802070	77	-1	0	5	
50.97564	1.790020	115	0	1	5	
51.57180	1.770720	6	1	2	4	
51.72798	1.765740	41	3	1	0	
51.93016	1.759340	10	-3	1	1	
52.15062	1.752420	5	-1	3	3	
52.51290	1.741180	14m	2	3	0	
52.51290	1.741180	14m	-1	1	5	
52.99304	1.726530	66	-2	3	1	
53.27851	1.717950	32	1	0	5	
53.67097	1.706310	150m	1	3	3	
53.67097	1.706310	150m	2	2	3	
54.03814	1.695580	54m	2	3	1	
54.03814	1.695580	54m	-3	1	2	
54.27391	1.688770	43	0	4	0	
55.23998	1.661500	18	0	4	1	
55.39452	1.657230	11m	2	1	4	
55.39452	1.657230	11m	-2	3	2	
56.32837	1.631950	181	-3	0	3	
56.52078	1.626850	93	0	2	5	
56.81622	1.619090	41	-2	2	4	
57.00869	1.614080	57m	0	3	4	
57.00869	1.614080	57m	1	4	0	
57.22235	1.608560	86m	3	1	2	
57.22235	1.608560	86m	3	2	0	
57.41147	1.603710	129m	2	3	2	
57.41147	1.603710	129m	-3	2	1	
57.69624	1.596470	23	-1	4	1	
57.95645	1.589920	33	-1	2	5	
58.07446	1.586970	71m	0	4	2	
58.07446	1.586970	71m	-3	1	3	
58.19216	1.584040	50	1	4	1	
58.61159	1.573700	139	-1	3	4	
58.89505	1.566800	15	3	2	1	
59.42190	1.554160	60m	-2	1	5	
59.42190	1.554160	60m	-3	2	2	
59.57674	1.550490	43	-2	3	3	
59.72520	1.546990	47	0	0	6	
60.21468	1.535580	7	-1	4	2	
60.56034	1.527640	69	1	3	4	
60.76164	1.523060	87m	2	2	4	
60.76164	1.523060	87m	3	0	3	
61.17838	1.513680	42	1	4	2	
61.43596	1.507950	10	0	1	6	
62.45837	1.485700	55m	2	3	3	
62.45837	1.485700	55m	3	1	3	
62.61593	1.482340	80m	0	4	3	
62.61593	1.482340	80m	-1	1	6	
63.22973	1.469420	1	-3	2	3	
63.70393	1.459620	10	-3	1	4	
64.18976	1.449740	39	2	1	5	
64.48818	1.443750	26m	-1	4	3	
64.48818	1.443750	26m	-2	2	5	
64.76633	1.438220	23	2	4	0	
65.06390	1.432360	7	0	3	5	
65.17330	1.430220	13	-2	4	1	
65.33557	1.427060	46m	1	1	6	
65.33557	1.427060	46m	-2	3	4	
65.71011	1.419830	17	3	3	0	
65.82343	1.417660	42m	1	4	3	
65.82343	1.417660	42m	-3	3	1	
66.09562	1.412480	42	2	4	1	
66.41395	1.406480	32m	0	2	6	
66.41395	1.406480	32m	-1	3	5	
67.01164	1.395380	53	-2	0	6	
67.30113	1.390080	35m	3	3	1	
67.30113	1.390080	35m	-2	4	2	
67.49765	1.386510	11	-1	2	6	
67.77442	1.381520	2	-3	3	2	
68.30873	1.372010	89	4	0	0	
68.62078	1.366530	35m	1	3	5	
68.62078	1.366530	35m	-2	1	6	
68.98278	1.360240	27m	2	2	5	
68.98278	1.360240	27m	2	3	4	
69.20524	1.356410	13m	3	1	4	
69.20524	1.356410	13m	-3	0	5	
69.90348	1.344560	20m	4	1	0	
69.90348	1.344560	20m	-4	0	2	
70.20031	1.339600	26m	1	2	6	
70.20031	1.339600	26m	-1	4	4	
70.36123	1.336930	10	0	5	1	
70.72824	1.330890	2	-3	1	5	
71.07411	1.325260	35	-2	4	3	
71.46804	1.318920	33	-4	1	2	
71.62971	1.316340	15	4	1	1	
71.85918	1.312700	10m	1	5	0	
71.85918	1.312700	10m	-1	0	7	
72.38910	1.304390	33	2	0	6	
72.50243	1.302630	36m	0	1	7	
72.50243	1.302630	36m	-2	3	5	
72.85651	1.297170	30	0	5	2	
72.96099	1.295570	44	1	5	1	
73.35044	1.289650	22m	-1	1	7	
73.35044	1.289650	22m	-2	2	6	
73.71838	1.284120	46	2	4	3	
73.91834	1.281140	4m	2	1	6	
73.91834	1.281140	4m	3	2	4	
74.33005	1.275060	4	0	3	6	
74.54534	1.271910	4m	4	2	0	
74.54534	1.271910	4m	-4	2	1	
74.71443	1.269450	2m	-1	5	2	
74.71443	1.269450	2m	-4	1	3	
74.92803	1.266360	1m	1	0	7	
74.92803	1.266360	1m	4	1	2	
75.26253	1.261560	4	3	3	3	
75.36282	1.260130	9m	-1	3	6	
75.36282	1.260130	9m	-3	2	5	
75.75487	1.254580	45	3	0	5	
76.13854	1.249210	47m	0	4	5	
76.13854	1.249210	47m	-4	2	2	
76.28181	1.247220	13	4	2	1	
76.41047	1.245440	26m	1	1	7	
76.41047	1.245440	26m	-3	3	4	
76.74302	1.240870	27	3	4	0	
76.90646	1.238640	20m	0	5	3	
76.90646	1.238640	20m	-3	4	1	
77.28689	1.233490	1m	0	2	7	
77.28689	1.233490	1m	3	1	5	
78.00205	1.223960	8m	-1	2	7	
78.00205	1.223960	8m	-4	0	4	
78.19745	1.221390	7	3	4	1	
78.54916	1.216800	37m	2	2	6	

01-080-8935 May 15, 2020 1:31 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
78.54916	1.216800	37m	-1	5	3		79.88408	1.199790	7m	4	1	3	
78.68419	1.215050	16	-3	4	2		80.14496	1.196540	25	2	5	1	
78.88343	1.212480	7m	2	5	0		81.02504	1.185750	8m	1	2	7	
78.88343	1.212480	7m	-2	1	7		81.02504	1.185750	8m	-2	3	6	
79.07443	1.210030	17	-3	1	6		81.26697	1.182830	14m	3	4	2	
79.30342	1.207110	9m	-2	5	1		81.26697	1.182830	14m	-2	5	2	
79.30342	1.207110	9m	-4	2	3		81.54823	1.179460	49	3	3	4	
79.52124	1.204350	7m	4	2	2		81.83339	1.176070	6	3	2	5	
79.52124	1.204350	7m	-4	1	4		82.04768	1.173540	26	-3	4	3	
79.88408	1.199790	7m	1	5	3		82.56017	1.167550	21	0	5	4	

01-089-0599

May 15, 2020 1:22 PM (Steve Simner)

Status Alternate **Quality Mark:** Star **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor) **Phase:** α
Chemical Formula: Fe2 O3 **Empirical Formula:** Fe2 O3 **Weight %:** Fe69.94 O30.06 **Atomic %:** Fe40.00 O60.00
Compound Name: Iron Oxide **Mineral Name:** Hematite, syn **Alternate Name:** iron(III) oxide
Entry Date: 09/01/2001 **Modification Date:** 09/01/2011 **Modifications:** Reflections

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 3.09 **I/Ic - CW ND:** 0.79

Crystal System: Rhombohedral **SPGR:** R-3c (167)
Author's Unit Cell [a: 5.032(1) Å **c:** 13.733(4) Å **Volume:** 301.15 Å³ **Z:** 6.00 **MolVol:** 50.19 **c/a:** 2.729 **]**
Calculated Density: 5.283 g/cm³ **Structural Density:** 5.28 g/cm³ **SS/FOM:** F(30) = 999.9(0.0002, 31)
R-factor: 0.045

Space Group: R-3c (167) **Molecular Weight:** 159.69 g/mol
Crystal Data [a: 5.032 Å **b:** 5.032 Å **c:** 13.733 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **XtlCell Vol:** 301.15 Å³
XtlCell Z: 6.00 **c/a:** 2.729 **a/b:** 1.000 **c/b:** 2.729 **]**
Reduced Cell [a: 5.032 Å **b:** 5.032 Å **c:** 5.422 Å **α:** 62.35° **β:** 62.35° **γ:** 60.00° **RedCell Vol:** 100.38 Å³ **]**

Atomic parameters are cross-referenced from PDF entry 04-003-2900 **AC Space Group:** R-3cH (167)
AC Unit Cell [a: 5.0342(3) Å **b:** 5.0342(3) Å **c:** 13.7483(4) Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	4	y,-x+y,-z	7	-y,-x,z+1/2	10	-x,-x+y,-z+1/2
2	-x,-y,-z	5	-x+y,-x,z	8	y,x,-z+1/2	11	-x+y,y,z+1/2
3	-y,x-y,z	6	x-y,x,-z	9	x,x-y,z+1/2	12	x-y,-y,-z+1/2

ADP Type: B

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Biso	AET
Fe	1	12c	3.	0.0	0.0	0.3553	1.0	0.61876	6-a
O	2	18e	.2	0.6942	0.0	0.25	1.0	0.68187	4-a

Anisotropic Displacement Parameters:

Atom	Num	Bani11	Bani22	Bani33	Bani12	Bani13	Bani23
Fe	1	0.629	0.629	0.599	0.315	0.0	0.0
O	2	0.693	0.681	0.667	0.34	0.044	0.088

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Common Phase, Forensic, Inorganic, Metal & Alloy, Mineral Related (Mineral, Synthetic), Pharmaceutical (Excipient), Pigment/Dye

Mineral Classification: Corundum (supergroup), corundum (group) **Pearson Symbol:** hR10.00
Prototype Structure [Alpha Order]: Al2 O3 **LPF Prototype Structure [Formula Order]:** Al2 O3,hR30,167
LPF Prototype Structure [Alpha Order]: Al2 O3,hR30,167 **ANX:** A2X3

Cross-Ref PDF #'s: 00-001-1053 (Alternate), 00-002-0915 (Deleted), 00-002-0919 (Deleted), 00-003-0800 (Deleted), 00-013-0534 (Deleted), 00-033-0664 (Primary), 01-071-5088 (Primary), 01-072-6225 (Alternate), 01-072-6226 (Alternate), 01-073-3825 (Alternate), 01-076-4579 (Alternate), 01-079-0007 (Alternate), 01-079-1741 (Alternate), 01-080-2377 (Alternate), 01-080-5405 (Alternate), 01-080-5406 (Alternate), 01-080-5407 (Alternate), 01-080-5408 (Alternate), 01-080-5409 (Alternate), 01-080-5410 (Alternate), 01-080-5411 (Alternate), 01-080-5413 (Alternate), 01-080-5414 (Alternate), 01-080-7077 (Alternate), 01-084-9870 (Alternate), 01-085-0599 (Alternate), 01-087-1164 (Alternate), 01-087-1166 (Alternate), 01-088-2359 (Primary), 01-089-0596 (Alternate), 01-089-0597 (Alternate), 01-089-0598 (Alternate), 01-089-8103 (Alternate), 01-089-8104 (Alternate), 04-002-2983 (Alternate), 04-002-4944 (Alternate), 04-002-5211 (Alternate), 04-002-7501 (Alternate), 04-003-1445 (Alternate), 04-003-2900 (Primary), 04-003-5818 (Alternate), 04-004-8410 (Alternate), 04-004-8979 (Alternate), 04-005-4425 (Alternate), 04-005-4630 (Alternate), 04-005-6837 (Alternate), 04-005-8668 (Alternate), 04-006-0285 (Alternate), 04-006-2616 (Alternate), 04-006-5321 (Alternate), 04-006-6607 (Alternate), 04-006-8177 (Alternate), 04-006-8208 (Alternate), 04-006-9058 (Alternate), 04-007-6009 (Alternate), 04-007-9266 (Alternate), 04-008-7622 (Alternate), 04-008-7623 (Alternate), 04-008-7624 (Alternate), 04-008-7625 (Alternate), 04-008-7626 (Alternate), 04-008-7627 (Alternate), 04-008-8479 (Alternate), 04-010-3230 (Alternate), 04-011-9585 (Alternate), 04-011-9586 (Alternate), 04-013-4794 (Alternate), 04-015-6943 (Alternate), 04-015-6944 (Alternate), 04-015-6945 (Alternate), 04-015-6947 (Alternate), 04-015-7029 (Alternate), 04-015-9569 (Alternate), 04-015-9570 (Alternate), 04-015-9571 (Alternate), 04-015-9572 (Alternate), 04-015-9573 (Alternate), 04-015-9574 (Alternate), 04-015-9575 (Alternate), 04-015-9576 (Alternate), 04-015-9577 (Alternate), 04-015-9578 (Alternate), 04-015-9579 (Alternate), 04-015-9580 (Alternate), 04-015-9685 (Alternate), 04-015-9752 (Alternate), 04-015-9903 (Alternate), 04-015-9904 (Alternate), 04-015-9905 (Alternate), 04-015-9906 (Alternate), 04-015-9907 (Alternate), 04-015-9908 (Alternate), 04-018-0098 (Alternate)

01-089-0599 Mav 15. 2020 1:22 PM (Steve Simner)

References:

Type	DOI	Reference
Primary Reference		Calculated from ICSD using POWD-12++.
Additional Reference		Sadykov, V.A., Isupova, L.A., Tsybulya, S.V., Cherepanova, S.V., Litvak, G.S., Burgina, E.B., Kustova, G.N., Kolomiichuk, V.N., Ivanov, V.P., Paukshtis, E.A., Golovin, A.V., Avvakumov, E.G. Golden Book of Phase Transitions, Wroclaw 2002, 1, 1.
Crystal Structure		Crystal Structure Source: LPF.
Structure	10.1006/jssc.1996.0168	Sadykov, V.A., Isupova, L.A., Tsybulya, S.V., Cherepanova, S.V., Litvak, G.S., Burgina, E.B., Kustova, G.N., Kolomiichuk, V.N., Ivanov, V.P., Paukshtis, E.A., Golovin, A.V., Avvakumov, E.G. "Effect of mechanical activation on the real structure and reactivity of iron(III) oxide with corundum-type structure". J. Solid State Chem. 1996, 123, 191.

Database Comments: ANX: A2X3. Analysis: Fe2 O3. Formula from original source: Fe2 O3. ICSD Collection Code: 82137. Calculated Pattern Original Remarks: Sample calcinated at 1073 K. Stable above 773 K (2nd ref., Tomaszewski), below Ia3-, m.p. 1523 K. Wyckoff Sequence: e c(R3-CH). Unit Cell Data Source: Powder Diffraction.

d-Spacings (64) - Fe2 O3 - 01-089-0599 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
24.16852	3.679390	354	0	1	2		96.92853	1.029040	1	1	3	7	
33.19208	**2.696850**	1000	1	0	4		101.15899	0.997118	1	3	2	1	
35.65504	**2.516000**	749	1	1	0		101.46381	0.994946	1	1	2	11	
39.33227	2.288830	25	0	0	6		102.41649	0.988263	20	3	1	8	
40.89483	2.204910	206	1	1	3		105.04012	0.970656	4	2	2	9	
43.54064	2.076860	22	2	0	2		106.73314	0.959887	51	3	2	4	
49.50492	1.839700	357	0	2	4		107.20192	0.956984	24	0	1	14	
54.12444	**1.693080**	475	1	1	6		108.19252	0.950959	41	4	1	0	
56.19935	1.635390	6	2	1	1		110.15384	0.939456	1	2	3	5	
57.49141	1.601670	27	1	2	2		111.64420	0.931080	6	1	4	3	
57.66858	1.597170	67	0	1	8		113.73299	0.919848	10	0	4	8	
62.48879	1.485050	274	2	1	4		116.20171	0.907301	52	1	3	10	
64.04782	1.452610	276	3	0	0		117.93307	0.898950	5	3	0	12	
66.09035	1.412580	3	1	2	5		118.89457	0.894466	24	2	0	14	
69.67437	1.348420	20	2	0	8		119.70625	0.890764	1	3	2	7	
72.04314	1.309800	98	1	0	10		120.05107	0.889214	1	2	1	13	
72.36918	1.304700	19	1	1	9		122.59716	0.878178	63	1	4	6	
75.26883	1.261470	3	2	1	7		125.00628	0.868375	1	3	1	11	
75.51275	1.258000	57	2	2	0		126.15235	0.863921	7	2	3	8	
77.81302	1.226460	24	3	0	6		127.09817	0.860344	4	1	1	15	
78.84070	1.213030	10	2	2	3		128.97585	0.853501	27	4	0	10	
79.54975	1.203990	1	1	3	1		130.98799	0.846538	1	2	2	12	
80.64719	1.190350	17	3	1	2		131.51617	0.844772	21	0	5	4	
80.79867	1.188500	28	1	2	8		132.11784	0.842791	42	1	2	14	
83.05902	1.161800	47	0	2	10		133.40322	0.838667	32	3	3	0	
84.60961	1.144420	3	0	0	12		138.05271	0.824936	1	3	3	3	
85.00908	1.140060	65	1	3	4		139.10494	0.822077	1	2	4	1	
88.64533	1.102450	68	2	2	6		140.78549	0.817694	3	4	2	2	
91.42968	1.076000	7	0	4	2		144.73121	0.808261	31	3	2	10	
93.82015	1.054770	67	2	1	10		145.27258	0.807058	6	4	1	9	
95.36593	1.041720	2	1	1	12		148.24335	0.800836	31	2	4	4	
95.76544	1.038430	18	4	0	4		149.39836	0.798587	7	0	2	16	

04-007-2718 Mav 15. 2020 1:22 PM (Steve Simner)

Status Alternate **Quality Mark:** Star **Environment:** Ambient **Temp:** 296.0 K **Phase:** Room temperature phase.
Chemical Formula: Fe3 O4 **Empirical Formula:** Fe3 O4 **Weight %:** Fe72.36 O27.64 **Atomic %:** Fe42.86 O57.14
Compound Name: Iron Oxide **Mineral Name:** Magnetite, syn **Alternate Name:** iron diiron(III) oxide
Entry Date: 09/01/2005 **Modification Date:** 09/01/2011 **Modifications:** Reflections

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 4.91 **I/Ic - CW ND:** 1.4

Crystal System: Cubic **SPGR:** Fd-3m (227)
Author's Unit Cell [a: 8.375(2) Å **Volume:** 587.43 Å³ **Z:** 8.00 **MolVol:** 73.43 **] Calculated Density:** 5.236 g/cm³
Structural Density: 5.24 g/cm³ **SS/FOM:** F(30) = 999.9(0.0001, 30) **R-factor:** 0.016

Space Group: Fd-3m (227) **Molecular Weight:** 231.53 g/mol
Crystal Data [a: 8.375 Å **b:** 8.375 Å **c:** 8.375 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **XtlCell Vol:** 587.43 Å³
XtlCell Z: 8.00 **a/b:** 1.000 **c/b:** 1.000 **]**
Reduced Cell [a: 5.922 Å **b:** 5.922 Å **c:** 5.922 Å **α:** 60.00° **β:** 60.00° **γ:** 60.00° **RedCell Vol:** 146.86 Å³ **]**

AC Space Group: Fd-3m (227)
AC Unit Cell [a: 8.375(2) Å **b:** 8.375(2) Å **c:** 8.375(2) Å **α:** 90° **β:** 90° **γ:** 90° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	11	z,-x+1/4,-y+1/4	21	-y+1/4,z,-x+1/4	31	-x+1/4,-z+1/4,y	41	z,y,x
2	-x,-y,-z	12	-z,x+3/4,y+3/4	22	y+3/4,-z,x+3/4	32	x+3/4,z+3/4,-y	42	-z,-y,-x
3	x,-y+1/4,-z+1/4	13	-z+1/4,x,-y+1/4	23	-y+1/4,-z+1/4,x	33	y,x,z	43	z,-y+1/4,-x+1/4
4	-x,y+3/4,z+3/4	14	z+3/4,-x,y+3/4	24	y+3/4,z+3/4,-x	34	-y,-x,-z	44	-z,y+3/4,x+3/4
5	-x+1/4,y,-z+1/4	15	-z+1/4,-x+1/4,y	25	x,z,y	35	y,-x+1/4,-z+1/4	45	-z+1/4,y,-x+1/4
6	x+3/4,-y,z+3/4	16	z+3/4,x+3/4,-y	26	-x,-z,-y	36	-y,x+3/4,z+3/4	46	z+3/4,-y,x+3/4
7	-x+1/4,-y+1/4,z	17	y,z,x	27	x,-z+1/4,-y+1/4	37	-y+1/4,x,-z+1/4	47	-z+1/4,-y+1/4,x
8	x+3/4,y+3/4,-z	18	-y,-z,-x	28	-x,z+3/4,y+3/4	38	y+3/4,-x,z+3/4	48	z+3/4,y+3/4,-x
9	z,x,y	19	y,-z+1/4,-x+1/4	29	-x+1/4,z,-y+1/4	39	-y+1/4,-x+1/4,z		
10	-z,-x,-y	20	-y,z+3/4,x+3/4	30	x+3/4,-z,y+3/4	40	y+3/4,x+3/4,-z		

Origin: O2

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	IDP	AET
Fe	1	8a	-43m	0.125	0.125	0.125	1.0		4-a
Fe	2	16d	.-3m	0.5	0.5	0.5	1.0		6-a
O	3	32e	.3m	0.2555	0.2555	0.2555	1.0		4-a

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Battery Material, Common Phase, Forensic, Inorganic, Metal & Alloy, Micro & Mesoporous, Mineral Related (Mineral, Synthetic), Pharmaceutical (Excipient), Pigment/Dye

Mineral Classification: Spinel (supergroup), 1C-oxide (group) **Pearson Symbol:** cF56.00
Prototype Structure [Alpha Order]: Fe3 O4 **LPF Prototype Structure [Formula Order]:** Fe3 O4,cF56,227
LPF Prototype Structure [Alpha Order]: Fe3 O4,cF56,227 **ANX:** AB2X4

04-007-2718 Mav 15. 2020 1:22 PM (Steve Simner)

Cross-Ref PDF #'s: 00-001-1111 (Deleted), 00-002-1035 (Deleted), 00-003-0862 (Deleted), 00-007-0322 (Deleted), 00-011-0614 (Deleted), 00-019-0629 (Primary), 00-026-1136 (Primary), 00-065-0731 (Primary), 01-071-4918 (Alternate), 01-072-2303 (Alternate), 01-074-1909 (Alternate), 01-074-1910 (Alternate), 01-075-0449 (Alternate), 01-075-1610 (Alternate), 01-075-9710 (Alternate), 01-076-1849 (Alternate), 01-076-5948 (Alternate), 01-078-6086 (Primary), 01-080-6402 (Alternate), 01-080-6403 (Alternate), 01-080-6404 (Alternate), 01-080-6405 (Alternate), 01-080-6406 (Alternate), 01-080-6407 (Alternate), 01-080-6408 (Alternate), 01-080-6409 (Alternate), 01-080-6410 (Alternate), 01-080-7683 (Alternate), 01-082-3507 (Alternate), 01-084-2782 (Alternate), 01-084-6015 (Alternate), 01-084-6684 (Alternate), 01-084-6685 (Alternate), 01-084-6686 (Alternate), 01-084-6687 (Alternate), 01-084-6688 (Alternate), 01-084-6689 (Alternate), 01-084-6690 (Alternate), 01-084-6691 (Alternate), 01-084-6692 (Alternate), 01-084-6693 (Alternate), 01-084-6694 (Alternate), 01-084-6695 (Alternate), 01-084-6696 (Alternate), 01-084-6697 (Primary), 01-084-6698 (Alternate), 01-084-6699 (Alternate), 01-084-6700 (Alternate), 01-087-2334 (Alternate), 01-088-0866 (Alternate), 01-089-3854 (Alternate), 01-089-4319 (Alternate), 03-065-3107 (Alternate), 04-001-7822 (Alternate), 04-001-7909 (Alternate), 04-001-9000 (Alternate), 04-001-9326 (Alternate), 04-002-0264 (Alternate), 04-002-0618 (Alternate), 04-002-0643 (Alternate), 04-002-1855 (Alternate), 04-002-2487 (Alternate), 04-002-2707 (Alternate), 04-002-2709 (Alternate), 04-002-2981 (Alternate), 04-002-3194 (Alternate), 04-002-3668 (Alternate), 04-002-5310 (Alternate), 04-002-5448 (Alternate), 04-002-5632 (Alternate), 04-002-5683 (Alternate), 04-002-5903 (Alternate), 04-002-6866 (Alternate), 04-002-6955 (Alternate), 04-002-8141 (Alternate), 04-002-8629 (Alternate), 04-002-9019 (Alternate), 04-002-9635 (Alternate), 04-003-1446 (Alternate), 04-004-2838 (Alternate), 04-005-4307 (Alternate), 04-005-4319 (Primary), 04-005-4404 (Alternate), 04-005-4551 (Alternate), 04-005-5733 (Alternate), 04-005-6268 (Alternate), 04-005-9786 (Alternate), 04-005-9788 (Alternate), 04-005-9815 (Alternate), 04-006-0225 (Alternate), 04-006-0424 (Alternate), 04-006-0425 (Alternate), 04-006-1668 (Alternate), 04-006-2406 (Alternate), 04-006-2467 (Alternate), 04-006-2752 (Alternate), 04-006-4615 (Alternate), 04-006-6497 (Alternate), 04-006-6550 (Alternate), 04-006-6692 (Alternate), 04-006-8076 (Alternate), 04-007-1427 (Alternate), 04-007-6010 (Alternate), 04-007-8567 (Alternate), 04-007-8976 (Alternate), 04-007-9093 (Alternate), 04-008-0315 (Alternate), 04-008-0777 (Alternate), 04-008-4423 (Alternate), 04-008-4511 (Alternate), 04-008-4512 (Alternate), 04-008-8145 (Alternate), 04-008-8146 (Alternate), 04-008-8147 (Alternate), 04-008-8148 (Alternate), 04-009-4225 (Alternate), 04-009-8417 (Alternate), 04-009-8418 (Alternate), 04-009-8419 (Alternate), 04-009-8420 (Alternate), 04-009-8421 (Alternate), 04-009-8422 (Alternate), 04-009-8423 (Alternate), 04-009-8424 (Alternate), 04-009-8425 (Alternate), 04-009-8426 (Alternate), 04-009-8427 (Alternate), 04-009-8428 (Alternate), 04-009-8429 (Alternate), 04-009-8430 (Alternate), 04-009-8431 (Alternate), 04-009-8432 (Alternate), 04-009-8433 (Alternate), 04-009-8434 (Alternate), 04-009-8435 (Alternate), 04-009-8436 (Alternate), 04-009-8437 (Alternate), 04-009-8438 (Alternate), 04-009-8439 (Alternate), 04-009-8440 (Alternate), 04-009-8441 (Alternate), 04-009-8442 (Alternate), 04-009-8443 (Alternate), 04-011-5952 (Alternate), 04-013-7099 (Alternate), 04-013-7100 (Alternate), 04-013-9806 (Alternate), 04-013-9807 (Alternate), 04-013-9808 (Alternate), 04-013-9809 (Alternate), 04-013-9810 (Alternate), 04-013-9811 (Alternate), 04-014-1396 (Alternate), 04-014-9664 (Alternate), 04-015-3100 (Alternate), 04-015-3101 (Alternate), 04-015-3102 (Alternate), 04-015-8200 (Alternate), 04-015-8203 (Alternate), 04-015-8204 (Alternate), 04-015-8207 (Alternate), 04-015-8209 (Alternate), 04-015-8211 (Alternate), 04-015-8213 (Alternate), 04-015-8214 (Alternate), 04-017-1024 (Alternate), 04-020-0779 (Alternate), 04-021-0451 (Alternate), 04-022-0447 (Alternate)

Former PDF Numbers: 01-088-0315

References:

Type	DOI	Reference
Primary Reference		Calculated from LPF using POWD-12++.
Structure	10.1107/S0108768197007842	Sasaki, S. "Radial Distribution of Electron Density in Magnetite, \Fe3 O4\." Acta Crystallogr., Sect. B: Struct. Sci. 1997, 53, 762.

Database Comments: ANX: AB2X4. LPF Collection Code: 1252695. Polymorphism: Room temperature phase. Sample Preparation: STARTING MATERIALS:Fe3O4. Compound Preparation: crystals grown at 1300 K. CRUCIBLE: sealed evacuated silica tube. Temperature of Data Collection: 296 K. Unit Cell Data Source: Single Crystal.

d-Spacings (34) - Fe3 O4 - 04-007-2718 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
18.33290	4.835310	92	1	1	1		89.89604	1.090330	86	7	3	1	
30.15685	**2.961010**	290	2	2	0		94.74740	1.046880	32	8	0	0	
35.52139	**2.525160**	1000	3	1	1		97.67342	1.023170	1	7	3	3	
37.15740	2.417650	78	2	2	2		98.65255	1.015620	1	6	4	4	
43.17170	2.093750	205	4	0	0		102.59867	0.987003	11	8	2	2	
47.26935	1.921360	5	3	3	1		105.59729	0.967062	44	7	5	1	
53.56146	1.709540	83	4	2	2		106.60634	0.960678	9	6	6	2	
57.09790	1.611770	271	5	1	1		110.69959	0.936353	19	8	4	0	
62.70257	**1.480500**	348	4	4	0		113.84231	0.919276	2	9	1	1	
65.92981	1.415630	8	5	3	1		114.90667	0.913787	1	8	4	2	
66.98718	1.395830	1	4	4	2		119.26262	0.892778	5	6	6	4	
71.13964	1.324200	26	6	2	0		122.65416	0.877939	32	9	3	1	
74.18592	1.277180	63	5	3	3		128.62053	0.854770	65	8	4	4	
75.19117	1.262580	27	6	2	2		132.44761	0.841719	1	9	3	3	
79.16836	1.208830	20	4	4	4		139.42086	0.821236	13	10	2	0	
82.11656	1.172730	4	7	1	1		144.12143	0.809642	39	9	5	1	
86.98604	1.119160	24	6	4	2		145.81003	0.805885	9	10	2	2	

04-008-6822

Jun 1, 2020 6:53 PM (Steve Simner)

Status Primary **Quality Mark:** Indexed **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca2 Fe Al O5 **Empirical Formula:** Al Ca2 Fe O5 **Weight %:** Al11.10 Ca32.99 Fe22.98 O32.92
Atomic %: Al11.11 Ca22.22 Fe11.11 O55.56 **Compound Name:** Calcium Iron Aluminum Oxide
Mineral Name: Brownmillerite, ferrian, syn **Alternate Name:** iron(III) aluminium oxide bis(calcium oxide)
Entry Date: 09/01/2005 **Modification Date:** 09/01/2011 **Modifications:** Reflections

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 1.87 **I/Ic - CW ND:** 0.3

Crystal System: Orthorhombic **SPGR:** Pcmn (62)
Author's Unit Cell [a: 5.58 Å **b:** 14.5 Å **c:** 5.34 Å **Volume:** 432.06 Å³ **Z:** 4.00 **MolVol:** 108.02
c/a: 0.957 **a/b:** 0.385 **c/b:** 0.368 **] Calculated Density:** 3.735 g/cm³ **Structural Density:** 3.73 g/cm³
SS/FOM: F(30) = 457.2(0.0019, 35) **R-factor:** 0.147

Space Group: Pcmn (62) **Molecular Weight:** 242.98 g/mol
Crystal Data [a: 5.580 Å **b:** 14.500 Å **c:** 5.340 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **XtlCell Vol:** 432.06 Å³
XtlCell Z: 4.00 **c/a:** 0.957 **a/b:** 0.385 **c/b:** 0.368 **]**
Reduced Cell [a: 5.340 Å **b:** 5.580 Å **c:** 14.500 Å **α:** 90.00° **β:** 90.00° **γ:** 90.00° **RedCell Vol:** 432.06 Å³ **]**

AC Space Group: Pcmn (62)
AC Unit Cell [a: 5.58 Å **b:** 14.5 Å **c:** 5.34 Å **α:** 90° **β:** 90° **γ:** 90° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	3	-x+1/2,-y+1/2,z+1/2	5	-x,y+1/2,-z	7	x+1/2,-y,-z+1/2
2	-x,-y,-z	4	x+1/2,y+1/2,-z+1/2	6	x,-y+1/2,z	8	-x+1/2,y,z+1/2

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	IDP	AET
Fe	1	4a	-1	0.0	0.0	0.0	0.5		6-a
Fe	2	4c	.m.	-0.072	0.25	-0.055	0.5		4-a
Al	3	4a	-1	0.0	0.0	0.0	0.5		6-a
Al	4	4c	.m.	-0.072	0.25	-0.055	0.5		4-a
Ca	5	8d	1	0.028	0.112	0.48	1.0		7-g
O	6	8d	1	0.25	-0.015	0.25	1.0		2#a
O	7	8d	1	0.055	0.133	0.0	1.0		1#a
O	8	4c	.m.	-0.137	0.25	0.607	1.0		4-a

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Forensic, Inorganic, Mineral Related (Mineral, Synthetic) **Pearson Symbol:** oP36.00
Prototype Structure [Formula Order]: Ca2 Fe2 O5 **Prototype Structure [Alpha Order]:** Ca2 Fe2 O5
LPF Prototype Structure [Formula Order]: Ca2 Fe2 O5,oP36,62
LPF Prototype Structure [Alpha Order]: Ca2 Fe2 O5,oP36,62 **ANX:** A2B2X5

Cross-Ref PDF #'s: 00-011-0124 (Deleted), 04-002-2560 (Alternate) **Former PDF Numbers:** 01-074-0803, 01-074-1346

References:

Type	DOI	Reference
Primary Reference		Calculated from LPF using POWD-12++.
Structure	10.1107/S0365110X59000433	Bertaut E.F., Blum P., Sagnieres A. "Structure du Ferrite Bicalcique et de la Brownmillerite". Acta Crystallogr. 1959, 12, 149.

Database Comments: ANX: A2B2X5. LPF Collection Code: 1900467. Minor Warning: No e.s.d reported/abstracted on the cell dimension. 10%<R factor<15% (for powder). LPF Editor Comment: unit for interplanar spacing d omitted, assumed to be Å. Unit Cell Data Source: Powder Diffraction.

d-Spacings (198) - Ca2 Fe Al O5 - 04-008-6822 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
12.19786	7.250000	201	0	2	0		34.83637	2.573230	128	1	5	0	
17.01186	5.207700	63	1	1	0		35.80966	2.505490	9	0	2	2	
23.03384	3.858010	30	1	0	1		36.29898	2.472830	19	2	0	1	
23.84684	3.728290	34	1	1	1		36.84184	2.437630	27	2	1	1	
24.34334	3.653360	164	1	3	0		37.17302	2.416670	6	0	6	0	
24.53675	3.625000	18	0	4	0		37.30408	2.408480	1	1	0	2	
26.14296	3.405810	1	1	2	1		37.83441	2.375930	1	1	1	2	
29.60206	3.015230	90	1	3	1		38.43045	2.340440	21m	0	3	2	
32.05349	2.790000	409	2	0	0		38.43045	2.340440	21m	2	2	1	
33.53568	**2.670000**	496	0	0	2		38.81506	2.318130	26	1	5	1	
33.90442	**2.641800**	1000	1	4	1		39.38908	2.285660	1	1	2	2	
34.11665	2.625850	48	0	1	2		40.77775	2.210970	88	2	4	0	

04-008-6822 Jun 1, 2020 6:53 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
41.87231	2.155670	78	1	3	2		76.60510	1.242760	2m	1	10	2	
41.99204	2.149800	77	0	4	2		76.60510	1.242760	2m	3	3	3	
44.18537	2.048040	121	1	6	1		76.92998	1.238320	3	1	8	3	
45.16044	2.006060	1	1	4	2		77.07068	1.236410	11m	2	9	2	
46.17640	1.964260	1	0	5	2		77.07068	1.236410	11m	4	0	2	
47.07081	**1.929000**	459	2	0	2		77.40221	1.231940	1	4	1	2	
47.51073	1.912160	27	2	1	2		77.64451	1.228700	4	3	7	2	
48.33007	1.881640	15	2	5	1		78.02404	1.223670	7	4	5	1	
48.81292	1.864150	84	2	2	2		78.39394	1.218820	5m	3	9	0	
49.13108	1.852820	8	1	5	2		78.39394	1.218820	5m	4	2	2	
49.88178	1.826680	17m	1	7	1		78.66950	1.215240	7	2	7	3	
49.88178	1.826680	17m	2	6	0		78.92155	1.211990	29m	0	5	4	
50.29906	1.812500	225	0	8	0		78.92155	1.211990	29m	3	4	3	
50.92351	1.791730	23m	0	6	2		79.22091	1.208160	7m	0	12	0	
50.92351	1.791730	23m	2	3	2		79.22091	1.208160	7m	4	6	0	
52.42960	1.743750	4	3	1	1		79.52995	1.204240	20	2	0	4	
52.68490	1.735900	95	3	3	0		79.85850	1.200110	9	2	1	4	
52.93256	1.728360	10	2	6	1		80.84301	1.187960	1m	2	2	4	
53.64381	1.707110	2m	1	6	2		80.84301	1.187960	1m	3	9	1	
53.64381	1.707110	2m	3	2	1		81.08619	1.185010	1	1	5	4	
53.78675	1.702910	1	2	4	2		81.33770	1.181980	1	0	11	2	
54.03021	1.695810	2	1	0	3		81.63883	1.178380	1	4	6	1	
54.42878	1.684330	6	1	1	3		81.87310	1.175600	1	3	5	3	
55.61284	1.651240	1m	1	2	3		82.33084	1.170220	9	4	4	2	
55.61284	1.651240	1m	3	3	1		82.51969	1.168020	6m	1	9	3	
56.00960	1.640480	11	1	8	1		82.51969	1.168020	6m	3	8	2	
56.15263	1.636640	8	0	7	2		83.29915	1.159060	19	2	10	2	
57.31694	1.606130	3	2	5	2		83.54088	1.156320	13m	1	11	2	
57.55034	1.600170	53	1	3	3		83.54088	1.156320	13m	2	8	3	
58.03640	1.587920	21	2	7	1		83.82701	1.153100	25	1	12	1	
58.32655	1.580710	144	3	4	1		84.75479	1.142830	9m	1	6	4	
58.74351	1.570480	5	1	7	2		84.75479	1.142830	9m	2	4	4	
58.94299	1.565640	28	3	5	0		85.45298	1.135270	15	3	6	3	
59.68737	1.547880	9	1	9	0		85.86924	1.130830	17	4	7	1	
60.19478	1.536040	123	1	4	3		86.69767	1.122140	7	0	7	4	
60.90003	1.519930	77	2	8	0		87.07844	1.118210	3	3	10	1	
61.45086	1.507620	4	2	6	2		87.61789	1.112710	2m	2	5	4	
61.81500	1.499610	100m	0	8	2		87.61789	1.112710	2m	5	1	0	
61.81500	1.499610	100m	2	0	3		88.08808	1.107980	2m	2	12	0	
62.13561	1.492640	10m	2	1	3		88.08808	1.107980	2m	3	9	2	
62.13561	1.492640	10m	3	2	2		88.33899	1.105480	10	4	8	0	
62.41211	1.486690	3	1	9	1		88.68494	1.102060	6	1	10	3	
63.22781	1.469460	13	2	2	3		88.88260	1.100120	3m	1	7	4	
63.49638	1.463890	22	1	5	3		88.88260	1.100120	3m	4	6	2	
63.91293	1.455350	43	3	3	2		89.10183	1.097980	3m	2	9	3	
64.17687	1.450000	6m	0	10	0		89.10183	1.097980	3m	4	0	3	
64.17687	1.450000	6m	1	8	2		89.42479	1.094850	3	4	1	3	
65.02463	1.433130	1	2	3	3		89.53894	1.093750	4	1	13	0	
65.65751	1.420840	27	3	6	1		89.66177	1.092570	4m	3	7	3	
66.13788	1.411680	10	2	7	2		89.66177	1.092570	4m	5	0	1	
66.40755	1.406600	6	3	4	2		90.00320	1.089310	1	5	1	1	
67.03230	1.395000	21	4	0	0		90.20572	1.087390	7m	2	11	2	
67.40777	1.388140	25m	1	6	3		90.20572	1.087390	7m	5	3	0	
67.40777	1.388140	25m	2	4	3		90.39551	1.085600	3m	2	12	1	
67.89051	1.379440	1	0	9	2		90.39551	1.085600	3m	4	2	3	
68.43023	1.369870	29	4	2	0		90.82948	1.081540	4	3	1	4	
69.15341	1.357300	20	1	10	1		90.97272	1.080210	6m	1	12	2	
69.54745	1.350570	35m	3	5	2		90.97272	1.080210	6m	5	2	1	
69.54745	1.350570	35m	4	0	1		91.54998	1.074900	24m	0	8	4	
69.94282	1.343900	18	4	1	1		91.54998	1.074900	24m	3	11	0	
70.22918	1.339120	3m	1	9	2		91.80052	1.072620	4	3	2	4	
70.22918	1.339120	3m	3	7	1		92.59129	1.065520	1	5	3	1	
70.47805	1.335000	35	0	0	4		93.02694	1.061670	1	4	7	2	
70.82064	1.329380	11	0	1	4		93.41923	1.058240	5	3	3	4	
70.97236	1.326910	3	4	2	1		93.73662	1.055490	1	1	8	4	
71.34450	1.320900	124	2	8	2		93.87361	1.054310	1	3	11	1	
71.84463	1.312930	16m	0	2	4		94.23581	1.051210	2m	3	10	2	
71.84463	1.312930	16m	1	7	3		94.23581	1.051210	2m	4	4	3	
72.54826	1.301920	1	4	4	0		94.85936	1.045940	10m	1	1	5	
72.67446	1.299970	1	4	3	1		94.85936	1.045940	10m	5	4	1	
73.11767	1.293180	2	1	1	4		95.48451	1.040740	15m	1	11	3	
73.30085	1.290400	1	3	6	2		95.48451	1.040740	15m	2	7	4	
73.55154	1.286620	6m	2	10	0		95.68984	1.039050	6	3	4	4	
73.55154	1.286620	6m	3	0	3		96.09884	1.035710	2	0	14	0	
73.80211	1.282870	1	1	11	0		96.23324	1.034620	1	4	9	1	
73.92979	1.280970	2	3	1	3		96.90968	1.029190	1m	0	13	2	
74.12831	1.278030	1	1	2	4		96.90968	1.029190	1m	5	0	2	
74.34572	1.274830	10m	0	10	2		97.17443	1.027090	4m	4	5	3	
74.34572	1.274830	10m	2	6	3		97.17443	1.027090	4m	5	1	2	
74.93635	1.266240	4m	3	2	3		97.42696	1.025100	10	1	3	5	
74.93635	1.266240	4m	4	4	1		97.90080	1.021400	8m	4	8	2	
75.27583	1.261370	1	3	8	1		97.90080	1.021400	8m	5	5	1	
75.88514	1.252750	2m	0	4	4		98.15413	1.019440	2	5	2	2	
75.88514	1.252750	2m	1	3	4		98.50576	1.016740	3	2	13	1	
76.02306	1.250820	1	2	10	1		99.79127	1.007070	19m	1	4	5	
76.27027	1.247380	3	1	11	1		99.79127	1.007070	19m	5	3	2	

04-008-6822 Jun 1, 2020 6:53 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
100.03113	1.005300	12m	3	9	3		100.03113	1.005300	12m	4	10	0	

04-011-4223 May 15, 2020 3:03 PM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 293.0 K
Chemical Formula: Ca4 Al2 (C O3) (O H)12 (H2 O)5 **Empirical Formula:** Al2 C Ca4 H22 O20
Weight %: Al9.49 C2.11 Ca28.20 H3.90 O56.29 **Atomic %:** Al4.08 C2.04 Ca8.16 H44.90 O40.82
Compound Name: Calcium Aluminum Carbonate Hydroxide Hydrate **Entry Date:** 09/01/2008
Modification Date: 09/01/2011 **Modifications:** Reflections

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 2.02 **I/Ic - CW ND:** 0.53

Crystal System: Triclinic (Anorthic) **SPGR:** P1 (1)
Author's Unit Cell [a: 5.7747(14) Å **b:** 8.4689(11) Å **c:** 9.923(3) Å **α:** 64.77(2)° **β:** 82.75(2)° **γ:** 81.43(2)°
Volume: 433.02 Å³ **Z:** 1.00 **MolVol:** 433.02 **c/a:** 1.718 **a/b:** 0.682 **c/b:** 1.172 **]**
Calculated Density: 2.18 g/cm³ **Structural Density:** 2.18 g/cm³ **Color:** Colorless **SS/FOM:** F(30) = 204.8(0.0041, 36)
R-factor: 0.027

Space Group: P1 (1) **Molecular Weight:** 568.44 g/mol
Crystal Data [a: 8.469 Å **b:** 9.923 Å **c:** 5.775 Å **α:** 97.25° **β:** 98.57° **γ:** 64.77° **XtlCell Vol:** 433.02 Å³
XtlCell Z: 1.00 **c/a:** 0.682 **a/b:** 0.853 **c/b:** 0.582 **]**
Reduced Cell [a: 5.775 Å **b:** 8.469 Å **c:** 9.923 Å **α:** 64.77° **β:** 82.75° **γ:** 81.43° **RedCell Vol:** 433.02 Å³ **]**

AC Space Group: P1 (1)
AC Unit Cell [a: 5.7747(14) Å **b:** 8.4689(11) Å **c:** 9.923(3) Å **α:** 64.77(2)° **β:** 82.75(2)° **γ:** 81.43(2)° **]**
Space Group Symmetry Operators:

Seq	Operator
1	x,y,z

ADP Type: U
Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Uiso	AET
Ca	1	1a	1	0.51859	0.12026	0.80513	1.0	0.00895	
Ca	2	1a	1	0.47978	0.8882	0.19288	1.0	0.00885	
Ca	3	1a	1	0.01418	0.62138	0.30979	1.0	0.00889	
Ca	4	1a	1	0.977	0.38122	0.69525	1.0	0.00899	
Al	5	1a	1	0.0	2.1E-4	-1.8E-4	1.0	0.00667	
Al	6	1a	1	0.4929	0.50613	0.50349	1.0	0.00685	
O	7	1a	1	0.1537	0.2112	0.9215	1.0	0.0102	
O	8	1a	1	0.8474	0.7896	0.0808	1.0	0.00942	
O	9	1a	1	0.3064	0.891	0.979	1.0	0.01019	
O	10	1a	1	0.6953	0.115	0.0185	1.0	0.00995	
O	11	1a	1	0.3452	0.2925	0.5784	1.0	0.00934	
O	12	1a	1	0.6416	0.7186	0.4259	1.0	0.01099	
O	13	1a	1	0.7985	0.3945	0.4857	1.0	0.00913	
O	14	1a	1	0.1862	0.6129	0.5262	1.0	0.0097	
O	15	1a	1	0.0678	0.9336	0.2025	1.0	0.00923	
O	16	1a	1	0.9301	0.0676	0.7998	1.0	0.00993	
O	17	1a	1	0.4271	0.5743	0.3012	1.0	0.00949	
O	18	1a	1	0.564	0.4347	0.7062	1.0	0.01	
O	19	1a	1	0.5887	0.9018	0.6866	1.0	0.02777	
O	20	1a	1	0.4084	0.1055	0.3088	1.0	0.02065	
O	21	1a	1	0.9112	0.6001	0.8143	1.0	0.02329	
O	22	1a	1	0.3436	0.5562	0.9531	1.0	0.02584	
O	23	1a	1	0.1221	0.9433	0.5596	1.0	0.03031	
O	24	1a	1	0.7565	0.4126	0.0904	1.0	0.02676	
O	25	1a	1	0.0767	0.3952	0.2037	1.0	0.01863	
O	26	1a	1	0.8248	0.1929	0.3118	1.0	0.02107	
C	27	1a	1	0.8852	0.3334	0.2016	1.0	0.01418	
H	28	1a	1	0.181	0.241	0.992	1.0	0.013	
H	29	1a	1	0.842	0.719	0.032	1.0	0.011	
H	30	1a	1	0.309	0.807	0.942	1.0	0.012	
H	31	1a	1	0.69	0.218	0.022	1.0	0.012	
H	32	1a	1	0.347	0.225	0.526	1.0	0.011	
H	33	1a	1	0.664	0.746	0.503	1.0	0.014	
H	34	1a	1	0.798	0.314	0.445	1.0	0.011	
H	35	1a	1	0.173	0.724	0.519	1.0	0.011	
H	36	1a	1	0.004	0.968	0.277	1.0	0.011	
H	37	1a	1	1.025	0.005	0.755	1.0	0.012	
H	38	1a	1	0.502	0.499	0.268	1.0	0.011	
H	39	1a	1	0.504	0.468	0.782	1.0	0.012	
H	40	1a	1	0.676	0.798	0.736	1.0	0.034	
H	41	1a	1	0.43	0.853	0.695	1.0	0.034	
H	42	1a	1	0.308	0.205	0.268	1.0	0.024	
H	43	1a	1	0.543	0.143	0.313	1.0	0.024	
H	44	1a	1	1.049	0.627	0.817	1.0	0.027	
H	45	1a	1	0.872	0.534	0.914	1.0	0.027	
H	46	1a	1	0.49	0.49	0.996	1.0	0.031	
H	47	1a	1	0.233	0.496	1.032	1.0	0.031	

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Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Uiso	AET
H	48	1a	1	-0.022	0.983	0.519	1.0	0.036	
H	49	1a	1	0.241	0.988	0.488	1.0	0.036	

Anisotropic Displacement Parameters:

Atom	Num	Uani11	Uani22	Uani33	Uani12	Uani13	Uani23
Ca	1	0.0079	0.0099	0.0069	1.0E-4	-0.0027	-0.0012
Ca	2	0.0077	0.0095	0.0071	-0.0011	-0.0018	-9.0E-4
Ca	3	0.0078	0.0098	0.007	-2.0E-4	-0.0029	-0.0011
Ca	4	0.0081	0.0096	0.0069	-0.0011	-0.0021	-7.0E-4
Al	5	0.0061	0.0077	0.005	-2.0E-4	-0.0021	-0.0012
Al	6	0.0066	0.0074	0.0051	-2.0E-4	-0.0018	-0.001
O	7	0.012	0.0098	0.0097	-0.0024	-0.0019	-0.0042
O	8	0.0112	0.0099	0.0078	-0.0019	-0.0026	-0.0035
O	9	0.0089	0.0102	0.0115	0.0016	-0.0032	-0.0047
O	10	0.0089	0.0093	0.0115	3.0E-4	-0.0021	-0.0042
O	11	0.0109	0.0102	0.0076	-0.0015	-0.0033	-0.0035
O	12	0.0129	0.0111	0.0091	-0.0023	-0.0022	-0.0036
O	13	0.0094	0.0101	0.008	0.0012	-0.0028	-0.004
O	14	0.0088	0.009	0.0099	0.0	-0.0014	-0.0027
O	15	0.0101	0.0108	0.0069	-5.0E-4	-0.0027	-0.0034
O	16	0.0106	0.0136	0.0052	0.0019	-0.0026	-0.004
O	17	0.0107	0.012	0.0052	0.0018	-0.0024	-0.0035
O	18	0.0106	0.0125	0.0067	-7.0E-4	-0.0023	-0.0035
O	19	0.0261	0.0288	0.0318	0.0032	-0.009	-0.0159
O	20	0.0207	0.0196	0.0243	-0.004	-0.0017	-0.011
O	21	0.0253	0.0226	0.0225	-0.0053	6.0E-4	-0.0096
O	22	0.0242	0.0306	0.0243	5.0E-4	-0.0047	-0.0132
O	23	0.0405	0.0269	0.025	-3.0E-4	-0.0042	-0.0127
O	24	0.0261	0.0298	0.0232	-0.001	-0.0092	-0.0085
O	25	0.0138	0.0203	0.0233	-0.0028	-0.0042	-0.0094
O	26	0.0206	0.0226	0.0236	-0.0042	-0.0023	-0.0122
C	27	0.0127	0.0171	0.0175	0.002	-0.002	-0.0126

Crystal (Symmetry Allowed): Non-centrosymmetric - Enantiomorphic, Optical Activity, Pyro / Piezo (p), Piezo (2nd Harm.)

Subfiles: Inorganic **Pearson Symbol:** aP49.00 **Pearson Symbol w/o H:** aP27
LPF Prototype Structure [Formula Order]: Ca4 Al2 [C O3] [O H]12 [H2 O]5,aP27,1
LPF Prototype Structure [Alpha Order]: Al2 C Ca4 H22 O20,aP27,1 **ANX:** AB2C4X20

Former PDF Numbers: 01-087-0493

References:

Type	DOI	Reference
Primary Reference		Calculated from LPF using POWD-12++.
Structure	10.1107/S0108270198004223	Francois, M., Renaudin, G., Evrard, O. "A Cementitious Compound with Composition 3CaO·Al2O3·CaCO3·11H2O". Acta Crystallogr., Sect. C: Cryst. Struct. Commun. 1998, 54, 1214-1217.

Database Comments: ANX: AB2C4X20. Habit: plate-like. LPF Collection Code: 1413466. Sample Preparation: STARTING MATERIALS:Ca(OH)2. Compound Preparation: hydrothermal synthesis at 393 K and 0.2 GPa for 1 m. CRUCIBLE: sealed silver. Temperature of Data Collection: 293 K. Unit Cell Data Source: Single Crystal.

d-Spacings (198) - Ca4 Al2 (C O3) (O H)12 (H2 O)5 - 04-011-4223 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
9.86974	8.954320	24	0	0	1		26.94439	3.306300	4	0	-1	2	
11.70558	**7.553770**	1000	0	1	1		27.16354	3.280120	2	0	1	3	
15.54352	5.696190	7	1	0	0		28.25294	3.156070	1	-1	2	1	
17.84524	4.966330	9m	1	0	1		29.01420	3.074970	2	0	-2	1	
17.84524	4.966330	9m	1	1	1		29.34130	3.041430	2	0	2	3	
18.13997	4.886300	10	0	-1	1		29.53873	3.021550	6	-1	2	0	
18.33106	4.835790	10	0	1	2		29.68978	3.006520	5	1	1	3	
18.41707	4.813400	11	1	1	0		29.91116	2.984770	3	0	0	3	
19.02673	4.660520	16	-1	0	1		30.44562	2.933580	2	-1	2	2	
19.81366	4.477160	27	0	0	2		31.17498	2.866590	71	1	-1	2	
20.41770	4.346050	42	-1	1	0		31.33325	2.852470	128m	2	0	0	
21.00235	4.226370	4	-1	1	1		31.33325	2.852470	128m	-1	-1	2	
21.20252	4.186920	5	0	2	1		31.67499	2.822470	28	2	1	1	
22.33056	3.977900	42	1	1	2		32.19130	2.778370	51	-1	-2	1	
23.33516	3.808870	58	0	2	0		32.34220	2.765750	10m	0	3	1	
23.53563	**3.776880**	283m	0	2	2		32.34220	2.765750	10m	2	1	0	
23.53563	**3.776880**	283m	-1	-1	1		32.49152	2.753380	4	0	3	2	
24.41112	3.643370	23m	1	0	2		32.85902	2.723420	102	1	0	3	
24.41112	3.643370	23m	1	2	1		33.11778	2.702730	3	-1	1	3	
25.79491	3.450970	46	-1	1	2		33.65521	2.660790	5	-2	0	1	
26.05560	3.417030	4	1	2	2		33.81580	2.648520	19	1	3	2	
26.14187	3.405950	4	-1	0	2		34.04343	2.631330	2	1	3	1	
26.71693	3.333930	2	1	2	0		34.14560	2.623690	4	2	1	2	

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2θ (°)	d (Å)	I	h	k	l	*
34.74955	2.579460	6	-2	1	0	
34.87063	2.570780	10	-1	0	3	
35.43410	2.531180	109	-2	1	1	
35.62694	2.517920	41	0	3	3	
35.99251	2.493180	58	-2	-1	1	
36.14275	2.483160	79m	2	0	2	
36.14275	2.483160	79m	2	2	2	
36.75562	2.443150	73m	0	1	4	
36.75562	2.443150	73m	0	-2	2	
36.97289	2.429290	25	2	-1	1	
37.17461	**2.416570**	185m	0	2	4	
37.17461	**2.416570**	185m	1	3	0	
37.33268	2.406700	27	2	2	0	
38.06894	2.361830	4	-1	3	1	
38.47932	2.337580	159m	1	1	4	
38.47932	2.337580	159m	-1	3	2	
38.60498	2.330260	51	-2	0	2	
38.89359	2.313630	4	-2	1	2	
39.23233	2.294430	23	2	1	3	
39.61017	2.273410	3m	1	-1	3	
39.61017	2.273410	3m	-1	-2	2	
39.80721	2.262610	2	2	2	3	
40.25289	2.238580	28m	0	0	4	
40.25289	2.238580	28m	-1	-1	3	
40.57670	2.221460	3	1	-2	2	
40.72927	2.213490	21	0	-3	1	
41.18526	2.190030	1	0	3	4	
41.52247	2.173020	16m	-1	3	3	
41.52247	2.173020	16m	-2	2	0	
41.71489	2.163440	43m	1	3	4	
41.71489	2.163440	43m	2	-1	2	
41.82883	2.157810	22m	2	3	2	
41.82883	2.157810	22m	-1	1	4	
42.30537	2.134600	17m	1	0	4	
42.30537	2.134600	17m	2	3	1	
42.75514	2.113180	75	-2	2	2	
43.17798	2.093460	48	0	4	2	
43.86929	2.062060	15	2	3	3	
44.28941	2.043470	1	0	4	1	
44.58213	2.030730	22m	2	-2	1	
44.58213	2.030730	22m	-2	1	3	
44.80862	2.020990	19	1	4	3	
45.11179	2.008110	45	1	4	1	
45.57068	1.988950	26m	2	2	4	
45.57068	1.988950	26m	-2	0	3	
46.15378	1.965170	2	0	2	5	
46.21448	1.962730	2	2	1	4	
46.72061	1.942640	47m	0	-1	4	
46.72061	1.942640	47m	-1	3	4	
46.83811	1.938040	52	1	2	5	
47.01474	1.931170	30m	0	1	5	
47.01474	1.931170	30m	-2	2	3	
47.37163	1.917450	1	3	1	1	
47.71526	1.904440	20	0	4	0	
47.92940	1.896430	2	0	-3	2	
48.14499	1.888440	14m	0	4	4	
48.14499	1.888440	14m	1	1	5	
48.25423	1.884420	14m	3	0	1	
48.25423	1.884420	14m	-2	-2	2	
48.49074	1.875780	6m	0	3	5	
48.49074	1.875780	6m	-1	4	2	
48.70911	1.867880	4m	1	3	5	
48.70911	1.867880	4m	1	4	0	
48.93765	1.859690	52m	3	1	2	
48.93765	1.859690	52m	-2	3	1	
49.24898	1.848660	4	-1	4	1	
49.51009	1.839520	4m	-2	3	2	
49.51009	1.839520	4m	-2	-1	3	
49.62728	1.835450	7	3	2	1	
49.73955	1.831570	19	-3	0	1	
49.88207	1.826670	24m	2	-2	2	
49.88207	1.826670	24m	-1	-3	2	
50.02810	1.821680	53m	2	0	4	
50.02810	1.821680	53m	2	4	2	
50.59170	1.802700	1	-2	3	0	
50.72938	1.798130	1m	2	4	3	
50.72938	1.798130	1m	-3	1	0	
50.95002	1.790860	1	0	0	5	
51.07962	1.786620	1m	-1	2	5	
51.07962	1.786620	1m	-3	-1	1	
51.49500	1.773180	2m	1	-3	2	
51.49500	1.773180	2m	2	4	1	
52.25484	1.749170	6m	-1	4	0	
52.25484	1.749170	6m	-2	3	3	
52.42993	1.743740	1	1	0	5	
52.64668	1.737070	1m	3	1	3	
52.64668	1.737070	1m	3	2	3	
53.02780	1.725480	4m	2	2	5	
53.02780	1.725480	4m	-2	2	4	
53.50131	1.711320	11m	1	4	5	
53.50131	1.711320	11m	-1	4	4	
53.69511	1.705600	2m	0	4	5	
53.69511	1.705600	2m	-3	0	2	
54.11822	1.693260	9	3	3	2	
54.32369	1.687340	15m	2	-3	1	
54.32369	1.687340	15m	-3	1	2	
54.56840	1.680350	7	2	1	5	
54.75160	1.675160	9m	3	3	1	
54.75160	1.675160	9m	-1	0	5	
55.08562	1.665790	11m	0	5	3	
55.08562	1.665790	11m	2	4	0	
55.22302	1.661970	42	-3	-2	1	
55.45957	1.655440	44	3	0	3	
55.54303	1.653150	53m	0	-2	4	
55.54303	1.653150	53m	3	3	3	
55.63628	1.650600	40	3	-1	2	
55.93466	1.642500	27	-3	-1	2	
56.02520	1.640060	39m	0	2	6	
56.02520	1.640060	39m	-3	2	1	
56.44818	1.628770	18m	0	-3	3	
56.44818	1.628770	18m	2	-1	4	
56.94900	1.615630	2m	-2	3	4	
56.94900	1.615630	2m	-2	-3	2	
57.06581	1.612600	4m	0	3	6	
57.06581	1.612600	4m	1	-4	1	
57.38175	1.604470	14	3	3	0	
57.50083	1.601430	17	0	-1	5	
57.77345	1.594520	1m	0	1	6	
57.77345	1.594520	1m	-3	2	2	
58.13345	1.585500	9m	3	1	4	
58.13345	1.585500	9m	-1	-2	4	
58.43477	1.578040	21m	1	1	6	
58.43477	1.578040	21m	-2	4	2	
58.64758	1.572820	10	3	-2	1	
59.01671	1.563860	6m	3	3	4	
59.01671	1.563860	6m	-3	1	3	
59.44926	1.553510	6m	1	-3	3	
59.44926	1.553510	6m	-3	0	3	
59.75712	1.546240	15	2	5	3	
60.13258	1.537480	7m	0	-4	2	
60.13258	1.537480	7m	-1	5	3	
60.57348	1.527340	7m	-1	-1	5	
60.57348	1.527340	7m	-2	1	5	
60.74004	1.523550	1m	0	5	0	
60.74004	1.523550	1m	1	5	5	
60.83672	1.521360	1m	0	4	6	
60.83672	1.521360	1m	-3	-2	2	
60.93684	1.519100	2	3	-1	3	
61.12652	1.514840	10	3	4	3	
61.23482	1.512420	19	1	5	0	
61.30846	1.510780	20m	0	5	5	
61.30846	1.510780	20m	-2	4	0	
61.41971	1.508310	17m	-1	5	1	
61.41971	1.508310	17m	-3	2	3	
61.64850	1.503260	12	2	2	6	
61.86674	1.498480	8m	2	3	6	
61.86674	1.498480	8m	-1	1	6	
61.98199	1.495970	4	-1	3	6	
62.14717	1.492390	8	0	0	6	
62.21898	1.490840	9	3	4	1	
62.45931	1.485680	1m	-1	5	4	
62.45931	1.485680	1m	-3	-1	3	

04-013-3303

Jun 3, 2020 3:17 PM (Steve Simner)

Status Primary **Quality Mark:** Blank **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Ca2 Al (S O4)0.5 (O H)6 (H2 O)3 **Empirical Formula:** Al Ca2 H12 O11 S0.5
Weight %: Al8.67 Ca25.75 H3.89 O56.54 S5.15 **Atomic %:** Al3.77 Ca7.55 H45.28 O41.51 S1.89
Compound Name: Calcium Aluminum Sulfate Hydroxide Hydrate **Mineral Name:** Kuzelite, syn **Entry Date:** 09/01/2010
Modification Date: 09/01/2011 **Modifications:** Reflections

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 3.03 **I/Ic - CW ND:** 0.28

Crystal System: Rhombohedral **SPGR:** R-3 (148)
Author's Unit Cell [a: 5.7586(3) Å **c:** 26.7946(12) Å **Volume:** 769.51 Å³ **Z:** 3.00 **MolVol:** 256.50 **c/a:** 4.653 **] Calculated Density:** 2.015 g/cm³ **Structural Density:** 1.98 g/cm³ **SS/FOM:** F(30) = 999.9(0.0006, 31)
R-factor: 0.038

Space Group: R-3 (148) **Molecular Weight:** 311.25 g/mol
Crystal Data [a: 5.759 Å **b:** 5.759 Å **c:** 26.795 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **XtlCell Vol:** 769.51 Å³
XtlCell Z: 3.00 **c/a:** 4.653 **a/b:** 1.000 **c/b:** 4.653 **]**
Reduced Cell [a: 5.759 Å **b:** 5.759 Å **c:** 9.530 Å **α:** 72.41° **β:** 72.41° **γ:** 60.00° **RedCell Vol:** 256.50 Å³ **]**

AC Space Group: R-3H (148)
AC Unit Cell [a: 5.7586(3) Å **b:** 5.7586(3) Å **c:** 26.7946(12) Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	2	-x,-y,-z	3	-y,x-y,z	4	y,-x+y,-z	5	-x+y,-x,z	6	x-y,x,-z

ADP Type: B

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Biso	AET
Al	1	3a	-3.	0.0	0.0	0.0	1.0	1.0111	
Ca	2	6c	3.	0.66666	0.33333	0.02134	1.0	1.17665	
O	3	18f	1	0.2511	-0.0547	0.03731	1.0	1.34998	
H	4	18f	1	0.211	-0.115	0.0667	1.0	3.2	
O	5	6c	3.	0.66666	0.33333	0.11542	1.0	3.50107	
S	6	6c	3.	0.0	0.0	0.4955	0.25	2.22664	
O	7	6c	3.	0.0	0.0	0.5486	0.25	4.61106	
O	8	18f	1	0.2688	0.1821	0.4771	0.2502	4.63661	
O	9	18f	1	0.2688	0.1821	0.4771	0.1668	4.63661	

Anisotropic Displacement Parameters:

Atom	Num	Bani11	Bani22	Bani33	Bani12	Bani13	Bani23
Al	1	0.57	0.57	1.9	0.29	0.0	0.0
Ca	2	0.64	0.64	2.25	0.32	0.0	0.0
O	3	0.91	1.07	2.13	0.54	-0.03	0.17
O	5	3.83	3.83	2.85	1.92	0.0	0.0
S	6	1.96	1.96	2.76	0.98	0.0	0.0
O	7	6.1	6.1	1.7	3.1	0.0	0.0
O	8	3.58	4.64	5.07	1.59	0.23	-0.04
O	9	3.58	4.64	5.07	1.59	0.23	-0.04

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Cement and Hydration Product, Inorganic, Mineral Related (Mineral, Synthetic) **Pearson Symbol:** hR26.50
Pearson Symbol w/o H: hR14.5
LPF Prototype Structure [Formula Order]: Ca2 Al (S O4)0.5 (O H)6 (H2 O)3,hR63,148
LPF Prototype Structure [Alpha Order]: Al Ca2 H12 O11 S0.5,hR63,148 **ANX:** A2B3C6X33

Former PDF Numbers: 01-073-6176, 01-083-1289

References:

Type	DOI	Reference
Primary Reference		Calculated from LPF using POWD-12++.
Structure		Allmann R. "Refinement of the hydrid layer structure (Ca2Al(OH)6)+·(1/2SO4·3H2O)-". Neues Jahrb. Mineral., Monatsh. 1977136-144.

Database Comments: ANX: A2B3C6X33. LPF Collection Code: 1214961. Minor Warning: Density calculated using chemical formula and reported structure differ by 1.736%. short interatomic distances for partly occupied sites. Significant Warning: Significant warning from the LPF Editor exist. Unit Cell Data Source: Single Crystal.

04-013-3303 Jun 3, 2020 3:17 PM (Steve Simner)

d-Spacings (137) - Ca2 Al (S O4)0.5 (O H)6 (H2 O)3 - 04-013-3303 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
9.89499	**8.931530**	1000	0	0	3		78.73912	1.214340	1	0	4	5	
18.07807	4.902890	28	1	0	1		79.14407	1.209140	1m	2	1	-17	
18.97225	4.673780	2	0	1	2		79.14407	1.209140	1m	2	2	-12	
19.86471	**4.465770**	248	0	0	6		79.64495	1.202790	8	1	3	-11	
22.20432	**4.000230**	233	1	0	4		81.04816	1.185470	1	4	0	7	
24.36074	3.650790	35	0	1	5		81.23955	1.183160	4	1	0	22	
29.39021	3.036480	1	1	0	7		81.48795	1.180180	2	2	0	20	
29.98921	2.977180	1	0	0	9		82.48356	1.168440	2	0	4	8	
31.03389	2.879300	184	1	1	0		82.64816	1.166530	7	1	1	-21	
32.16644	2.780460	15	0	1	8		84.23787	1.148520	6	3	1	-13	
32.64947	2.740420	95	1	1	-3		84.73193	1.143080	1	3	2	1	
36.14787	2.482820	11	0	2	1		85.01738	1.139970	3	3	2	-2	
36.62674	2.451450	113	2	0	2		85.53044	1.134440	2	0	1	23	
37.12127	2.419920	203	1	1	-6		85.91075	1.130390	4	4	0	10	
38.09373	2.360350	52	1	0	10		86.02418	1.129190	8	1	2	-19	
38.49113	2.336890	55	0	2	4		86.15696	1.127790	5	2	3	-4	
39.84061	2.260790	35	2	0	5		86.80289	1.121050	2m	1	3	-14	
40.36011	2.232880	14	0	0	12		86.80289	1.121050	2m	2	2	15	
41.21063	2.188740	50	0	1	11		87.01130	1.118900	6	3	2	-5	
43.26763	2.089330	18	0	2	7		87.90249	1.109840	3m	0	3	18	
43.69878	2.069710	110	1	1	-9		87.90249	1.109840	3m	0	4	11	
45.30223	2.000110	37	2	0	8		89.28516	1.096200	3	2	3	-7	
47.70435	1.904850	32	1	0	13		89.47456	1.094370	2	0	2	22	
48.36671	1.880300	9	1	2	-1		89.72125	1.092000	1	2	1	-20	
48.74579	1.866560	13	2	1	-2		90.11277	1.088270	14	1	4	0	
49.91945	1.825390	83	0	2	10		90.70606	1.082690	3	3	2	-8	
50.24036	1.814480	25	1	2	-4		90.96517	1.080280	9	1	4	-3	
51.08912	1.786310	6m	0	0	15		92.48679	1.066450	7	3	1	-16	
51.08912	1.786310	6m	0	1	14		93.52397	1.057330	12	1	4	-6	
51.34058	1.778150	11	2	1	-5		93.73894	1.055470	2	2	0	23	
51.76766	1.764480	15	1	1	-12		94.11860	1.052210	12	2	3	-10	
52.47138	1.742460	6	2	0	11		94.63103	1.047860	1	1	0	25	
54.19542	1.691030	6	1	2	-7		95.01112	1.044670	1	0	4	14	
55.20896	1.662360	76	3	0	0		95.46149	1.040930	1	1	1	-24	
55.92836	1.642670	13	2	1	-8		95.62047	1.039620	1	1	3	-17	
56.24017	1.634300	50	0	3	3		96.11484	1.035580	2	3	2	-11	
58.00679	1.588660	27m	0	2	13		97.69905	1.022970	5	1	2	-22	
58.00679	1.588660	27m	1	0	16		97.80686	1.022130	5	4	1	-9	
59.26372	1.557930	31	0	3	6		99.10945	1.012160	2	0	3	21	
59.95194	1.541680	29	1	2	-10		99.49917	1.009240	1	0	1	26	
60.98926	1.517920	3	1	1	-15		100.71243	1.000330	8m	3	2	13	
61.66579	1.502880	1	0	1	17		100.71243	1.000330	8m	4	0	16	
62.22408	1.490730	14	2	1	-11		101.21355	0.996728	1	0	5	1	
64.10609	1.451430	6	3	0	9		101.50327	0.994666	1	5	0	2	
64.69416	1.439650	28	2	2	0		101.82522	0.992392	1	0	0	27	
65.63359	1.421300	12	2	2	-3		102.02530	0.990988	1	2	1	-23	
67.25178	1.390980	34m	0	2	16		102.52932	0.987482	5	3	1	-19	
67.25178	1.390980	34m	2	1	13		103.32630	0.982027	1	3	2	-14	
67.78500	1.381330	4	3	1	-1		103.54054	0.980579	3	5	0	5	
68.09133	1.375860	4	1	3	-2		103.88498	0.978267	1m	0	4	17	
68.41089	1.370210	18	2	2	-6		103.88498	0.978267	1m	4	1	12	
69.16912	1.357030	9	1	0	19		105.89058	0.965190	2	0	5	7	
69.31091	1.354600	12	1	3	4		106.34392	0.962323	1	1	3	-20	
69.99774	1.342980	2	2	1	-14		106.75241	0.959767	4	3	3	0	
70.21955	1.339280	1	1	3	-5		107.37315	0.955932	2	5	0	8	
70.57461	1.333410	1m	2	0	17		107.64465	0.954273	4	3	3	-3	
70.57461	1.333410	1m	3	0	12		107.94925	0.952425	1	2	0	26	
71.25619	1.322320	3	1	1	-18		109.24860	0.944696	4	2	3	-16	
72.61745	1.300850	1	3	1	-7		109.73083	0.941890	1	2	4	1	
72.92831	1.296070	7	2	2	-9		110.09291	0.939805	1m	1	0	28	
74.10055	1.278440	3	1	3	-8		110.09291	0.939805	1m	4	2	2	
74.27082	1.275930	1	0	0	21		110.34910	0.938341	5m	1	1	27	
75.94295	1.251940	15	1	2	-16		110.34910	0.938341	5m	3	3	-6	
76.41120	1.245430	1	4	0	1		110.98336	0.934756	5m	0	5	10	
76.70356	1.241410	1	0	4	2		110.98336	0.934756	5m	4	0	19	
77.61676	1.229070	7	3	1	-10		111.53180	0.931701	1	1	2	-25	
77.73242	1.227530	3	0	2	19		111.95350	0.929380	1	1	4	-15	
77.86886	1.225720	4	4	0	4		112.16482	0.928226	1	4	2	5	
78.53915	1.216930	3	0	3	15								

04-013-3691 May 15, 2020 2:18 PM (Steve Simner)

Status Primary **Quality Mark:** Star **Environment:** Ambient **Temp:** 296.0 K
Chemical Formula: Ca6 Al2 (S O4)3 (O H)12 (H2 O)26 **Empirical Formula:** Al2 Ca6 H64 O50 S3
Weight %: Al4.30 Ca19.16 H5.14 O63.74 S7.66 **Atomic %:** Al1.60 Ca4.80 H51.20 O40.00 S2.40
Compound Name: Calcium Aluminum Sulfate Hydroxide Hydrate **Mineral Name:** Ettringite, syn **Entry Date:** 09/01/2010
Modification Date: 09/01/2011 **Modifications:** Reflections

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 1.65 **I/Ic - CW ND:** 0.35

Crystal System: Hexagonal **SPGR:** P31c (159)
Author's Unit Cell [a: 11.229(1) Å **c:** 21.478(3) Å **Volume:** 2345.34 Å³ **Z:** 2.00 **MolVol:** 1172.67 **c/a:** 1.913 **] Calculated Density:** 1.777 g/cm³ **Structural Density:** 1.78 g/cm³ **SS/FOM:** F(30) = 999.9(0.0000, 33)
R-factor: 0.033

Space Group: P31c (159) **Molecular Weight:** 1255.08 g/mol
Crystal Data [a: 11.229 Å **b:** 11.229 Å **c:** 21.478 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00°
XtlCell Vol: 2345.34 Å³ **XtlCell Z:** 2.00 **c/a:** 1.913 **a/b:** 1.000 **c/b:** 1.913 **]**
Reduced Cell [a: 11.229 Å **b:** 11.229 Å **c:** 21.478 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00°
RedCell Vol: 2345.34 Å³ **]**

AC Space Group: P31c (159)
AC Unit Cell [a: 11.229(1) Å **b:** 11.229(1) Å **c:** 21.478(3) Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	3	-x+y,-x,z	5	-x,-x+y,z+1/2
2	-y,x-y,z	4	y,x,z+1/2	6	x-y,-y,z+1/2

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	IDP	AET
Al	1	2a	3..	0.0	0.0	0.0	1.0		
Al	2	2a	3..	0.0	0.0	0.25	1.0		
Ca	3	6c	1	0.002	0.813	0.875	1.0		
Ca	4	6c	1	0.993	0.184	0.122	1.0		
O	5	6c	1	0.997	0.122	0.945	1.0		
H	6	6c	1	0.985	0.192	0.966	1.0		
O	7	6c	1	0.002	0.869	0.054	1.0		
H	8	6c	1	0.019	0.81	0.026	1.0		
O	9	6c	1	0.999	0.128	0.801	1.0		
H	10	6c	1	0.99	0.2	0.778	1.0		
O	11	6c	1	0.003	0.871	0.196	1.0		
H	12	6c	1	0.006	0.791	0.213	1.0		
O	13	6c	1	0.994	0.342	0.046	1.0		
H	14	6c	1	0.06	0.405	0.017	1.0		
H	15	6c	1	0.92	0.362	0.044	1.0		
O	16	6c	1	0.018	0.684	0.957	1.0		
H	17	6c	1	0.936	0.599	0.965	1.0		
H	18	6c	1	0.091	0.666	0.965	1.0		
O	19	6c	1	0.003	0.34	0.206	1.0		
H	20	6c	1	0.078	0.401	0.232	1.0		
H	21	6c	1	0.923	0.334	0.221	1.0		
O	22	6c	1	0.99	0.63	0.795	1.0		
H	23	6c	1	0.918	0.536	0.793	1.0		
H	24	6c	1	0.073	0.627	0.792	1.0		
O	25	6c	1	0.265	0.406	0.622	1.0		
H	26	6c	1	0.293	0.476	0.654	1.0		
H	27	6c	1	0.295	0.456	0.584	1.0		
O	28	6c	1	0.789	0.624	0.362	1.0		
H	29	6c	1	0.754	0.556	0.33	1.0		
H	30	6c	1	0.764	0.573	0.4	1.0		
O	31	6c	1	0.266	0.408	0.126	1.0		
H	32	6c	1	0.337	0.388	0.116	1.0		
H	33	6c	1	0.291	0.492	0.104	1.0		
O	34	6c	1	0.752	0.598	0.865	1.0		
H	35	6c	1	0.699	0.639	0.877	1.0		
H	36	6c	1	0.687	0.502	0.86	1.0		
O	37	2b	3..	0.33333	0.66666	0.425	1.0		
O	38	2b	3..	0.33333	0.66666	0.819	1.0		
O	39	2b	3..	0.33333	0.66666	0.076	1.0		
O	40	6c	1	0.195	0.628	0.519	1.0		
O	41	6c	1	0.195	0.62	0.724	1.0		
O	42	6c	1	0.192	0.585	0.982	1.0		
O	43	6c	1	0.227	0.685	0.243	0.667		
H	44	6c	1	0.325	0.728	0.239	0.667		
H	45	6c	1	0.2	0.727	0.211	0.667		
S	46	2b	3..	0.33333	0.66666	0.492	1.0		
S	47	2b	3..	0.33333	0.66666	0.751	1.0		
S	48	2b	3..	0.33333	0.66666	0.009	1.0		

04-013-3691 Mav 15. 2020 2:18 PM (Steve Simner)
Crystal (Symmetry Allowed): Non-centrosymmetric

Subfiles: Cement and Hydration Product, Common Phase, Inorganic, Mineral Related (Mineral, Synthetic)
Mineral Classification: Ettringite (supergroup), 2H (group) **Pearson Symbol:** hP250.00
Pearson Symbol w/o H: hP122
LPF Prototype Structure [Formula Order]: Ca6 Al2 (S O4)3 (O H)12 (H2 O)26,hP124,159
LPF Prototype Structure [Alpha Order]: Al2 Ca6 H64 O50 S3,hP124,159

Cross-Ref PDF #'s: 01-084-8852 (Alternate), 04-011-5267 (Alternate)

References:

Type	DOI	Reference
Primary Reference		Calculated from LPF using POWD-12++.
Structure		Goetz Neunhoeffer F., Neubauer J. "Refined ettringite (Ca6Al2(SO4)3(OH)12·26H2O) structure for quantitative X-ray diffraction analysis". Powder Diffr. 2006, 21, 4-11.

Database Comments: LPF Collection Code: 1253398. Sample Preparation: STARTINGMATERIALS: Ca O (sucrose aqueous solution), Al2 (S O4)3 (H2 O) n (CO2-free aqueous solution). COMPOUND PREPARATION: reacted at 296 K for 1 d, precipitate washed with dilute ammonia, dried. Temperature of Data Collection: 296 K. Unit Cell Data Source: Powder Diffraction.

d-Spacings (136) - Ca6 Al2 (S O4)3 (O H)12 (H2 O)26 - 04-013-3691 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
8.22641	10.739000	26	0	0	2		42.69981	2.115790	6	4	0	5	
9.08625	**9.724600**	1000	1	0	0		42.78445	2.111800	6	-4	-1	1	
9.97636	8.858860	82	1	0	1		43.09583	2.097260	3	1	0	10	
12.26863	7.208340	14	1	0	2		43.43141	2.081830	21	-4	-1	2	
15.35577	5.765410	42	1	0	3		43.74434	2.067660	9	3	0	8	
15.77110	**5.614500**	478	1	1	0		43.91051	2.060220	36	-3	-2	4	
16.49561	5.369500	4	0	0	4		44.49302	2.034590	2	-4	-1	3	
17.81194	4.975540	131	-1	-1	2		44.69787	2.025740	2	-3	-1	7	
18.23027	4.862300	28	2	0	0		45.03938	2.011170	3	4	0	6	
18.86322	4.700550	265	1	0	4		45.16115	2.006030	11	-1	-1	10	
20.02936	4.429430	5	2	0	2		45.79128	1.979880	9	-3	-2	5	
22.08073	4.022340	54	2	0	3		45.94655	1.973550	18	-4	-1	4	
22.61026	3.929320	11	1	0	5		46.16646	1.964660	1	2	0	10	
22.89836	**3.880530**	399	-1	-1	4		46.66259	1.944920	66	5	0	0	
24.19415	3.675550	24	2	1	0		46.78107	1.940270	17	-2	-2	8	
24.55126	3.622890	16	-2	-1	1		47.25787	1.921800	3	3	0	9	
24.68078	3.604170	87	2	0	4		47.45330	1.914340	3m	1	0	11	
25.59469	3.477510	186	-2	-1	2		47.45330	1.914340	3m	5	0	2	
26.51146	3.359300	5	1	0	6		47.76001	1.902760	15m	-3	-1	8	
27.25084	3.269810	43	-2	-1	3		47.76001	1.902760	15m	-4	-1	5	
27.49323	3.241530	112	3	0	0		48.01200	1.893360	8	-3	-2	6	
27.81082	3.205230	7	3	0	1		48.46022	1.876890	6	5	0	3	
28.74417	3.103240	8	3	0	2		48.60879	1.871500	6	3	3	0	
29.42449	3.033020	7	-2	-1	4		49.08617	1.854410	52	-2	-1	10	
29.57056	3.018370	57	-1	-1	6		49.39003	1.843710	39	-3	-3	2	
30.24113	2.952950	2	3	0	3		49.82409	1.828660	19m	5	0	4	
30.52545	2.926090	6	1	0	7		49.82409	1.828660	19m	-4	-2	1	
30.99637	2.882700	2	2	0	6		50.33054	1.811440	26	-4	-2	2	
31.85127	2.807250	23	2	2	0		50.54038	1.804410	10	-3	-2	7	
32.02120	2.792740	21	-2	-1	5		50.61003	1.802090	11	4	0	8	
32.23074	2.775060	256	3	0	4		50.98144	1.789830	24m	0	0	12	
32.95147	2.715990	19	-2	-2	2		50.98144	1.789830	24m	-3	-1	9	
33.18866	2.697120	34	3	1	0		51.28118	1.780070	8	-4	-2	3	
33.34603	2.684750	59	0	0	8		51.68115	1.767230	34	-3	-3	4	
33.45698	2.676100	18	-3	-1	1		51.90068	1.760270	29	1	0	12	
34.25068	2.615880	131	-3	-1	2		52.33788	1.746590	13m	5	1	0	
34.63843	2.587480	8m	1	0	8		52.33788	1.746590	13m	-4	-1	7	
34.63843	2.587480	8m	3	0	5		52.52394	1.740840	2	5	1	1	
34.95962	2.564440	314	-2	-1	6		52.59191	1.738750	2	-4	-2	4	
35.53899	2.523950	23	-3	-1	3		53.07887	1.723940	16	-5	-1	2	
36.07347	2.487770	23	-2	-2	4		53.34820	1.715870	3	-3	-2	8	
36.94358	2.431150	11	4	0	0		53.70599	1.705280	35	-1	-1	12	
37.08696	2.422080	20	-1	-1	8		53.99544	1.696820	7	-5	-1	3	
37.27727	2.410150	61	-3	-1	4		54.24367	1.689640	3	-4	-2	5	
37.91358	2.371150	3	4	0	2		54.57544	1.680150	43	-3	-1	10	
38.26328	2.350280	33	2	0	8		54.84386	1.672560	3	3	0	11	
38.82290	2.317680	6	1	0	9		55.12044	1.664820	74	-4	-1	8	
39.09735	2.302040	5	4	0	3		55.26055	1.660930	33	-5	-1	4	
39.41547	2.284190	1	-3	-1	5		55.92725	1.642700	1	5	0	7	
40.39598	2.230980	52m	3	0	7		56.21732	1.634910	3	-4	-2	6	
40.39598	2.230980	52m	3	2	0		56.44667	1.628810	2m	1	0	13	
40.81593	2.208990	284	-2	-2	6		56.44667	1.628810	2m	-3	-2	9	
41.29743	2.184340	32	-3	-2	2		56.75199	1.620770	54	6	0	0	
41.90386	2.154120	151	-3	-1	6		57.19827	1.609180	4	-2	-1	12	
42.40197	2.129960	21	-3	-2	3		57.60782	1.598710	14	4	3	0	
42.56707	2.122080	21	4	1	0		57.78218	1.594300	2	4	3	1	

04-013-3691 May 15, 2020 2:18 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
58.12140	1.585800	4	-4	-1	9		60.35957	1.532240	4	-4	-3	4	
58.30309	1.581290	23	-4	-3	2		60.79297	1.522350	5m	4	0	11	
58.55647	1.575050	40m	5	0	8		60.79297	1.522350	5m	5	2	3	
58.55647	1.575050	40m	-4	-2	7		61.05201	1.516510	15	-4	-2	8	
58.77515	1.569710	3	-5	-1	6		61.38047	1.509180	43	-2	-2	12	
58.89299	1.566850	6	3	0	12		62.00086	1.495560	11	-5	-2	4	
59.16516	1.560290	3	-4	-3	3		62.19626	1.491330	7	-3	-1	12	
59.29512	1.557180	4	5	2	0		62.58538	1.482990	2	6	1	0	
59.52897	1.551620	1m	6	0	4		62.73134	1.479890	6m	1	1	14	
59.52897	1.551620	1m	-5	-2	1		62.73134	1.479890	6m	-6	-1	1	
59.71244	1.547290	3	-3	-2	10		62.89274	1.476480	1	6	0	6	
59.97811	1.541070	10	5	2	2		63.23549	1.469300	5	3	2	11	

04-015-4253 May 15, 2020 3:21 PM (Steve Simner)

Status Primary **Quality Mark:** Indexed **Environment:** Ambient **Temp:** 298.0 K (Assigned by ICDD editor)
Chemical Formula: Mg0.67 Al0.33 (C O3)0.17 (O H)2 (H2 O)0.5 **Empirical Formula:** Al0.33 C0.17 H3 Mg0.67 O3.01
Weight %: Al11.36 C2.60 H3.86 Mg20.77 O61.42 **Atomic %:** Al4.60 C2.37 H41.78 Mg9.33 O41.92
Compound Name: Magnesium Aluminum Carbonate Hydroxide Hydrate **Mineral Name:** Hydrotalcite
Entry Date: 09/01/2011

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 2.78 **I/Ic - CW ND:** 0.71

Crystal System: Rhombohedral **SPGR:** R-3m (166)
Author's Unit Cell [a: 3.054(3) Å **c:** 22.81(2) Å **Volume:** 184.24 Å³ **Z:** 3.00 **MolVol:** 61.41 **c/a:** 7.469 **]**
Calculated Density: 2.12 g/cm³ **Structural Density:** 2.09 g/cm³ **Color:** Colorless **SS/FOM:** F(30) = 999.9(0.0001, 32)
R-factor: 0.074

Space Group: R-3m (166) **Molecular Weight:** 78.41 g/mol
Crystal Data [a: 3.054 Å **b:** 3.054 Å **c:** 22.810 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00° **XtlCell Vol:** 184.24 Å³
XtlCell Z: 3.00 **c/a:** 7.469 **a/b:** 1.000 **c/b:** 7.469 **]**
Reduced Cell [a: 3.054 Å **b:** 3.054 Å **c:** 7.805 Å **α:** 78.72° **β:** 78.72° **γ:** 60.00° **RedCell Vol:** 61.41 Å³ **]**

AC Space Group: R-3mH (166)
AC Unit Cell [a: 3.054(3) Å **b:** 3.054(3) Å **c:** 22.81(2) Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	3	-y,x-y,z	5	-x+y,-x,z	7	-y,-x,z	9	x,x-y,z	11	-x+y,y,z
2	-x,-y,-z	4	y,-x+y,-z	6	x-y,x,-z	8	y,x,-z	10	-x,-x+y,-z	12	x-y,-y,-z

ADP Type: B

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Biso	AET
Mg	1	3a	-3m	0.0	0.0	0.0	0.667	1.56674	
Al	2	3a	-3m	0.0	0.0	0.0	0.333	1.56674	
O	3	6c	3m	0.0	0.0	0.3771	1.0	2.2301	
H	4	6c	3m	0.0	0.0	0.4144	1.0	2.70679	
O	5	18h	.m	0.0925	-0.0925	0.5	0.0835	7.43257	
O	6	18h	.m	0.0925	-0.0925	0.5	0.0835	7.43257	
C	7	6c	3m	0.0	0.0	0.16666	0.083	4.72022	

Anisotropic Displacement Parameters:

Atom	Num	Bani11	Bani22	Bani33	Bani12	Bani13	Bani23
Mg	1	1.47	1.47	1.76	0.735	0.0	0.0
Al	2	1.47	1.47	1.76	0.735	0.0	0.0
O	3	2.4	2.4	1.89	1.2	0.0	0.0
H	4	3.02	3.02	2.08	1.51	0.0	0.0
O	5	11.0	11.0	2.03	6.8	0.0	0.0
O	6	11.0	11.0	2.03	6.8	0.0	0.0
C	7	6.04	6.04	2.08	3.02	0.0	0.0

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Inorganic, Mineral Related (Mineral, Natural) **Mineral Classification:** Hydrotalcite (family), 7R (supergroup)
Pearson Symbol: hR7.18 **Pearson Symbol w/o H:** hR4.18
LPF Prototype Structure [Formula Order]: (Mg0.67 Al0.33) (C O3)0.17 (O H)2 (H2 O)0.5,hR33,166
LPF Prototype Structure [Alpha Order]: Al0.33 C0.17 H3 Mg0.67 O3.01,hR33,166 **ANX:** AB6X18

Cross-Ref PDF #'s: 04-014-8854 (Alternate) **Former PDF Numbers:** 01-070-2151

References:

Type	DOI	Reference
Primary Reference		Calculated from LPF using POWD-12++.
Structure		Allmann R., Jepsen H.P. "Die Struktur des Hydrotalkits". Neues Jahrb. Mineral., Monatsh. 1969544-551.

Database Comments: ANX: AB6X18. LPF Collection Code: 1714293. Sample Source or Locality: Specimen from Vezna, Vysocina Region, Czech Republic. Minor Warning: Density calculated using chemical formula and reported structure differ by 1.415%. OH2 refined as a group without locating individual atomic coordinates. 7%<R factor<12% (for single crystal). LPF Editor Comment: short interatomic distances for partly occupied sites. Unit Cell Data Source: Single Crystal.

d-Spacings (65) - Mg0.67 Al0.33 (C O3)0.17 (O H)2 (H2 O)0.5 - 04-015-4253 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
11.62902	**7.603330**	1000	0	0	3		23.37998	**3.801670**	287	0	0	6	

04-015-4253 May 15, 2020 3:21 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
34.09804	2.627240	9	1	0	1		97.21493	1.026770	2	2	0	14	
34.79116	2.576470	226	0	1	2		100.93884	0.998697	1	2	1	1	
35.38702	2.534440	18	0	0	9		101.33834	0.995838	4	1	2	2	
37.45146	2.399340	1	1	0	4		102.94242	0.984641	1	2	1	4	
39.34516	2.288110	231	0	1	5		104.15219	0.976487	10	1	2	5	
44.06059	2.053550	1	1	0	7		104.35209	0.975163	6	1	1	18	
46.81226	**1.939050**	239	0	1	8		105.21432	0.969527	5	0	2	16	
47.81152	1.900830	5	0	0	12		105.87391	0.965296	2	1	0	22	
52.96625	1.727340	56	1	0	10		107.41166	0.955696	1	2	1	7	
56.33137	1.631870	38	0	1	11		108.28284	0.950417	3	0	0	24	
60.58838	1.527000	70	1	1	0		109.47817	0.943356	9	1	2	8	
61.92959	1.497110	75	1	1	3		112.09566	0.928603	1	0	1	23	
63.58224	1.462120	31	1	0	13		114.55414	0.915589	3	2	1	10	
65.85954	1.416970	21	1	1	6		117.61001	0.900481	6	1	2	11	
67.45957	1.387200	12	0	1	14		120.12622	0.888878	3	0	2	19	
71.38812	1.320200	1	0	2	1		120.97929	0.885108	6	1	1	21	
71.80103	1.313620	9	2	0	2		121.78669	0.881614	4	3	0	0	
72.16104	1.307950	1	1	1	9		123.18110	0.875746	5	0	3	3	
73.44453	1.288230	1	0	2	4		124.95434	0.868580	6	2	1	13	
74.66761	1.270130	20	2	0	5		126.21796	0.863670	1	2	0	20	
74.86845	1.267220	7	0	0	18		126.52028	0.862519	1	1	0	25	
75.73001	1.254930	26	1	0	16		127.50756	0.858823	2	3	0	6	
77.89607	1.225360	2	0	2	7		129.38317	0.852061	2	1	2	14	
79.89368	1.199670	16	2	0	8		131.50322	0.844815	1	0	0	27	
80.63902	1.190450	1	1	1	12		135.35083	0.832693	2m	0	1	26	
84.64244	1.144060	5	0	2	10		135.35083	0.832693	2m	3	0	9	
87.39430	1.114980	8	2	0	11		140.47540	0.818486	5	2	1	16	
89.59822	1.093180	5	1	0	19		141.48457	0.815936	1	0	2	22	
90.33285	1.086190	2	0	0	21		145.34896	0.806890	6	1	1	24	
91.26516	1.077510	1	1	1	15		147.84525	0.801633	1	1	2	17	
93.66947	1.056070	7	0	2	13		148.78019	0.799779	1	3	0	12	
94.69986	1.047280	1	0	1	20								

04-015-8262

May 15, 2020 1:16 PM (Steve Simner)

Status Alternate **Quality Mark:** Star **Environment:** Non-ambient Pressure **Temp:** 298.0 K (Assigned by ICDD editor)
Pressure: 0.0001 GPa **Chemical Formula:** Ca (S O4) (H2 O)2 **Empirical Formula:** Ca H4 O6 S
Weight %: Ca23.28 H2.34 O55.76 S18.62 **Atomic %:** Ca8.33 H33.33 O50.00 S8.33
Compound Name: Calcium Sulfate Hydrate **Alternate Name:** gypsum **Entry Date:** 09/01/2012

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 1.73 **I/Ic - CW ND:** 0.6

Crystal System: Monoclinic **SPGR:** C2/c (15)
Author's Unit Cell [a: 6.277(2) Å **b:** 15.181(6) Å **c:** 5.672(2) Å **β:** 114.11(2)° **Volume:** 493.34 Å³ **Z:** 4.00
MolVol: 123.33 **c/a:** 0.904 **a/b:** 0.413 **c/b:** 0.374 **]** **Calculated Density:** 2.318 g/cm³
Structural Density: 2.32 g/cm³ **SS/FOM:** F(30) = 168.6(0.0051, 35)

Space Group: C2/c (15) **Molecular Weight:** 172.17 g/mol
Crystal Data [a: 6.277 Å **b:** 15.181 Å **c:** 5.672 Å **α:** 90.00° **β:** 114.11° **γ:** 90.00° **XtlCell Vol:** 493.34 Å³
XtlCell Z: 4.00 **c/a:** 0.904 **a/b:** 0.413 **c/b:** 0.374 **]**
Reduced Cell [a: 5.672 Å **b:** 6.277 Å **c:** 8.214 Å **α:** 67.54° **β:** 81.02° **γ:** 65.89° **RedCell Vol:** 246.67 Å³ **]**

AC Space Group: C12/c1 (15)
AC Unit Cell [a: 6.277(2) Å **b:** 15.181(6) Å **c:** 5.672(2) Å **α:** 90° **β:** 114.11(2)° **γ:** 90° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	2	-x,-y,-z	3	-x,y,-z+1/2	4	x,-y,z+1/2

ADP Type: U

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Uiso	AET
S	1	4e	2	0.0	0.32727	0.75	1.0	0.0099	
Ca	2	4e	2	0.0	0.1705	0.25	1.0	0.0117	
O	3	8f	1	0.08319	0.27218	0.59103	1.0	0.0169	
O	4	8f	1	0.19997	0.38195	0.91298	1.0	0.0169	
O	5	8f	1	-0.20823	0.06826	-0.07831	1.0	0.0241	
H	6	8f	1	-0.258	0.087	-0.234	1.0	0.033	
H	7	8f	1	-0.244	0.02	-0.077	1.0	0.044	

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Cement and Hydration Product, Ceramic (Bioceramic), Common Phase, Forensic, Inorganic, Mineral Related, Pharmaceutical (Excipient)

Pearson Symbol: mC48.00 **Pearson Symbol w/o H:** mC32
LPF Prototype Structure [Formula Order]: Ca (S O4) (H2 O)2,mS32,15
LPF Prototype Structure [Alpha Order]: Ca H4 O6 S,mS32,15 **ANX:** ABX6

Cross-Ref PDF #'s: 00-003-0044 (Deleted), 00-003-0053 (Deleted), 00-006-0047 (Deleted), 01-072-0596 (Alternate), 01-074-1905 (Alternate), 01-078-6179 (Alternate), 01-078-6180 (Alternate), 04-008-9805 (Alternate), 04-009-1810 (Alternate), 04-009-3817 (Alternate), 04-010-9409 (Primary), 04-012-1412 (Alternate), 04-015-7420 (Alternate), 04-015-8263 (Alternate), 04-015-8264 (Alternate), 04-015-8265 (Alternate), 04-015-8266 (Alternate)

Former PDF Numbers: 01-076-8724

References:

Type	DOI	Reference
Primary Reference		Calculated from LPF using POWD-12++.
Structure	10.2138/am.2008.2917	Comodi P., Nazzareni S., Zanazzi P.F., Speziale S. "High-pressure behavior of gypsum: A single-crystal X-ray study". Am. Mineral. 2008, 93, 1530-1537.

Database Comments: ANX: ABX6. LPF Collection Code: 1221415. Pressure of Datacollection: 0.0001 GPa. Sample Source or Locality: Specimen from Valle di Caramanico, Abruzzo, Italy. Unit Cell Data Source: Single Crystal.

d-Spacings (199) - Ca (S O4) (H2 O)2 - 04-015-8262 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
11.64874	**7.590500**	783	0	2	0		28.14652	3.167760	42	1	1	1	
16.52393	5.360360	1	1	1	0		29.15062	**3.060890**	699	0	4	1	
18.71755	4.736800	14	-1	1	1		31.14078	2.869660	477m	2	0	0	
20.75071	**4.277050**	1000	0	2	1		31.14078	2.869660	477m	-2	2	1	
23.42009	3.795250	129m	0	4	0		32.11567	2.784740	91	-1	1	2	
23.42009	3.795250	129m	1	3	0		32.80156	2.728060	11	1	3	1	
25.05256	3.551520	1	-1	3	1		33.40456	2.680180	343m	1	5	0	

04-015-8262 May 15, 2020 1:16 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
33.40456	2.680180	343m	2	2	0		71.29970	1.321620	22	-4	6	2	
34.55976	2.593190	43m	0	0	2		71.85918	1.312700	2m	-3	3	4	
34.55976	2.593190	43m	-1	5	1		71.85918	1.312700	2m	-3	9	1	
35.44872	2.530170	7	0	6	0		72.06096	1.309520	1	0	10	2	
35.99729	2.492860	109	-2	0	2		72.76406	1.298590	1	3	1	2	
36.31388	2.471850	12	-1	3	2		73.04416	1.294300	2	0	0	4	
36.64857	2.450040	68	0	2	2		73.73846	1.283820	2	3	7	1	
37.42864	2.400750	39	-2	4	1		74.21646	1.276730	19m	0	2	4	
37.95929	2.368400	2	-2	2	2		74.21646	1.276730	19m	0	8	3	
39.37455	2.286470	5	2	4	0		74.70065	1.269650	2	1	11	1	
39.61362	2.273220	1	0	6	1		75.01691	1.265080	6m	0	12	0	
40.69720	2.215160	121	1	5	1		75.01691	1.265080	6m	-1	5	4	
42.22390	2.138530	17	0	4	2		75.73711	1.254830	1	-1	9	3	
43.39286	2.083590	143	-2	4	2		76.09904	1.249760	12m	2	2	3	
43.51752	2.077910	58	2	2	1		76.09904	1.249760	12m	-5	1	2	
43.66418	2.071270	110m	-1	5	2		76.33524	1.246480	24m	4	6	0	
43.66418	2.071270	110m	-3	1	1		76.33524	1.246480	24m	-4	0	4	
44.25498	2.044980	43	1	1	2		76.65247	1.242110	15m	2	10	1	
44.63911	2.028270	6	1	7	0		76.65247	1.242110	15m	-4	6	3	
45.57625	1.988720	27	-1	7	1		76.93880	1.238200	24	4	2	1	
46.28035	1.960090	5	-2	6	1		77.10687	1.235920	10	-2	6	4	
46.48742	1.951840	27	-3	1	2		77.40668	1.231880	13	2	8	2	
46.99307	1.932010	2	-3	3	1		77.55008	1.229960	16m	0	12	1	
47.54214	1.910970	7	1	3	2		77.55008	1.229960	16m	-4	2	4	
47.93020	1.896400	135m	2	6	0		77.92178	1.225020	3m	0	4	4	
47.93020	1.896400	135m	3	1	0		77.92178	1.225020	3m	-5	1	1	
48.44209	1.877550	114	2	4	1		78.48299	1.217660	1	-5	3	2	
48.82994	1.863540	26	-1	1	3		79.71573	1.201900	32m	2	4	3	
49.66021	1.834310	3	-3	3	2		79.71573	1.201900	32m	-4	8	1	
50.39125	1.809400	116	0	6	2		79.83772	1.200370	21	-4	8	2	
50.76869	1.796830	52	-2	2	3		80.26683	1.195030	3	-5	3	1	
51.07441	1.786790	13	3	3	0		80.54846	1.191560	8	4	4	1	
51.23056	1.781710	23	0	8	1		81.15326	1.184200	1m	-3	9	3	
51.41472	1.775760	89	-2	6	2		81.15326	1.184200	1m	-4	4	4	
51.89498	1.760450	3	-1	3	3		81.85282	1.175840	6	-5	3	3	
53.15135	1.721760	2m	-1	7	2		82.05958	1.173400	6	-2	10	3	
53.15135	1.721760	2m	-3	5	1		82.23934	1.171290	1m	-1	7	4	
53.65365	1.706820	9	1	5	2		82.23934	1.171290	1m	-2	12	1	
54.48272	1.682790	20	0	2	3		83.13072	1.160980	1	1	1	4	
55.20356	1.662510	56	-2	4	3		83.45777	1.157260	12m	2	12	0	
55.58395	1.652030	2m	-3	1	3		83.45777	1.157260	12m	3	9	1	
55.58395	1.652030	2m	-3	5	2		83.90023	1.152280	17m	0	6	4	
55.91689	1.642980	29	2	6	1		83.90023	1.152280	17m	-3	11	1	
56.79938	1.619530	71m	2	0	2		84.71730	1.143240	7m	4	8	0	
56.79938	1.619530	71m	-2	8	1		84.71730	1.143240	7m	5	1	0	
57.64331	1.597810	4m	-1	5	3		85.02936	1.139840	34m	0	10	3	
57.64331	1.597810	4m	-1	9	1		85.02936	1.139840	34m	-4	8	3	
58.19860	1.583880	17m	2	2	2		85.32826	1.136610	12m	0	12	2	
58.19860	1.583880	17m	3	1	1		85.32826	1.136610	12m	-1	13	1	
58.27360	1.582020	19	2	8	0		85.47070	1.135080	9m	1	3	4	
58.39865	1.578930	11	-3	3	3		85.47070	1.135080	9m	-2	8	4	
58.72422	1.570950	9	0	4	3		85.67178	1.132930	1	2	6	3	
59.60340	1.549860	7	-4	0	2		85.94947	1.129980	1	-3	11	2	
60.43753	1.530450	20	0	8	2		86.12467	1.128130	5	-2	12	2	
60.92309	1.519410	14m	3	3	1		86.59851	1.123170	1	-5	5	3	
60.92309	1.519410	14m	-4	2	2		86.85706	1.120490	5	-2	2	5	
61.34669	1.509930	11	-2	8	2		87.08720	1.118120	9m	3	7	2	
61.56720	1.505050	5	-3	7	1		87.08720	1.118120	9m	-4	6	4	
61.97739	1.496070	2m	1	7	2		87.68414	1.112040	1	-1	11	3	
61.97739	1.496070	2m	1	9	1		87.91344	1.109730	1	-5	1	4	
62.11619	1.493060	7	-2	6	3		88.12812	1.107580	1	2	10	2	
62.27749	1.489580	2	2	4	2		89.04332	1.098550	1m	1	13	1	
62.47147	1.485420	8	1	1	3		89.04332	1.098550	1m	-1	1	5	
63.84375	1.456760	12m	0	10	1		89.32751	1.095790	2	-3	3	5	
63.84375	1.456760	12m	-3	7	2		90.39232	1.085630	22m	-2	4	5	
64.21108	1.449310	1	-1	9	2		90.39232	1.085630	22m	-4	10	1	
64.80628	1.437430	34	-4	4	1		90.97164	1.080220	7m	4	0	2	
65.06441	1.432350	20m	1	3	3		90.97164	1.080220	7m	-1	13	2	
65.06441	1.432350	20m	4	0	0		91.32391	1.076970	1	3	1	3	
65.40361	1.425740	13m	0	6	3		91.48431	1.075500	1	-1	3	5	
65.40361	1.425740	13m	2	8	1		91.86120	1.072070	1m	5	5	0	
65.88889	1.416410	23	-2	0	4		91.86120	1.072070	1m	-1	9	4	
66.35907	1.407510	2	4	2	0		92.15270	1.069440	12m	0	8	4	
66.74907	1.400230	19	-4	2	3		92.15270	1.069440	12m	4	2	2	
67.17572	1.392370	6	-2	2	4		92.98827	1.062010	13m	0	14	1	
67.59997	1.384660	7	-1	1	4		92.98827	1.062010	13m	-4	2	5	
68.80213	1.363370	44m	2	6	2		93.35835	1.058770	2	-3	9	4	
68.80213	1.363370	44m	-2	10	1		93.68682	1.055920	1m	3	3	3	
69.30799	1.354650	2	-3	1	4		93.68682	1.055920	1m	-5	7	3	
70.17086	1.340090	10m	4	4	0		93.93879	1.053750	5	2	8	3	
70.17086	1.340090	10m	-1	3	4		94.75811	1.046790	2	4	8	1	
70.55090	1.333800	14	-4	4	3		94.95010	1.045180	6m	-5	5	4	
70.77839	1.330070	9	-1	11	1		94.95010	1.045180	6m	-6	0	2	
70.96682	1.327000	4	-2	4	4		95.07223	1.044160	6	-2	12	3	
71.12479	1.324440	15m	-2	8	3		95.35747	1.041790	1m	4	10	0	
71.12479	1.324440	15m	-4	6	1		95.35747	1.041790	1m	-4	8	4	

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2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
95.71300	1.038860	9m	4	4	2		97.32017	1.025940	5m	0	2	5	
95.71300	1.038860	9m	-4	10	3		97.32017	1.025940	5m	1	7	4	
96.13578	1.035410	6m	-1	5	5		97.59280	1.023800	6m	-2	14	1	
96.13578	1.035410	6m	-6	2	2		97.59280	1.023800	6m	-6	2	3	
96.31237	1.033980	4	-2	6	5		98.04278	1.020300	1m	0	12	3	
96.54199	1.032130	9m	3	9	2		98.04278	1.020300	1m	-3	13	1	
96.54199	1.032130	9m	-4	4	5								

04-018-9908

Jun 1, 2020 6:25 PM (Steve Simner)

Status Primary **Quality Mark:** Blank **Environment:** Ambient **Temp:** 297.0 K **Phase:** Room temperature phase.
Chemical Formula: Ca2 Al (C O3)0.25 (O H)6.5 (H2 O)2 **Empirical Formula:** Al C0.25 Ca2 H10.5 O9.25
Weight %: Al10.04 C1.12 Ca29.83 H3.94 O55.07 **Atomic %:** Al4.35 C1.09 Ca8.70 H45.65 O40.22
Compound Name: Calcium Aluminum Carbonate Hydroxide Hydrate **Entry Date:** 09/01/2015

Radiation: CuKα1 (1.5406 Å) **d-Spacing:** Calculated **Intensity:** Calculated - Peak **I/Ic:** 4.81 **I/Ic - CW ND:** 0.34

Crystal System: Rhombohedral **SPGR:** R-3c (167)
Author's Unit Cell [a: 5.7757(1) Å **c:** 48.812(2) Å **Volume:** 1410.15 Å³ **Z:** 6.00 **MolVol:** 235.03 **c/a:** 8.451 **] Calculated Density:** 1.899 g/cm³ **Structural Density:** 1.82 g/cm³ **Color:** White
SS/FOM: F(30) = 745.1(0.0013, 32)

Space Group: R-3c (167) **Molecular Weight:** 268.71 g/mol
Crystal Data [a: 5.776 Å **b:** 5.776 Å **c:** 48.812 Å **α:** 90.00° **β:** 90.00° **γ:** 120.00°
XtlCell Vol: 1410.15 Å³ **XtlCell Z:** 6.00 **c/a:** 8.451 **a/b:** 1.000 **c/b:** 8.451 **]**
Reduced Cell [a: 5.776 Å **b:** 5.776 Å **c:** 16.609 Å **α:** 79.99° **β:** 79.99° **γ:** 60.00° **RedCell Vol:** 470.05 Å³ **]**

AC Space Group: R-3cH (167)
AC Unit Cell [a: 5.7757(1) Å **b:** 5.7757(1) Å **c:** 48.812(2) Å **α:** 90° **β:** 90° **γ:** 120° **]**

Space Group Symmetry Operators:

Seq	Operator	Seq	Operator	Seq	Operator	Seq	Operator
1	x,y,z	4	y,-x+y,-z	7	-y,-x,z+1/2	10	-x,-x+y,-z+1/2
2	-x,-y,-z	5	-x+y,-x,z	8	y,x,-z+1/2	11	-x+y,y,z+1/2
3	-y,x-y,z	6	x-y,x,-z	9	x,x-y,z+1/2	12	x-y,-y,-z+1/2

ADP Type: U

Atomic Coordinates:

Atom	Num	Wyckoff	Symmetry	x	y	z	SOF	Uiso	AET
Al	1	6b	-3.	0.0	0.0	0.0	1.0	0.0127	
Ca	2	12c	3.	0.66666	0.33333	0.67796	1.0	0.0214	
O	3	36f	1	0.3012	0.0484	0.5204	1.0	0.0224	
O	4	12c	3.	0.66666	0.33333	0.93875	1.0	0.063	
O	5	18e	.2	0.219	0.0	0.75	0.25	0.025	
O	6	36f	1	0.565	0.474	0.094	0.083	0.025	
C	7	6a	32	0.66666	0.33333	0.08333	0.25	0.025	

Crystal (Symmetry Allowed): Centrosymmetric

Subfiles: Inorganic **Pearson Symbol:** hR46.00 **Pearson Symbol w/o H:** hR25
LPF Prototype Structure [Formula Order]: Ca2 Al [C O3]0.25 [O H]6.5 [H2 O]2,hR126,167
LPF Prototype Structure [Alpha Order]: Al C0.25 Ca2 H10.5 O9.25,hR126,167 **ANX:** A2B6C12X55

Former PDF Numbers: 01-082-3613

References:

Type	DOI	Reference
Primary Reference		Calculated from LPF using POWD-12++.
Structure	10.1107/S010876811203042X	Runcevski T., Dinnebier R.E., Magdysyuk O.V., Pollmann H. "Crystal structures of calcium hemicarboaluminate and carbonated calcium hemicarboaluminate from synchrotron powder diffraction data". Acta Crystallogr., Sect. B: Struct. Sci. 2012, 68, 493-500.

Database Comments: ANX: A2B6C12X55. LPF Collection Code: 1128746. Polymorphism: Room temperature phase. Sample Preparation: Compound Preparation: heated. Temperature of Data Collection: 297 K. Test from external database: number of formula units Z is assumed to be misprinted as 6 instead of 3 ((Ca4Al2(OH)12)(OH(CO3)0.5·4H2O));in table 3;(agreement with refinement). Minor Warning: LPF comment on minor error in the publication exist. Minor warning from the LPF Editor exist. LPF Editor Comment: short interatomic distances for partly occupied sites. Significant Warning: Density calculated using chemical formula and reported structure differ by 4.160%. Unit Cell Data Source: Powder Diffraction.

d-Spacings (190) - Ca2 Al (C O3)0.25 (O H)6.5 (H2 O)2 - 04-018-9908 (Stick, Fixed Slit Intensity) - Cu Kα1 1.54056 Å

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
10.86619	**8.135330**	1000	0	0	6		30.93971	**2.887850**	116	1	1	0	
18.08864	4.900050	4	0	1	2		31.24562	2.860270	3	0	1	14	
19.16085	4.628200	28	1	0	4		31.43567	2.843410	8	1	1	3	
21.83162	**4.067670**	115	0	0	12		32.88324	2.721470	58	1	1	6	
22.97222	3.868220	104	0	1	8		34.40458	2.604530	26	1	0	16	
25.47616	3.493420	7	1	0	10		35.17798	2.549020	76	1	1	9	

04-018-9908 Jun 1, 2020 6:25 PM (Steve Simner)

2θ (°)	d (Å)	I	h	k	l	*	2θ (°)	d (Å)	I	h	k	l	*
36.07122	2.487920	2	2	0	2		84.25051	1.148380	1	1	1	39	
36.64873	2.450030	80	0	2	4		84.78683	1.142480	4m	2	0	38	
38.18766	2.354760	74	1	1	12		84.78683	1.142480	4m	3	2	4	
38.88537	2.314100	21	2	0	8		85.73185	1.132290	1	3	2	7	
40.49411	2.225800	20	0	2	10		85.86453	1.130880	1	1	3	25	
41.11872	2.193420	46	0	1	20		86.16171	1.127740	2m	2	2	27	
41.78525	2.159960	26	1	1	15		86.16171	1.127740	2m	2	3	8	
44.54815	2.032200	29m	0	0	24		86.68225	1.122300	1	1	2	35	
44.54815	2.032200	29m	2	0	14		87.19059	1.117060	2	3	2	10	
44.64190	2.028150	25	1	0	22		87.32170	1.115720	4	3	1	26	
45.86596	1.976830	59	1	1	18		87.59912	1.112900	2	0	4	20	
46.93898	1.934110	21	0	2	16		87.79018	1.110970	1	2	3	11	
48.12630	1.889130	1	2	1	1		89.23254	1.096710	1m	0	2	40	
48.24116	1.884900	1	1	2	2		89.23254	1.096710	1m	3	2	13	
48.69883	1.868250	19	2	1	4		89.77350	1.091500	7	4	1	0	
49.03966	1.856060	12	1	2	5		89.93068	1.090000	4m	2	3	14	
49.94049	1.824670	1	2	1	7		89.93068	1.090000	4m	4	0	22	
50.35195	1.810720	5	1	1	21		90.40507	1.085510	1	1	3	28	
50.49755	1.805840	3	1	2	8		90.80047	1.081810	6	1	4	6	
51.81655	1.762930	3	2	1	10		91.19455	1.078160	2	1	1	42	
51.98350	1.757660	15	0	1	26		91.64319	1.074050	2	3	2	16	
52.33401	1.746710	45	2	0	20		92.03162	1.070530	1m	3	1	29	
52.57498	1.739270	5	1	2	11		92.03162	1.070530	1m	4	1	9	
54.28051	1.688580	7	2	1	13		92.58566	1.065570	1	2	3	17	
55.03150	1.667300	47	3	0	0		92.93488	1.062480	5	1	2	38	
55.22410	1.661940	11m	1	1	24		93.88524	1.054210	6	1	4	12	
55.22410	1.661940	11m	1	2	14		94.14904	1.051950	1	3	0	36	
55.79905	1.646170	1	1	0	28		94.64407	1.047750	1	3	2	19	
56.27579	1.633350	42	3	0	6		95.48451	1.040740	1m	0	4	26	
56.51245	1.627070	4	0	0	30		95.48451	1.040740	1m	1	3	31	
57.28343	1.606990	6	2	1	16		95.76178	1.038460	7m	1	0	46	
58.39581	1.579000	2	1	2	17		95.76178	1.038460	7m	2	3	20	
59.90696	1.542730	15	3	0	12		96.40406	1.033240	1	2	2	33	
60.35479	1.532350	1	1	1	27		97.27322	1.026310	4	3	1	32	
60.77841	1.522680	2	2	1	19		98.17747	1.019260	1	3	2	22	
61.73186	1.501430	15	2	0	26		98.58957	1.016100	1	4	0	28	
62.04556	1.494590	22	1	2	20		98.67619	1.015440	1	1	1	45	
63.73223	1.459040	8	0	1	32		98.85526	1.014080	1	2	0	44	
64.47917	1.443930	12	2	2	0		99.05636	1.012560	2	1	4	18	
64.72694	1.439000	3m	2	1	22		99.47637	1.009410	1	2	3	23	
64.72694	1.439000	3m	2	2	3		99.74402	1.007420	1	1	2	41	
65.17740	1.430140	1	0	2	28		100.82166	0.999541	1	5	0	2	
65.61228	1.421710	12m	0	3	18		101.17046	0.997036	1m	0	5	4	
65.61228	1.421710	12m	2	2	6		101.17046	0.997036	1m	1	3	34	
65.82918	1.417550	1	1	1	30		102.39443	0.988416	1m	1	4	21	
66.13894	1.411660	1	1	2	23		102.39443	0.988416	1m	2	2	36	
67.01490	1.395320	1	2	2	9		102.57013	0.987200	1	5	0	8	
67.48605	1.386720	2m	1	3	1		103.10337	0.983542	1	3	1	35	
67.48605	1.386720	2m	3	1	2		103.76024	0.979102	4	2	3	26	
67.86312	1.379930	1	1	0	34		104.09905	0.976840	1	0	2	46	
67.94872	1.378400	1	1	3	4		104.64971	0.973204	1	2	1	43	
68.22555	1.373480	2	3	1	5		105.59823	0.967056	1	0	4	32	
68.95386	1.360740	8m	1	3	7		106.29719	0.962617	2	3	3	0	
68.95386	1.360740	8m	2	2	12		106.46192	0.961582	2m	4	1	24	
69.23556	1.355890	1	0	0	36		106.46192	0.961582	2m	5	0	14	
69.41928	1.352750	2	3	1	8		106.84600	0.959185	1m	1	1	48	
70.65194	1.332140	17	1	2	26		106.84600	0.959185	1m	3	2	28	
71.14707	1.324080	1	3	1	11		107.01120	0.958161	1	0	1	50	
71.41121	1.319830	1	2	2	15		107.23198	0.956799	1	1	2	44	
71.61904	1.316510	2	1	1	33		107.37054	0.955948	2m	1	3	37	
72.52566	1.302270	4m	1	3	13		107.37054	0.955948	2m	3	3	6	
72.52566	1.302270	4m	2	0	32		107.78431	0.953424	1	3	0	42	
73.39414	1.288990	1m	3	0	24		108.25582	0.950579	1	0	5	16	
73.39414	1.288990	1m	3	1	14		108.72108	0.947804	1	3	3	9	
73.88807	1.281590	1	2	1	28		109.18066	0.945094	1m	2	4	1	
74.36754	1.274510	5	2	2	18		109.18066	0.945094	1m	4	2	2	
75.17301	1.262840	2	1	3	16		109.61999	0.942532	3m	2	4	4	
75.57418	1.257130	1	1	2	29		109.61999	0.942532	3m	3	1	38	
76.16513	1.248840	1m	0	4	2		109.90739	0.940871	1	4	2	5	
76.16513	1.248840	1m	3	1	17		110.63059	0.936743	2m	2	4	7	
76.50329	1.244160	6m	0	1	38		110.63059	0.936743	2m	3	3	12	
76.50329	1.244160	6m	4	0	4		111.04599	0.934405	1m	4	1	27	
77.74672	1.227340	1m	1	1	36		111.04599	0.934405	1m	4	2	8	
77.74672	1.227340	1m	2	2	21		112.32154	0.927374	1m	2	4	10	
77.92253	1.225010	2	0	4	8		112.32154	0.927374	1m	3	2	31	
78.25162	1.220680	1	1	3	19		112.64259	0.925639	3m	2	1	46	
78.97062	1.211360	1	4	0	10		112.64259	0.925639	3m	5	0	20	
79.08459	1.209900	1	2	1	31		113.21359	0.922587	1m	1	0	52	
79.38613	1.206060	4	3	1	20		113.21359	0.922587	1m	3	3	15	
80.91044	1.187140	8	1	2	32		114.27908	0.917006	2m	2	3	32	
81.81650	1.176270	2m	0	4	14		114.27908	0.917006	2m	2	4	13	
81.81650	1.176270	2m	1	3	22		115.26568	0.911968	1m	0	5	22	
82.82653	1.164470	1	3	0	30		115.26568	0.911968	1m	4	2	14	
83.11147	1.161200	1m	0	0	42		115.77582	0.909411	1	2	0	50	
83.11147	1.161200	1m	3	1	23		116.23094	0.907157	1	3	3	18	
83.47632	1.157050	1	4	0	16		116.59792	0.905358	1	2	2	42	

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2θ (°)	d (Å)	I	h	k	l	*
117.10046	0.902921	1m	2	4	16	
117.10046	0.902921	1m	3	1	41	
118.18600	0.897760	1m	1	5	2	
118.18600	0.897760	1m	4	2	17	
118.55958	0.896016	1m	0	4	38	
118.55958	0.896016	1m	5	1	4	