

# **CHARACTERIZATION OF REFERENCE MATERIALS AND RELATED MATERIALS FOR THE CEMENTITIOUS BARRIERS PARTNERSHIP**

## **Main Report**

**Cementitious Barriers Partnership**

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RELATED MATERIALS FOR THE CEMENTITIOUS  
BARRIERS PARTNERSHIP**

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## FOREWORD

The Cementitious Barriers Partnership (CBP) Project is a multi-disciplinary, multi-institutional collaboration supported by the United States Department of Energy (US DOE) Office of Waste Processing. The objective of the CBP project is to develop a set of tools to improve understanding and prediction of the long-term structural, hydraulic, and chemical performance of cementitious barriers used in nuclear applications.

A multi-disciplinary partnership of federal, academic, private sector, and international expertise has been formed to accomplish the project objective. In addition to the US DOE, the CBP partners are the Savannah River National Laboratory (SRNL), Vanderbilt University (VU) / Consortium for Risk Evaluation with Stakeholder Participation (CRESP), Energy Research Center of the Netherlands (ECN), and SIMCO Technologies, Inc. The Nuclear Regulatory Commission (NRC) is providing support under a Memorandum of Understanding. The National Institute of Standards and Technology (NIST) is providing research under an Interagency Agreement. Neither the NRC nor NIST are signatories to the Cooperative Research and Development Agreement (CRADA).

The periods of cementitious performance being evaluated are up to or longer than 100 years for operating facilities and longer than 1000 years for waste management. The set of simulation tools and data developed under this project will be used to evaluate and predict the behavior of cementitious barriers used in near-surface engineered waste disposal systems, e.g., waste forms, containment structures, entombments, and environmental remediation, including decontamination and decommissioning analysis of structural concrete components of nuclear facilities (spent-fuel pools, dry spent-fuel storage units, and recycling facilities such as fuel fabrication, separations processes). Simulation parameters will be obtained from prior literature and will be experimentally measured under this project, as necessary, to demonstrate application of the simulation tools for three prototype applications (waste form in concrete vault, high-level waste tank grouting, and spent-fuel pool). Test methods and data needs to support use of the simulation tools for future applications will be defined.

The CBP project is a five-year effort focused on reducing the uncertainties of current methodologies for assessing cementitious barrier performance and increasing the consistency and transparency of the assessment process. The results of this project will enable improved risk-informed, performance-based decision-making and support several of the strategic initiatives in the DOE Office of Environmental Management Engineering & Technology Roadmap. Those strategic initiatives include 1) enhanced tank closure processes; 2) enhanced stabilization technologies; 3) advanced predictive capabilities; 4) enhanced remediation methods; 5) adapted technologies for site-specific and complex-wide D&D applications; 6) improved SNF storage, stabilization and disposal preparation; 7) enhanced storage, monitoring and stabilization systems; and 8) enhanced long-term performance evaluation and monitoring.

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and

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## ABSTRACT

The Cementitious Barriers Partnership Project (CBP) is a multi-disciplinary, multi-institution cross cutting collaborative effort supported by the US Department of Energy (DOE) to develop a reasonable and credible set of tools to improve understanding and prediction of the structural, hydraulic and chemical performance of cementitious barriers used in nuclear applications. The period of performance is >100 years for operating facilities and > 1000 years for waste management. In PTS-02 Task 6 the CBP has defined a set of reference cases to provide the following functions: (i) a common set of system configurations to illustrate the methods and tools developed by the CBP, (ii) a common basis for evaluating methodology for uncertainty characterization, (iii) a common set of cases to develop a complete set of parameter and changes in parameters as a function of time and changing conditions, and (iv) a basis for experiments and model validation, and (v) a basis for improving conceptual models and reducing model uncertainties. These reference cases include the following two reference disposal units and a reference storage unit: (i) a cementitious low activity waste form in a reinforced concrete disposal vault, (ii) a concrete vault containing a steel high-level waste tank filled with grout (closed high-level waste tank), and (iii) a spent nuclear fuel basin during operation. Each case provides a different set of desired performance characteristics and interfaces between materials and with the environment.

This report evaluates the chemical composition and leaching behaviors of four cementitious reference materials that have been selected for study: a ternary blend (type I/II cement + blast furnace slag + Class F fly ash binder) flowable, stable (zero-bleed) infill/backfill grout (material code BGM), a ternary blend (type I/II cement + blast furnace slag + Class F fly ash binder) structural vault concrete-analogous mortar (material code SVC), a binary blend (type I/II cement + blast furnace slag) vault concrete (material code VCO), and a quaternary blend (type V sulfate resistant cement + blast furnace slag + Class F fly ash binder + silica fume binder) vault concrete (material code VCT). Additional characterization was performed on four component materials of the cementitious materials: ground granulated blast furnace slag (material code BFS), coal combustion fly ash (material code FAF), quartz concrete sand (material code QS), and type I/II Portland cement powder (material code OTC).

The test methods employed aim at determining constituent solubility as functions of pH and of liquid-to-solid ratio for both granular and monolithic materials using EPA draft Methods 1313 and 1314, respectively. In addition, constituent leaching rate as a function of time was measured for monolithic materials using EPA draft Method 1315. Data obtained from these tests will support model calibration and validation for estimating constituent leaching as a part of system performance assessment.

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## LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BFS	blast furnace slag
COPC	constituent of potential concern
BGM	backfill grout mortar
DI water	deionized water
DIC	dissolved inorganic carbon
DOC	dissolved organic carbon
EPA	U.S. Environmental Protection Agency
FAF	class F fly ash
ICP-MS	inductively coupled plasma mass spectroscopy
ICP-OES	inductively coupled plasma optical emission spectroscopy
LS	liquid-to-solid ratio
MDL	method detection limit
ML	method limit (i.e. practical quantification limit)
OTC	type I/II Portland cement
QS	quartz sand
SRS	Savannah River Site
SVC	structural vault concrete-analogous mortar
VCO	Vaults 1 and 4 concrete
VCT	Vault 2 concrete

## 1.0 INTRODUCTION

The Cementitious Barriers Partnership Project (CBP) is a multi-disciplinary, multi-institution cross cutting collaborative effort supported by the US Department of Energy (DOE) to develop a reasonable and credible set of tools to improve understanding and prediction of the structural, hydraulic and chemical performance of cementitious barriers used in nuclear applications. The period of performance is >100 years for operating facilities and > 1000 years for waste management. The CBP has defined a set of reference cases to provide the following functions: (i) a common set of system configurations to illustrate the methods and tools developed by the CBP, (ii) a common basis for evaluating methodology for uncertainty characterization, (iii) a common set of cases to develop a complete set of parameter and changes in parameters as a function of time and changing conditions, and (iv) a basis for experiments and model validation, and (v) a basis for improving conceptual models and reducing model uncertainties. These reference cases include the following two reference disposal units and a reference storage unit: (i) a cementitious low activity waste form in a reinforced concrete disposal vault, (ii) a concrete vault containing a steel high-level waste tank filled with grout (closed high-level waste tank), and (iii) a spent nuclear fuel basin during operation. Each case provides a different set of desired performance characteristics and interfaces between materials and with the environment.

This report evaluates the chemical composition and leaching behaviors of four cementitious reference materials that have been selected for study: a ternary blend (type I/II cement + blast furnace slag + Class F fly ash binder) flowable, stable (zero-bleed) infill/backfill grout (material code BGM), a ternary blend (type I/II cement + blast furnace slag + Class F fly ash binder) structural vault concrete-analogous mortar (material code SVC), a binary blend (type I/II cement + blast furnace slag) vault concrete (material code VCO), and a quaternary blend (type V sulfate resistant cement + blast furnace slag + Class F fly ash binder + silica fume binder) vault concrete (material code VCT). Additional characterization was performed on four component materials of the cementitious materials: ground granulated blast furnace slag (material code BFS), coal combustion fly ash (material code FAF), quartz concrete sand (material code QS), and type I/II Portland cement powder (material code OTC).

The test methods employed aim at determining constituent solubility as functions of pH and of liquid-to-solid ratio for both granular and monolithic materials using EPA draft Methods 1313 and 1314, respectively. In addition, constituent leaching rate as a function of time was measured for monolithic materials using EPA draft Method 1315. Data obtained from these tests will support model calibration and validation for estimating constituent leaching as a part of system performance assessment.

## 2.0 OBJECTIVES AND EXPERIMENTAL OVERVIEW

In support of geochemical speciation and mass transport modeling, a number of experimental methods have been employed to assess the leaching behaviors of the set of reference materials chosen for study. A summary of the test methods employed and the data extracted from them is given below:

- EPA Draft Method 1313
  - Method: parallel batch extraction from granular materials at 11 fixed values of pH
  - Result: pH titration, solubility, and constituent release as a function of pH
  - Replication: 2 test replicates per material
- EPA Draft Method 1314
  - Method: up-flow of leachant through granular material in a column
  - Result: liquid-solid partitioning as a function of liquid-solid ratio
  - Replication: 2 test replicates per material; 3 replicates for FAF
- EPA Draft Method 1315
  - Method: extraction from a cementitious monolith with periodic renewal of leachant
  - Result: mass transfer rates and estimation of observed diffusivity
  - Replication: 2 test replicates per material for materials BGM, SVC, VCO and VCT
- X-ray Fluorescence (XRF) for total elemental content
  - Method: analysis of KBr pellets (see method description)
  - Result: total solid-phase elemental content for major species
  - Replication: 2 analysis replicates per material
- Acid Digestion and Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
  - Method: microwave-assisted hydrofluoric acid digestion of powdered specimens according to EPA SW-846 Method 3052; analysis by EPA SW-846 Method 6010
  - Result: total elemental content for major and minor species
  - Replication: 2 analysis replicates per material
- ASTM C 642 Standard Test Method
  - Method: oven-drying, saturation, and measurement of apparent mass of submerged monolith specimens
  - Result: specimen density and porosity
  - Replication: 10 replicates each for BGM and SVC

### 3.0 MATERIALS AND METHODS

#### 3.1 MATERIALS

Chemical and leaching and analysis were executed on both hardened cementitious specimens and on their component binders and aggregates individually. The primary binders investigated herein are type I/II cement, ground granulated blast furnace slag, and coal combustion fly ash.

##### 3.1.1 Type I/II Portland Cement (ASTM C 150)

The ordinary Portland cement (OTC) used in this study was acquired from Holcim Inc. (Holly Hill Plant, SC 29059) and conforms to specifications set out in ASTM C 150 for Type I/II cement which is suitable for general applications as well as providing moderate sulfate resistance and moderate heat of hydration [1]. Test data on ASTM standard requirements reported by Holcim are shown in Table 1. Data represent average test values for the testing period from June 21-22, 2009.

Table 1: Test results for ASTM C 150 specifications for Type I/II cement.

Test Data on ASTM Standard Requirements					
Chemical			Physical		
Item	Limit <sup>A</sup>	Result	Item	Limit <sup>A</sup>	Result
SiO <sub>2</sub> (%)	-	20.3	Air Content (%)	12 max	6
Al <sub>2</sub> O <sub>3</sub> (%)	6.0 max	5.0	Blaine Fineness (m <sup>2</sup> /kg)	260 min 430 max	393
Fe <sub>2</sub> O <sub>3</sub> (%)	6.0 max	3.8	Average <sup>B</sup> Blaine Fineness (m <sup>2</sup> /kg)	280 min 420 max	398.20
CaO (%)	-	64.3			
MgO (%)	6.0 max	1.2	Autoclave Expansion (%) (C151)	0.80 max	-0.01
SO <sub>3</sub> (%)	3.0 max	3.0	Compressive Strength MPa (psi):		
Loss on Ignition (%)	3.0 max	1.8			
Insoluble Residue (%)	0.75 max	0.14	3 Day	12.0 (1740) min	29.2 (4240)
CO <sub>2</sub> (%)	-	0.7	7 Day	19.0 (2760) min	36.2 (5250)
Limestone (%)	5.0 max	1.8			
CaCO <sub>3</sub> in Limestone (%)	70 min	93	Initial Vicat (minutes)	45-275	95
Potential Compounds:					
C <sub>3</sub> S (%)	-	56	Mortar Bar Expansion (%) (C1038)	-	0.010
C <sub>2</sub> S (%)	-	16			
C <sub>3</sub> A (%)	8 max	7	Heat of Hydration: 7 days, kJ/kg (cal/g)	-	338 (81)
C <sub>4</sub> AF (%)	-	12			
C <sub>3</sub> S + 4.75C <sub>3</sub> A (%)	100 max	89.3			

Test Data on ASTM Optional Requirements					
Chemical			Physical		
Item	Limit <sup>A</sup>	Result	Item	Limit <sup>A</sup>	Result
Equivalent Alkalies (%)	0.60 max	0.48			
Notes					
A – Dashes in the limits columns means Not Applicable B – Average of last five consecutive samples. This data may have been reported on previous mill certificates. It is typical of the cement being currently shipped which was produced in June of 2009.					

### 3.1.2 Grade 100 Ground Granulated Blast Furnace Slag (ASTM C 989)

Ground granulated blast furnace slag (BFS) was obtained from Holcim Inc. (Birmingham Plant, AL 35221). Test data required by ASTM C 989 [2] are shown in Table 2 and are averages over the test period from July 1-31, 2009.

Table 2: Test results for ASTM C 989 specifications for blast furnace slag as a concrete admixture.

Test Data on ASTM Standard Requirements					
Chemical (C 989, Table 2)			Physical (C 989, Table 1)		
Item	Limit	Result	Item	Limit	Result
			+45 $\mu$ m (No. 325) Sieve (%)	20 max	0.57
			Blaine Fineness ( $m^2/kg$ )	-	607
Sulfide S (%)	2.5 max	0.9	Air Content (%)	12 max	3.62
Sulfate Ion – $SO_3$ (%)	4.0 max	2.01	Slag Activity Index (%)		
			Avg 7 Day Index	75 min	83
			Avg 28 Day Index	95 min	126
			Compressive Strength Slag-Ref (psi)		
			7 Day		25 (3620)
			28 Day		43 (6240)
Test Data on Reference Cement					
Chemical			Physical		
Item	Limit	Result	Item	Limit	Result
Total Alkalies as $Na_2O$ (%)	0.60–0.90	0.82	Blaine Fineness ( $m^2/kg$ )	-	384
$C_3S$	-	53	Compressive Strength MPa (psi)		
$C_2S$	-	9	7 Day	-	31 (4500)
$C_3A$	-	-	28 Day	35 (5000) min	39 (5660)
$C_4AF$	-	-			
Notes					
Specific Gravity: 2.89					

### 3.1.3 Type F Coal Combustion Fly Ash

Coal combustion fly ash (FAF) was obtained from McKeekin Station (Thermally Beneficiated). Testing by TEC Services of Lawrenceville, GA indicated that the fly ash conformed to the standards for F classification fly ash set out in ASTM C 618 [3] as shown in Table 3.

Table 3: Test results for ASTM C 618 specifications for Class F fly ash as a concrete admixture.

Test Data on ASTM Standard Requirements					
Chemical*			Physical		
Item	Limit	Result	Item	Limit	Result
SiO <sub>2</sub> (%)	-	54.57	+45 µm (No. 325) Sieve (%)	34 max	16.3
AlO (%)	-	27.97	Strength Activity Index with Portland Cement (%)		
FeO (%)	-	8.81	7 Days	75 min	81
SO <sub>2</sub> + FeO + AlO (%)	70 min	91.35	28 Days	75 min	85
CaO (%)	-	1.33	Water Requirements (%)	105 max	98
MgO (%)	-	1.20	Autoclave Expansion (%)	± 0.8 max	-0.08
SO <sub>3</sub> (%)	5 max	0.05			
Loss on Ignition (%)	6 max	0.76			
Moisture Content (%)	3 max	0.05			
Available Alkalies as Na <sub>2</sub> O (%)	-	0.68			
Uniformity Requirements					
Item	Limit	Result	Item	Limit	Result
Specific Gravity (%):	±5 max	-2.1%			
% Retained on #325 Sieve	±5 max	2.1%			
Notes					
*Chemical Analysis performed by Wyoming Analytical					

### 3.1.4 Quartz Sand (ASTM C 33)

Concrete quartz sand (QS) provided by U.S. Aggregates was analyzed for fineness at Savannah River Site on June 10, 2009 according to the ASTM C 136 sieve analysis procedure [4]. This sand was found to conform to the ASTM C 33-02a standard specification for fine aggregates used in concretes [5]. Results for the sieve analysis are shown in Table 4.



Table 4: Test results for ASTM C 33 specifications for fine aggregate.

Test Data on ASTM Standard Requirements				
Sieve Size	Cumulative Weight Retained (g)	Cumulative % Retained	% Passing	Acceptance Criteria (%)
3/8"	0	0	100	100
#4	12.5	2	98	95-100
#8	31.8	5	95	80-100
#16	108.4	19	81	50-85
#30	287.2	49	51	25-69
#40	395.5	N/A	N/A	N/A
#50	480.3	83	17	5-30
#100	563.4	96.8	3.2	0-10
Pan Weight: 314.9 g				
Original Oven Dry Weight: 314.9 g				
Fineness Modulus: Sum of Cumulative % Retained/100 = 2.65				
Fineness Modulus Acceptance Criteria: greater than 2.3, less than 3.1				

### 3.1.5 Backfill/Infill Grout Mortar (BGM)

A ternary blend cement mortar analogous to mortars used in backfill and infill applications was mixed on August 26, 2009 at Savannah River Site (SRS). The ternary blend of cementitious materials consisted of Type I/II Portland cement (OTC), grade 100 blast furnace slag (BFS), class F fly ash (FAF), and mix water resulting in a water-to-binder ratio of 0.38. The mortar mixture included a volume replacement of 54% quartz sand (QS). Specific details of the mixture design are given in Table 5.

Table 5: Mixture proportions for backfill/infill grout mortar (BGM).

Ingredient	Mass %
Type I/II Cement (ASTM C 150)	5.88
Grade 100 Blast Furnace Slag (ASTM C 989)	13.48
Type F Fly Ash (ASTM C 618)	6.62
Silica Fume	-
Water (maximum)	11.76
Quartz Sand (ASTM C 33)	62.25
No. 67 Granite Aggregate (ASTM C 33)	-

### 3.1.6 Structural Vault Concrete-Analogous Mortar (SVC)

A ternary blend cement mortar analogous to a structural vault concrete was mixed on August 26, 2009 at Savannah River Site. Specifics of the mixture design are given in Table 6.

Table 6: Mixture proportions for structural vault concrete-analogous mortar (SVC).

<b>Ingredient</b>	<b>Mass %</b>
Type I/II Cement (ASTM C 150)	5.40
Grade 100 Blast Furnace Slag (ASTM C 989)	8.03
Type F Fly Ash (ASTM C 618)	16.93
Silica Fume	-
Water (maximum)	14.62
Quartz Sand (ASTM C 33)	55.02
No. 67 Granite Aggregate (ASTM C 33)	-

### 3.1.7 Vaults 1 and 4 Concrete (VCO)

A ternary blend cement mortar analogous to a structural vault concrete was prepared at SIMCO Technologies Inc. Specifics of the mixture design are given in Table 7.

Table 7: Mixture proportions for Vaults 1 and 4 concrete (VCO).

<b>Ingredient</b>	<b>Mass %</b>
Type I/II Cement (ASTM C 150)	10.75
Grade 100 Blast Furnace Slag (ASTM C 989)	7.12
Type F Fly Ash (ASTM C 618)	-
Silica Fume	-
Water (maximum)	6.83
Quartz Sand (ASTM C 33)	29.12
No. 67 Granite Aggregate (ASTM C 33)	46.19

### 3.1.8 Vault 2 Concrete (VCT)

A ternary blend cement mortar analogous to a structural vault concrete was prepared at SIMCO Technologies Inc. Specifics of the mixture design are given in Table 8.

Table 8: Mixture proportions for Vault 2 concrete (VCT).

Ingredient	Mass %
Type I/II Cement (ASTM C 150)	5.46
Grade 100 Blast Furnace Slag (ASTM C 989)	7.31
Type F Fly Ash (ASTM C 618)	4.29
Silica Fume	1.22
Water (maximum)	6.86
Quartz Sand (ASTM C 33)	24.73
No. 67 Granite Aggregate (ASTM C 33)	50.14

### 3.1.9 Cementitious Material Summary

A comparison of the relative mass proportions of component materials used in BGM, SVC, VCO, and VCT is presented in Figure 1. Relative proportions of the binder materials for each material are shown in Figure 2.

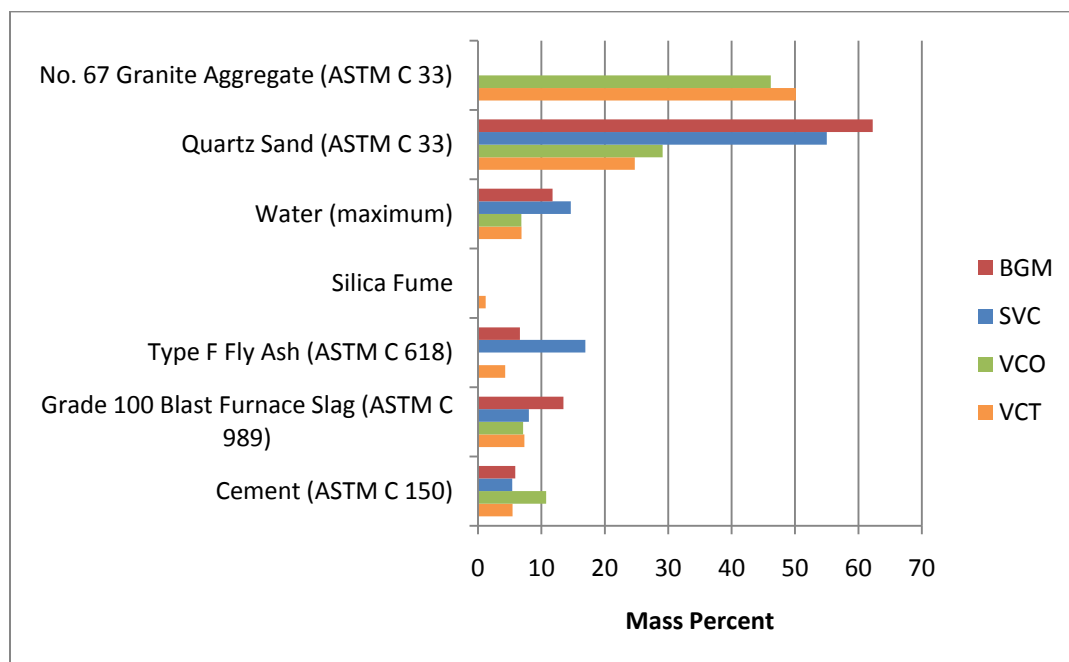


Figure 1: Comparison of total mixture proportions for the four cast cementitious materials: backfill/infill grout mortar (BGM), structural vault concrete-analogous mortar (SVC), Vaults 1 and 4 concrete (VCO), and Vault 2 concrete (VCT).

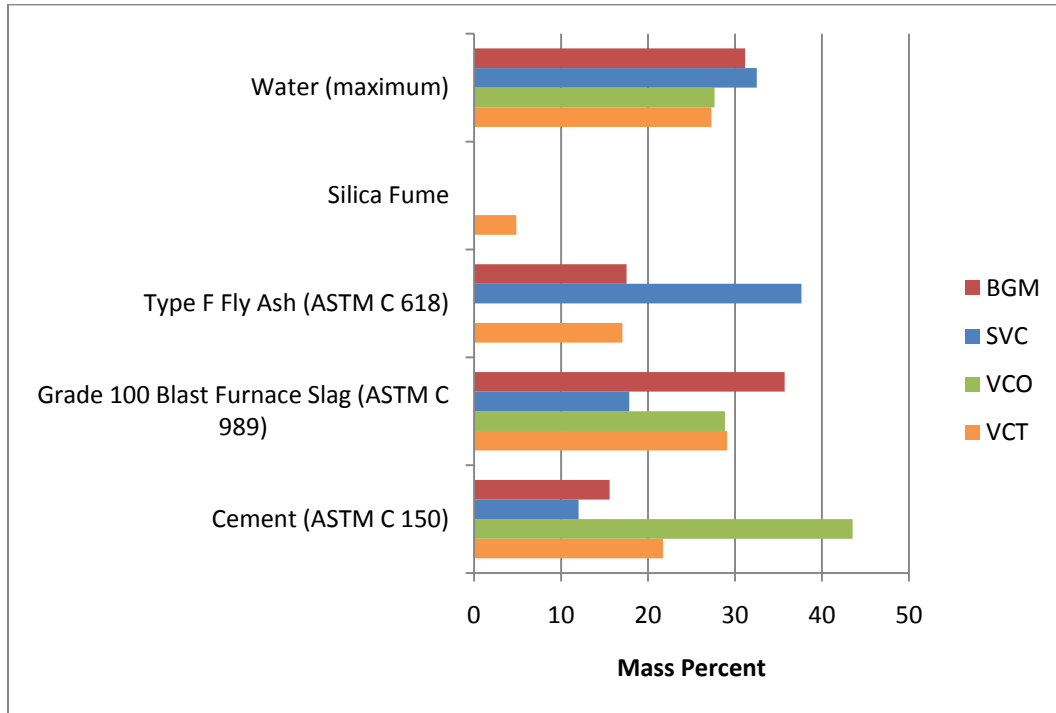


Figure 2: Comparison of binder mixture proportions for the four cast cementitious materials: backfill/infill grout mortar (BGM), structural vault concrete-analogous mortar (SVC), Vaults 1 and 4 concrete (VCO), and Vault 2 concrete (VCT).

## **3.2 LEACHING ASSESSMENT METHODS**

### **3.2.1 Alkalinity, Solubility and Release as a Function of pH (Method 1313)**

Alkalinity, solubility, and release as a function of pH were determined at Vanderbilt University according to EPA Draft Method 1313 [6]. This protocol consists of 11 parallel extractions of particle size-reduced material, at different pH values ranging from pH 2-13 and LS ratio of 10 mL extractant/g dry sample. In this method, particle size reduction is used when necessary to prepare large-grained samples for extraction so that the approach toward liquid-solid equilibrium concentrations of the COPCs is enhanced. For the samples evaluated in this study, particle size reduction to a maximum diameter of 2 mm was required for the hardened cementitious materials. Each extraction condition was carried out with replication as appropriate using 40 g of material for each material evaluated. In addition, three method blanks were included, consisting of the DI water, nitric acid, and potassium hydroxide used for extractions. Typical particle size of the tested materials was less than 300  $\mu\text{m}$  using standard sieves according to ASTM E-11 [7]. An acid or base addition schedule is formulated based on initial screening for eleven eluates with final solution pH values between 3 and 12, through addition of aliquots of nitric acid or potassium hydroxide as needed. The exact schedule is adjusted based on the nature of the material; however, the range of pH values includes the natural pH of the matrix that may extend the pH domain (e.g., for very alkaline or acidic materials). The final LS ratio is 10 mL extractant/g dry sample which includes DI water, the added acid or base, and the amount of moisture that is inherent to the waste matrix as determined by moisture content analysis. The eleven extractions were tumbled in an end-over-end fashion at  $29 \pm 2$  rpm for 24 hours followed by filtration separation of the solid phase from the eluate using a 0.45  $\mu\text{m}$  polypropylene filter. Each eluate then was analyzed for constituents of interest. The acid and base neutralization behavior of the materials is evaluated by plotting the pH of each eluate as a function of equivalents of acid or base added per gram of dry solid. Concentration of constituents of interest for each eluate is plotted as a function of eluate final pH to provide liquid-solid partitioning equilibrium as a function of pH.

### **3.2.2 Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio in Solid Materials Using an Up-Flow Percolation Column (Method 1314)**

Liquid-solid partitioning as a function of liquid-solid ratio (L/S, mL eluant/g dry weight of material tested) was performed at Vanderbilt University according to EPA Draft Method 1314 [6], wherein eluant is introduced into a column of moderately-packed granular material in an up-flow pumping mode, with eluate collection performed as a function of the cumulative LS ratio. For the samples evaluated in this study, particle size reduction to a maximum diameter of 2 mm was required for the hardened cementitious materials. Up-flow pumping is used to minimize air entrainment and flow channeling. The default eluant for all materials was reagent water. The flow rate is maintained between 0.5-1.0 L/S per day to increase the likelihood of local equilibrium between the solid and liquid phases, with a residence time longer than 1 day. Eluate

samples are chemically analyzed for inorganic analytes, total organic carbon, electrical conductivity and pH. For the purposes of chemical speciation modeling, the entire eluant volume up to 10 mL/g dry sample (g-dry) is collected in nine specific aliquots of varying volume.

### **3.2.3 Mass Transfer Rates of Constituents in Monolithic Materials (Method 1315)**

Determination of mass transfer rates of constituents in monolithic materials was performed at Vanderbilt University according to EPA Draft Method 1315 [6]. This protocol consists of tank leaching of continuously water-saturated monolithic material with periodic renewal of the leaching solution. The vessel and sample dimensions are chosen so that the sample is fully immersed in the leaching solution. Cylinders of 10.2-cm diameter and 5.1-cm minimum height were contacted with DI water using a liquid-to-surface area ratio of 10 mL of DI water for every  $\text{cm}^2$  of exposed solid surface area. Leaching solution was exchanged with fresh DI water at predetermined cumulative times of 2 hours, 1, 2, 7, 14, 28, 42, 49, 63, 77, 91, 105, 126, 147, 168, 189, 210, and 231 days. At the completion of each contact period, the mass of the monolithic sample after being freely drained was recorded to monitor the amount of leachant absorbed into the solid matrix. The solution pH and conductivity for each leachate was measured for each time interval. Leachate samples were prepared for chemical analysis by vacuum filtration through a 0.45-mm pore size polypropylene filtration membrane and preservation as appropriate. Leachate concentrations are plotted as a function of time along with the analytical detection limit and the equilibrium concentration determined from Method 1313 at the extract pH for quality control to ensure that release was not limited by saturation of the leachate. Cumulative release and flux as a function of time for each constituent of interest are plotted and used to estimate mass transfer parameters (i.e., observed diffusivity).

### **3.3 SOLID PHASE COMPOSITION AND POROSITY**

#### **3.3.1 X-ray Fluorescence Analysis for Total Composition**

X-ray fluorescence analysis for total content was performed on powdered samples by ARCADIS U.S., Inc. (Research Triangle Park, NC) in August 2010.

XRF analysis was performed on each material to provide additional information on total elemental composition. For each material two pellets were prepared as follows. 3000 mg of material was weighed and mixed with 1.5 mL (100 mg dry solids) of liquid binder to give a 32 mm diameter pellet weighing 3150 mg with a material-to-diluent ratio of 0.05. For high carbon content samples 3.0 ml (100 mg dry solids) of liquid binder was used to give a 32 mm diameter pellet weighing 3300 mg with a material-to-diluent ratio of 0.1. XRF intensities were collected on each side of each pellet using Philips SuperQ data collection software and evaluated using Omega Data System's UniQuant 4 XRF "standardless" data analysis software. The UQ/fly ash calibration was used to analyze the samples. The pellets were evaluated as oxides. Known fly ash Standard Reference Materials (SRMs) were also run to assess the accuracy of the analysis. This information is useful in supplementing ICP results.

X-Ray Fluorescence Spectrometry was used in the Research Triangle Park laboratories of EPA-NRMRL to analyze these samples. A Philips model PW 2404 wavelength dispersive instrument, equipped with a PW 2540 VRC sample changer, was used for these analyses. The manufacturer's software suite, "SuperQ", was used to operate the instrument, collect the data, and perform quantification.

The instrument was calibrated at the time of installation of the software plus a new X-ray tube using a manufacturer-supplied set of calibration standards. On a monthly basis, manufacturer-supplied drift correction standards were used to create an updated drift correction factor for each potential analytical line. On a monthly basis, a dedicated suite of QC samples were analyzed before and after the drift correction procedure. This data was used to update and maintain the instrument's QC charts.

The software suite's "Measure and Analyze" program was used to collect and manage the sample data. Quantification was performed post-data collection using the program "IQ+". IQ+ is a "first principles" quantification program that includes complex calculations to account for a wide variety of sample-specific parameters. For this reason, sample-specific calibrations were not necessary. This program calculates both peak heights and baseline values. The difference is then used, after adjustment by drift correction factors, for elemental quantification versus the calibration data. Inter-element effects are possible and the software includes a library of such parameters. Data from secondary lines may be used for quantification where inter-element effects are significant or the primary peak is overloading the data acquisition system. Where the difference between the calculated peak height and baseline is of low quality, the program will not identify a peak and will not report results. IQ<sup>+</sup> permits the inclusion of data from other sources

by manual entry. Carbon was an example of this for these samples. Entry of other source data for elements indeterminable by XRF improves the mass balance.

Table 9 presents detection limit data in two forms. The two forms are not mutually exclusive. The “reporting limit” is built into the software and reflects the manufacturer’s willingness to report low-level data. Data listed in the “detection limit” column were based upon the short-term reproducibility of replicate analyses (two standard deviations,  $2\sigma$ ) and were sample matrix specific. These calculations are likely to report higher detection limits for elements present at high concentrations than what would be reported if the same element was present at trace levels. In this data set, calcium is a likely example of this behavior.



Table 9: XRF detection limits.

<b>Analyte</b>	<b>Reporting Limit mg/kg</b>	<b>Detection Limit, wt % 2σ (wt. %)</b>
Al	20	0.016
As	20	0.038
Ba	20	0.0084
Br	20	0.02
Ca	20	0.1
Cd	20	0.064
Ce	20	0.022
Cl	20	0.0046
Co	20	0.0024
Cr	20	0.0028
Cu	20	0.0014
F	20	0.082
Fe	20	0.034
Ga	20	0.0016
Ge	20	0.0014
K	20	0.0048
La	20	0.0054
Mg	20	0.01
Mn	20	0.0032
Mo	20	0.0026
Na	20	0.0076
Nb	20	0.0018
Ni	20	0.0048
Pb	20	0.0034
Px	20	0.004
Rb	20	0.0016
Sc	20	0.0016
Se	20	0.0018
Si	20	0.092
Sr	20	0.0016
Sx	20	0.05
Ti	20	0.003
V	20	0.0038
W	20	0.0036
Y	20	0.0018
Zn	20	0.0014
Zr	20	0.0024

### **3.3.2 Acid Digestion with Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) and Inductively Coupled Plasma Mass Spectroscopy (ICP-MS)**

Hydrofluoric acid was used to digest powdered samples by ARCADIS U.S., Inc. in August 2010 according to USEPA SW-846 Method 3052 [8]. Digested samples were then analyzed for major and minor cations using ICP-OES and ICP-MS at Vanderbilt University.

### **3.3.3 Porosity (ASTM C 642)**

Density and porosity of BGM and SVC samples were measured according to the ASTM C 642 standard procedure [9].

### **3.3.4 Moisture Content**

Moisture content of the “as received” reference materials was determined according to ASTM D 2216 [10]. Tabular results are provided in Appendix C.

## **3.4 ANALYTICAL METHODS FOR LEACHING TEST ELUATES AND SOLID SAMPLE DIGESTS**

### **3.4.1 pH and Conductivity**

pH and electrical conductivity were measured for all aqueous eluates using an Accumet 925 pH/ion meter. The pH of the leachates was measured using a combined pH electrode accurate to 0.1 pH units. A 4-point calibration was performed daily using pH buffer solutions at pH 4.0, 7.0, 10.0, and 12.45. Conductivity of the leachates was measured using a standard conductivity probe. The conductivity probe was calibrated using appropriate standard conductivity solutions for the conductivity range of concern. Conductivity meters typically are accurate to  $\pm 1\%$  and have a precision of  $\pm 1\%$ .

### **3.4.2 Dissolved Inorganic Carbon (DIC) and Dissolved Organic Carbon (DOC)**

Analyses of total organic carbon and inorganic carbon were performed on a Shimadzu model TOC-V CPH/CPN. Five-point calibration curves, for both dissolved inorganic carbon (DIC) and non-purgeable dissolved organic carbon (DOC) analyses, were generated for an analytical range between 5 ppm and 100 ppm and are accepted with a correlation coefficient of at least 0.995. An analytical blank and check standard at approximately 10 ppm were run every 10 samples. The standard was required to be within 15% of the specified value. A volume of approximately 16 mL of undiluted sample was loaded for analysis. DIC analysis was performed first for the analytical blank and standard and then the samples. DOC analysis was carried out separately after completion of DIC analysis. DOC analysis began using addition of 2 M (moles/L) of hydrochloric acid to achieve a pH of 2 along with a sparge gas flow rate of 50 mL/min to purge inorganic carbon prior to analysis. Method detection limits (MDLs) and minimum levels of quantification (MLs) for DIC-DOC analysis are listed in Table 10.

Table 10: Method detection limits (MDLs) and minimum level of quantification (ML) for DIC-DOC analysis on liquid samples.

Symbol	Units	MDL	ML
DIC	µg/L	0.08	0.25
DOC	µg/L	0.13	0.40

### 3.4.3 Metals (ICP-MS, ICP-OES)

Liquid samples for ICP-MS and ICP-OES analysis were preserved through addition of 1 mL of concentrated nitric acid (trace metal grade) per 99 mL of sample. Known quantities of each analyte were also added to sample aliquots for analytical matrix spikes.

#### 3.4.3.1 ICP-MS Analysis (SW-846 Method 6020)

ICP-MS analyses of aqueous samples from laboratory leaching tests were carried out at Vanderbilt University (Department of Civil and Environmental Engineering) using a Perkin Elmer model ELAN DRC II in both standard and dynamic reaction chamber (DRC) modes. Standard analysis mode was used for all analytes except for As and Se, which were run in DRC mode with 0.5 mL/min of oxygen as the reaction gas. Seven-point standard curves were used for an analytical range of approximately 0.5 µg/L to 600 µg/L and were completed before each analysis. Analytical blanks and analytical check standards at approximately 50 µg/L were run every 10 to 20 samples and required to be within 15% of the specified value. Samples for analysis were diluted gravimetrically to within the targeted analytical range using 2% v/v Optima grade nitric acid (Fisher Scientific). Initially, analyses for 10:1 dilutions were performed to minimize total dissolved loading to the instrument. Additional dilutions at 100:1 and 1000:1 were analyzed if the calibration range was exceeded with the 10:1 dilution. 50 µL of a 10 mg/L internal standard consisting of indium (In) (for mass range below 150) and bismuth (Bi) (for mass range over 150) was added to 10 mL of sample aliquot prior to analysis. Analytical matrix spikes were completed for one of each of the replicate eluates from Method 1313. For each analytical matrix spike, a volume between 10 µL and 100 µL of a 10mg/L standard solution was added to 10 mL of sample aliquot. Table 11 provides the element analyzed, method detection limit (MDL), and minimum level of quantification (ML). Analyte concentrations measured that are less than the ML and greater than the MDL are reported as estimated value using the instrument response. The values reflect the initial 10:1 dilution used for samples from laboratory leaching tests.

Table 11: Method detection limits (MDLs) and minimum level of quantification (ML) for ICP-MS analysis on liquid samples.

Symbol	Units	MDL	ML
Al	µg/L	0.52	1.64
Sb	µg/L	0.05	0.14
As	µg/L	0.06	0.20
Ba	µg/L	0.41	1.32
Be	µg/L	0.37	1.19
B	µg/L	0.32	1.01
Cd	µg/L	0.06	0.18
Ca	µg/L	0.94	2.98
Cs	µg/L	0.29	0.93
Cr	µg/L	0.27	0.87
Co	µg/L	0.38	1.19
Cu	µg/L	0.43	1.38
Fe	µg/L	0.73	2.32
Pb	µg/L	0.03	0.10
Li	µg/L	0.37	1.18
Mg	µg/L	0.44	1.39
Mn	µg/L	0.22	0.69
Mo	µg/L	0.37	1.18
Ni	µg/L	0.54	1.71
K	µg/L	1.27	4.05
Re	µg/L	0.22	0.71
Rb	µg/L	0.27	0.85
Se	µg/L	0.12	0.37
Si	µg/L	1.35	4.28
Na	µg/L	0.61	1.94
Sr	µg/L	0.38	1.20
Tl	µg/L	0.06	0.19
Sn	µg/L	0.69	2.21
Ti	µg/L	0.51	1.62
U	µg/L	0.17	0.53
V	µg/L	0.19	0.59
Zn	µg/L	0.62	1.98
Zr	µg/L	0.37	1.16

### 3.4.3.2 ICP-OES Analysis (SW-846 Method 6010)

ICP-OES analyses of aqueous samples from laboratory leaching tests were carried out at Vanderbilt University (Department of Civil and Environmental Engineering) using a Varian ICP Model 720-ES. Five-point standard curves were used for an analytical range between approximately 0.1 mg/L and 25 mg/L for trace metals. Seven-point standard curves were used for an analytical range between approximately 0.1 mg/L and 500 mg/L for minerals. Analytical blanks and analytical check standards at approximately 0.5 mg/L were run every 10 to 20 samples and required to be within 15% of the specified value. Initially, analyses were performed on undiluted samples to minimize total dissolved loading to the instrument. Samples for analysis

were diluted gravimetrically to within the targeted analytical range using 1% v/v Optima grade nitric acid (Fisher Scientific) if the maximum calibration was exceeded. Yttrium at 10 mg/L was used as the internal standard. Analytical matrix spikes were completed for three test positions from one of the replicate eluates from Method 1313. For each analytical matrix spike, a volume of 500 µL of a 10 mg/L standard solution was added to 5 mL of sample aliquot. Table 12 provides the element analyzed, method detection limit (MDL) and minimum level of quantification (ML). Analyte concentrations measured that are less than the ML and greater than the MDL are reported as estimated value using the instrument response.

Table 12: Method detection limits (MDLs) and minimum level of quantification (ML) for ICP-OES analysis on liquid samples.

Symbol	Units	MDL	ML
Al	µg/L	1.00	3.18
Sb	µg/L	8.00	25.44
As	µg/L	15.00	47.70
Ba	µg/L	1.00	3.18
Be	µg/L	5.00	15.90
B	µg/L	1.00	3.18
Cd	µg/L	6.00	19.08
Ca	µg/L	3.50	11.13
Cr	µg/L	1.00	3.18
Co	µg/L	1.00	3.18
Cu	µg/L	4.10	13.04
Fe	µg/L	2.90	9.22
Pb	µg/L	7.00	22.26
Li	µg/L	6.00	19.08
Mg	µg/L	1.00	3.18
Mn	µg/L	3.60	11.45
Mo	µg/L	1.00	3.18
Ni	µg/L	2.20	7.00
K	µg/L	1.50	4.77
Se	µg/L	17.00	54.06
Si	µg/L	2.80	8.90
Ag	µg/L	18.00	57.24
Na	µg/L	3.50	11.13
Sr	µg/L	1.00	3.18
Tl	µg/L	5.00	15.90
Sn	µg/L	17.00	54.06
Ti	µg/L	6.40	20.35
V	µg/L	1.30	4.13
Zn	µg/L	2.50	7.95
Zr	µg/L	2.70	8.59
P	µg/L	6.20	19.72
S	µg/L	8.30	26.39

## **4.0 RESULTS AND DISCUSSION**

### **4.1 LEACHING ASSESSMENT RESULTS**

Leaching test results for Methods 1313, 1314, and 1314 are illustrated in their entirety as summary graphs in Appendices C, D, and E, respectively. Explanations of these graphs are provided in the following sections. Results are grouped by test method to facilitate comparison of leaching behaviors between materials. Results are also available electronically in the form of a LeachXS Lite™ database (Filename: “CBP\_RM1\_(12-2010).mdb”). LeachXS Lite can be obtained free of charge from [www.vanderbilt.edu/leaching](http://www.vanderbilt.edu/leaching).

#### **4.1.1 Plotting Conventions**

In general, two repetitions of each test method were analyzed for major and minor constituents. The first repetition is indicated by a solid line and a filled symbol at each data point; the second repetition is indicated by an open symbol of the same color and shape as the first repetition with no line connecting data points. Analytical method detection limit (MDL) is denoted by a dot-dash line and method limit (ML, also frequently referred to as the practical quantification limit) is denoted by a dark, dotted line. In cases where two analytical methods are employed, the MDL and ML correspond to values of the ICP-OES method.

#### **4.1.2 Alkalinity, Solubility and Release as a Function of pH (Method 1313)**

Annotated example output graphs for Method 1313 are shown in Figure 3 and Figure 4. Large circles surrounding data points indicate the equilibrium pH of the system when exposed to deionized water only.

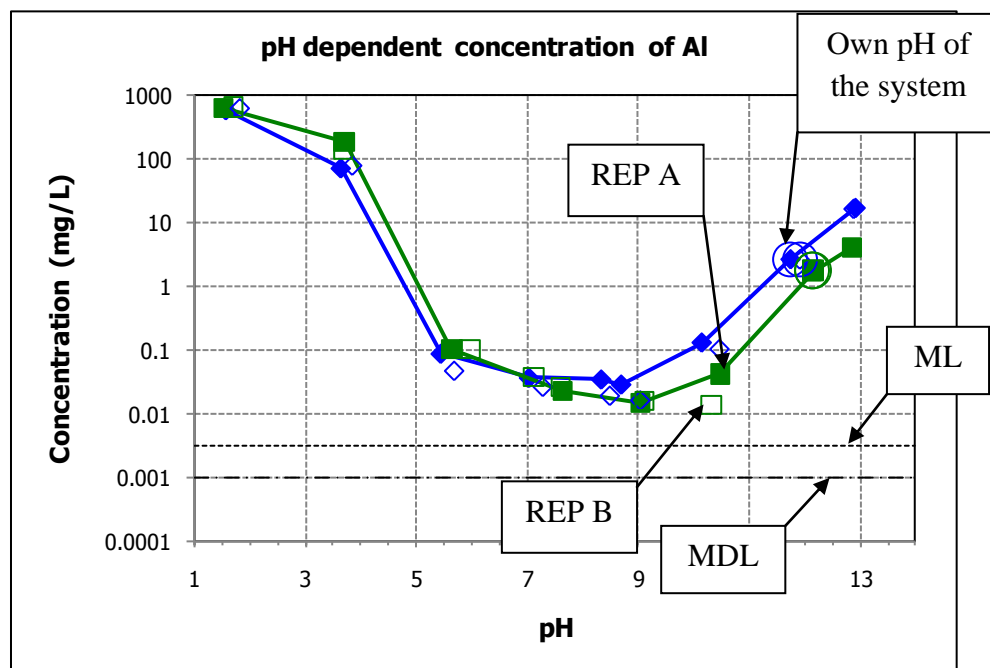


Figure 3: Example output graph for Method 1313 pH dependent concentration.

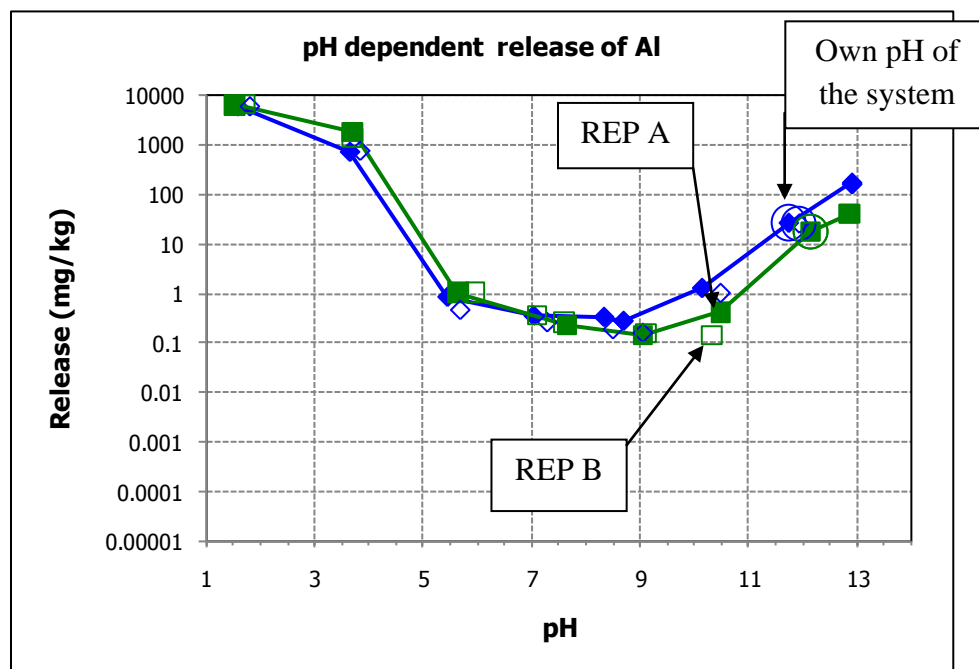


Figure 4: Example output graph for Method 1313 pH dependent release.

#### 4.1.3 Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio in Solid Materials Using an Up-Flow Percolation Column (Method 1314)

Annotated example output graphs for Method 1314 concentration, cumulative release, and eluate pH are shown in Figure 5, Figure 6, and Figure 7, respectively.

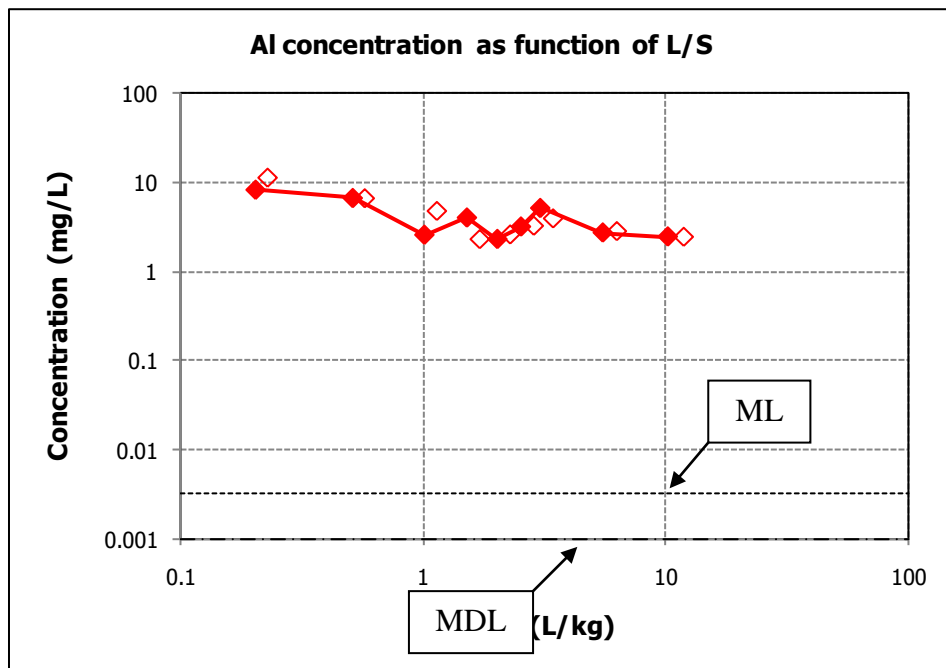


Figure 5: Example output graph for Method 1314 concentration as a function of L/S.

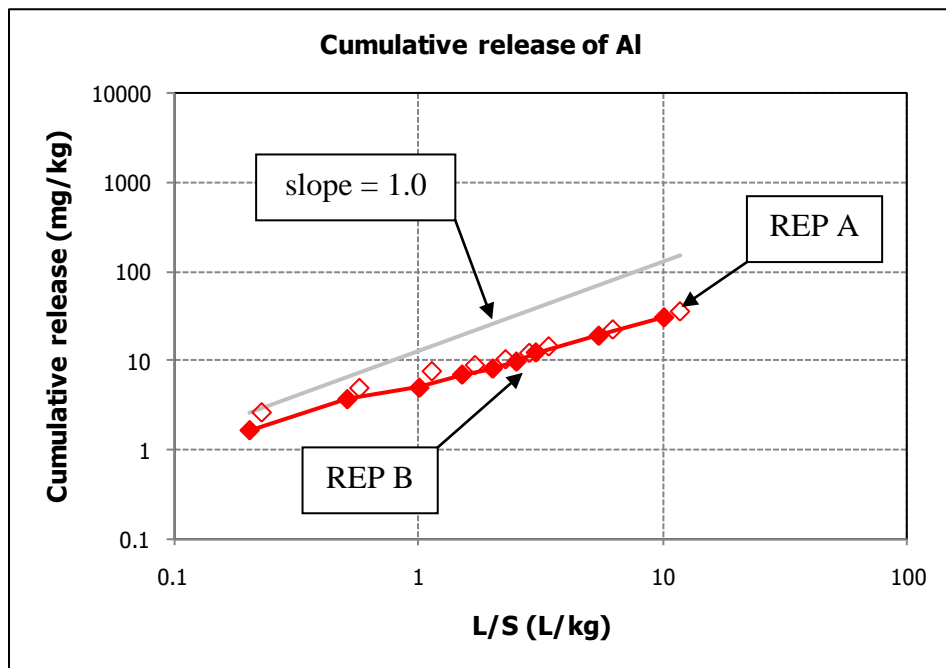


Figure 6: Example output graph for Method 1314 cumulative release as a function of L/S.



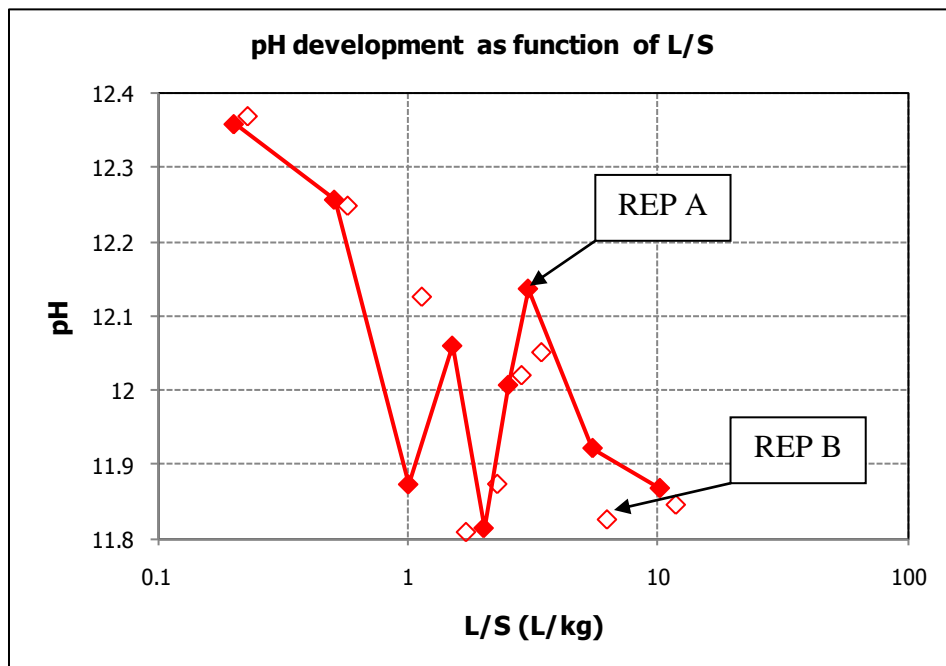


Figure 7: Example output graph for Method 1314 pH development as a function of L/S.

Additionally, a useful comparison is made by superimposing plots of Method 1314 constituent concentration and release as a function of pH onto the Method 1313 solubility curves as a function of release, as demonstrated in Figure 8 and Figure 9, respectively.

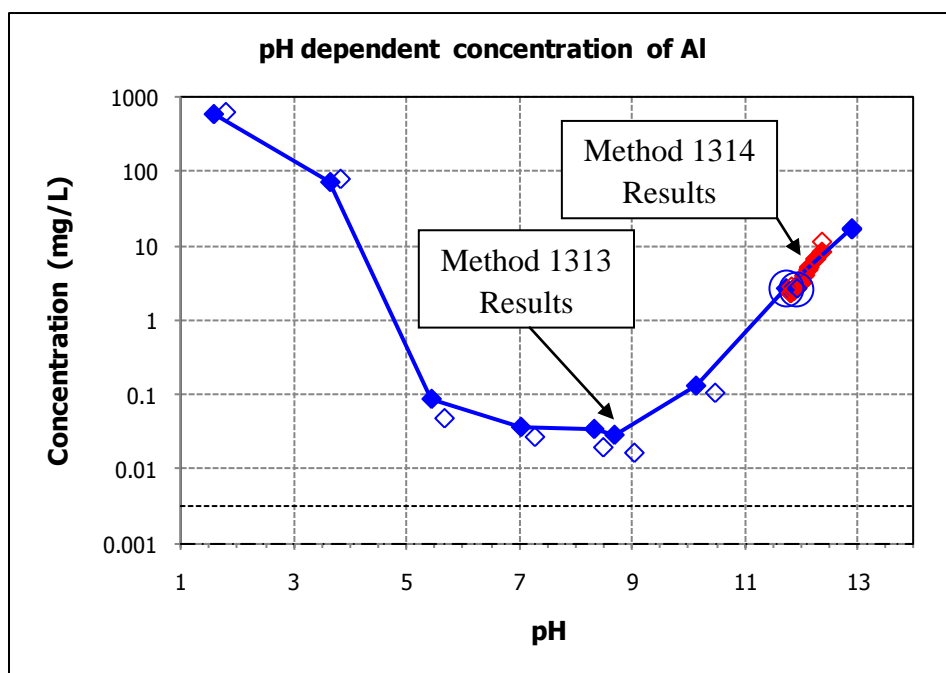


Figure 8: Example output graph for Method 1314 concentration as a function of pH.

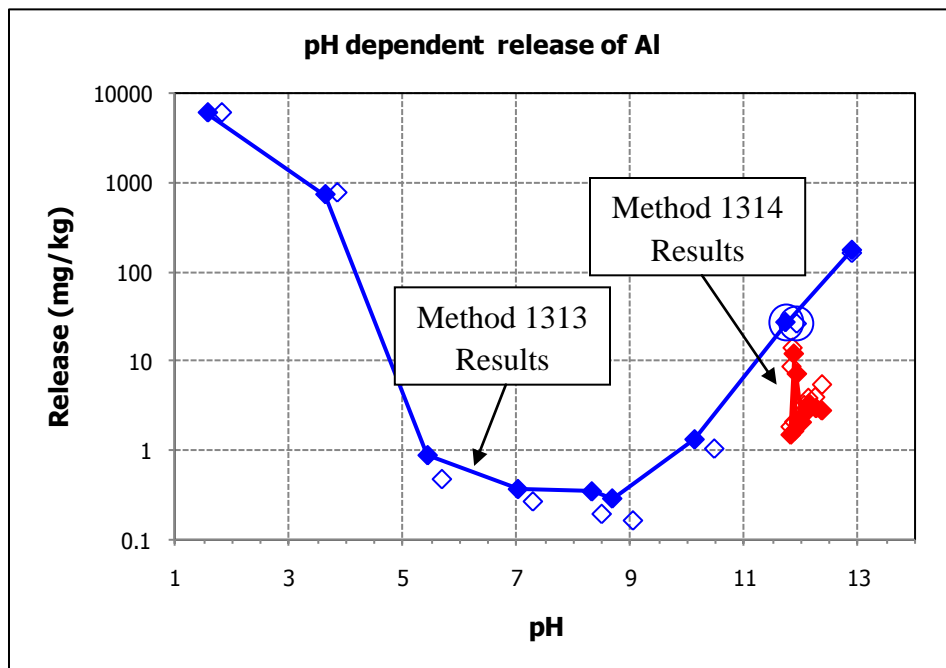


Figure 9: Example output graph for Method 1314 release as a function of pH.

#### 4.1.4 Mass Transfer Rates of Constituents in Monolithic Materials (Method 1315)

Annotated example output graphs for Method 1315 eluate pH, concentration, cumulative release, and flux as a function of time are shown in , respectively. In Figure 12 the gray line with slope of 0.5 serves as a visual indicator of ideal diffusive behavior wherein rate of release is proportional to the square root of time. Similarly, in Figure 13 the gray line with slope of -0.5 illustrates the inverse dependence of ideal flux on the square root of time.

Figure 14 demonstrates Method 1315 eluate concentration as a function of pH superimposed onto Method 1313 concentration as a function of pH.

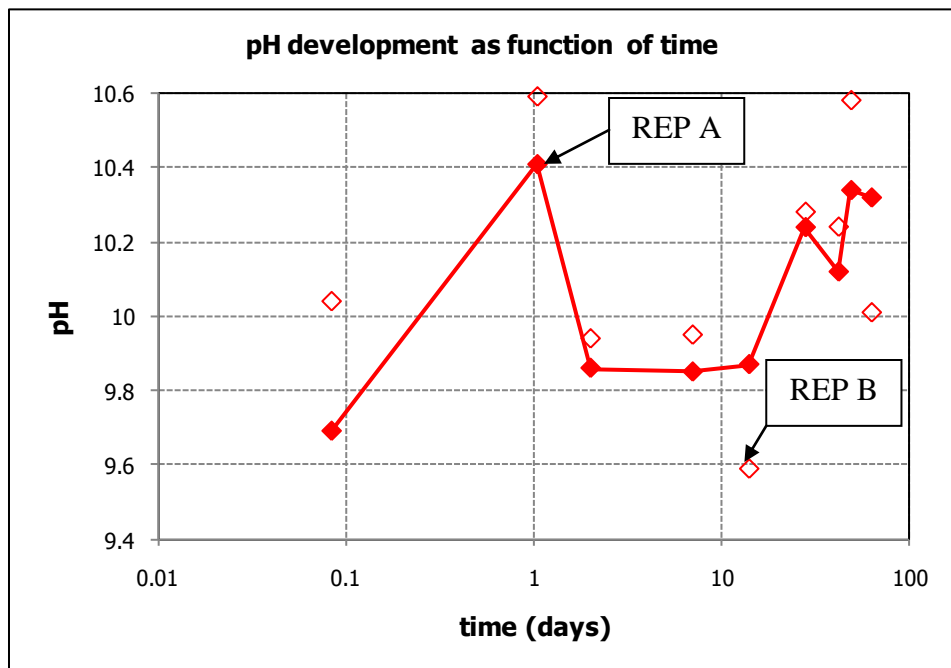


Figure 10: Example output graph for Method 1315 eluate pH as a function of time.

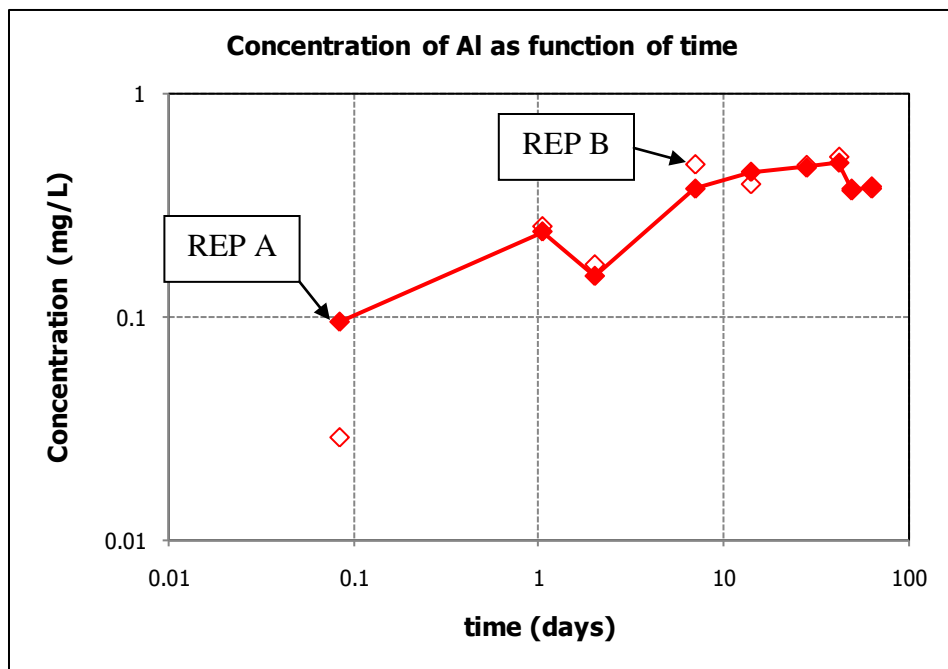


Figure 11: Example output graph for Method 1315 eluate pH as a function of time.

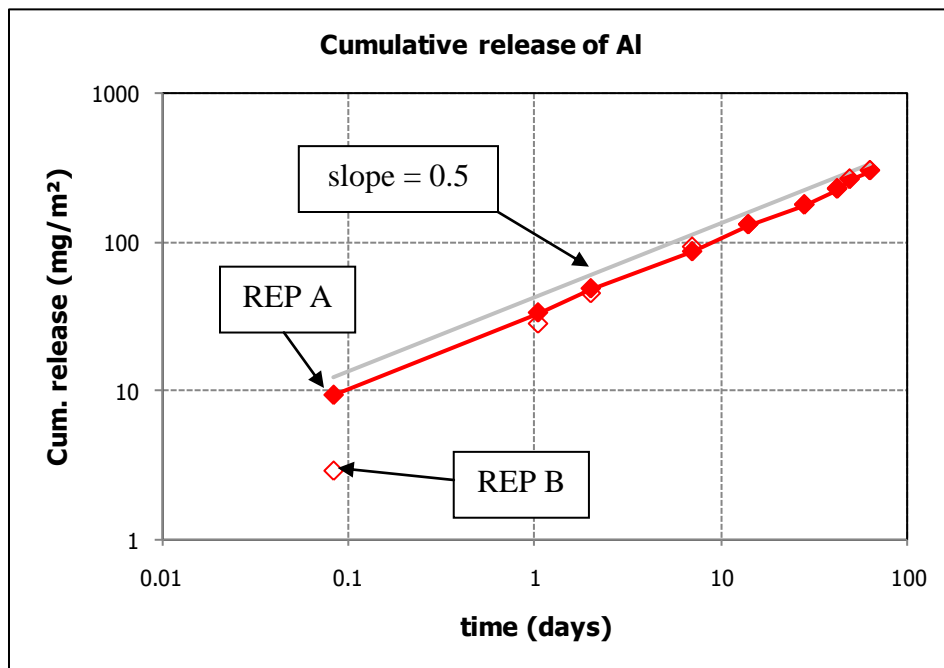


Figure 12: Example output graph for Method 1315 cumulative release as a function of time.

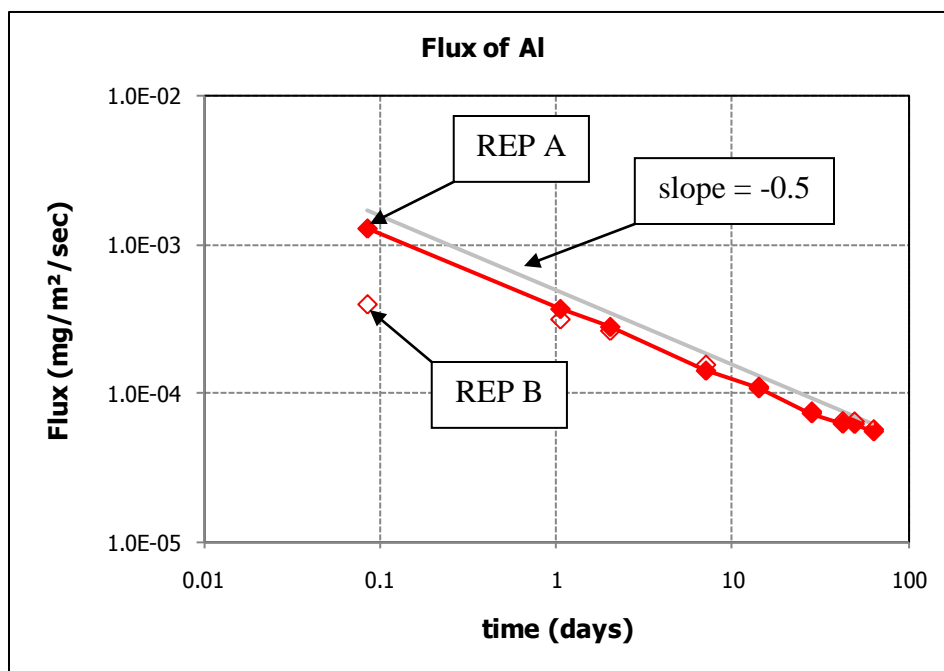


Figure 13: Example output graph for Method 1315 cumulative release as a function of time.

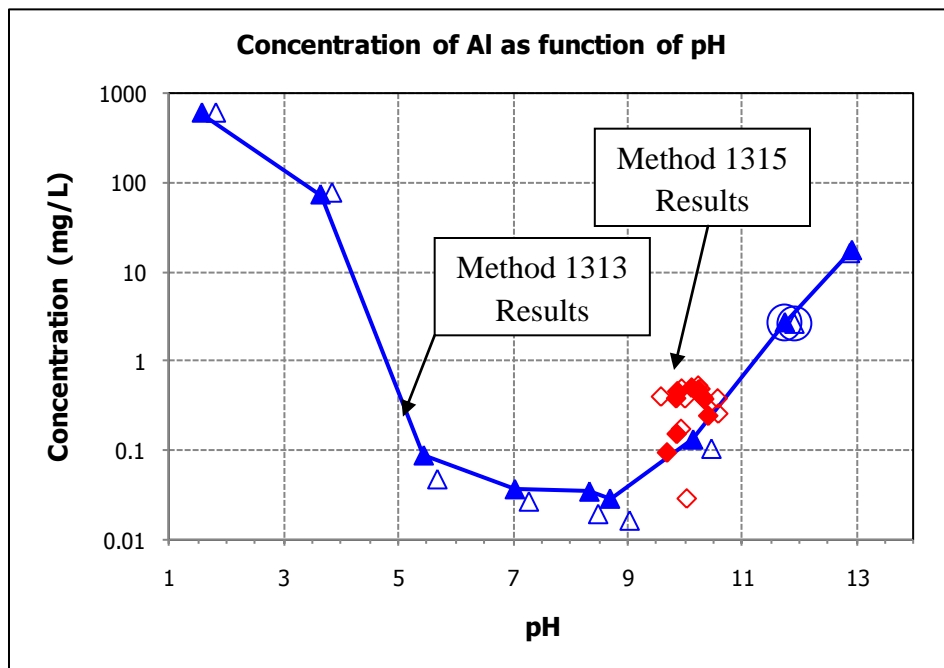


Figure 14: Example output graph for Method 1315 concentration as a function of pH.

## 4.2 SOLID PHASE COMPOSITION AND POROSITY

### 4.2.1 XRF Analysis for Total Content

Results for XRF analyses on cementitious and component materials are given in Table 13 and Table 14, respectively.

Table 13: XRF analysis results for cementitious materials expressed in elemental mass percents. "nd" denotes not detected (below detection limit).

	<b>BGM</b>		<b>SVC</b>		<b>VCO</b>		<b>VCT</b>	
Symbol	<i>Rep A</i>	<i>Rep B</i>	<i>Rep A</i>	<i>Rep B</i>	<i>Rep A</i>	<i>Rep B</i>	<i>Rep A</i>	<i>Rep B</i>
Al	4.231	4.609	3.307	3.725	4.314	3.457	4.481	5.233
As	0.000	0.000	nd	nd	nd	nd	nd	nd
Ba	0.038	0.041	nd	0.053	0.070	0.065	0.068	0.065
C	3.091	3.091	4.330	4.330	6.452	6.452	3.379	3.379
Ca	7.980	9.020	10.102	13.722	8.927	11.699	8.026	6.809
Ce	0.025	nd	nd	nd	nd	nd	nd	nd
Cl	nd	0.125	0.154	0.138	0.070	0.064	0.056	0.043
Co	nd	nd	nd	nd	nd	nd	nd	nd
Cr	0.012	nd	0.010	0.016	nd	0.015	nd	nd
Cu	0.006	0.009	nd	0.008	nd	nd	nd	nd
Fe	1.545	1.757	0.912	1.295	1.144	1.113	1.401	1.173
Ga	nd	nd	nd	nd	nd	nd	nd	nd
K	0.579	0.632	0.333	0.443	1.775	1.444	1.701	1.851
Mg	1.179	1.360	1.855	2.140	0.886	0.890	1.160	1.096
Mn	0.040	0.052	0.072	0.101	0.045	0.046	0.062	0.050
Na	0.064	0.081	0.081	0.126	1.005	0.692	0.861	1.252
Nb	nd	nd	nd	nd	nd	nd	nd	nd
Ni	0.006	0.007	nd	nd	nd	nd	nd	nd
O	32.415	34.904	38.256	34.139	35.379	32.775	34.062	40.926
P	0.050	0.059	0.031	0.036	0.042	nd	0.058	0.060
Pb	0.000	0.000	nd	nd	nd	nd	nd	nd
Rb	0.003	0.004	nd	0.003	0.010	0.009	0.010	0.009
S	0.281	0.331	0.394	0.432	0.261	0.319	0.225	0.211
Si	20.342	21.573	25.261	19.614	22.462	20.015	21.290	27.175
Sr	0.026	0.029	0.021	0.029	0.029	N.03	0.028	0.028
Th	nd	nd	0.004	nd	nd	nd	nd	nd
Ti	0.274	0.312	0.182	0.224	0.179	0.161	0.225	0.186
Y	0.003	0.003	0.004	0.003	0.002	0.002	0.003	0.002
Zn	0.006	0.003	nd	0.006	0.004	0.006	0.006	0.004
Zr	0.012	0.015	0.013	0.020	0.012	0.015	0.016	0.016
SUM	72.208	78.017	85.322	80.603	83.068	79.239	77.118	89.568

Table 14: XRF analysis results for component materials expressed in elemental mass percents.

"nd" denotes nondetect.

	<b>BFS</b>		<b>FAF</b>	<b>OTC</b>		<b>QS</b>	
Symbol	<i>Rep A</i>	<i>Rep B</i>	<i>Rep A</i>	<i>Rep A</i>	<i>Rep B</i>	<i>Rep A</i>	<i>Rep B</i>
Al	3.412	3.500	15.075	2.089	2.204	1.768	1.672
As	nd	nd	0.010	nd	nd	nd	nd
Ba	0.060	0.059	0.116	0.039	0.033	nd	nd
C	nd	nd	1.498	2.502	2.502	0.649	0.649
Ca	22.999	24.492	0.856	39.776	42.815	0.010	nd
Ce	nd	nd	nd	nd	nd	nd	nd
Cl	nd	0.061	0.021	0.028	0.034	0.005	0.041
Co	nd	nd	0.008	nd	nd	nd	nd
Cr	nd	nd	0.019	nd	nd	0.011	0.012
Cu	nd	nd	0.020	nd	0.009	nd	nd
Fe	0.202	0.213	4.976	2.582	2.818	0.170	0.123
Ga	nd	nd	0.007	nd	nd	nd	nd
K	0.330	0.379	2.144	0.357	0.404	0.131	0.123
Mg	6.725	7.038	0.623	0.558	0.607	nd	nd
Mn	0.325	0.320	0.018	0.023	0.019	nd	nd
Na	0.134	0.133	0.250	0.125	0.134	0.013	nd
Nb	nd	nd	0.003	nd	nd	nd	nd
Ni	nd	nd	0.016	nd	nd	nd	nd
O	37.393	38.948	45.488	30.330	32.354	39.200	34.279
P	nd	nd	0.143	0.057	0.060	0.007	0.006
Pb	nd	nd	0.011	nd	nd	nd	nd
Rb	0.002	0.002	0.016	nd	0.002	nd	nd
S	0.802	0.843	0.037	1.496	1.600	0.003	nd
Si	16.801	17.318	24.422	7.499	7.833	32.854	28.645
Sr	0.034	0.038	0.090	0.082	0.087	nd	nd
Th	nd	nd	nd	nd	nd	nd	nd
Ti	0.143	0.156	0.772	0.147	0.176	0.087	0.086
Y	0.002	0.004	0.014	0.002	0.002	nd	nd
Zn	nd	nd	0.015	0.057	0.009	nd	nd
Zr	0.010	0.038	0.025	0.006	0.007	0.028	0.016
SUM	89.374	93.542	96.693	87.755	93.709	74.936	65.652

#### 4.2.2 Acid Digestion with Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

Results for total elemental content of both major and minor species are given in Table 15 and Table 16, respectively. Data are expressed in elemental mass percents, normalized to the total dry mass of the specimen.

Table 15: ICP-OES and ICP-MS analysis results for total content of acid digested cementitious materials, in mass percents.

Symbol	Method	BGM	SVC	VCT	VCO
Al	OES	3.284	2.451	4.560	4.044
Sb	MS	1.89(10) <sup>-04</sup>	1.29(10) <sup>-04</sup>	7.46(10) <sup>-05</sup>	9.00(10) <sup>-05</sup>
As	MS	0.002	8.40(10) <sup>-04</sup>	1.81(10) <sup>-04</sup>	4.23(10) <sup>-04</sup>
Ba	OES	0.022	0.017	0.038	0.043
Be	MS	4.95(10) <sup>-04</sup>	4.12(10) <sup>-04</sup>	2.32(10) <sup>-04</sup>	3.50(10) <sup>-04</sup>
Cd	MS	9.87(10) <sup>-06</sup>	1.49(10) <sup>-05</sup>	1.29(10) <sup>-05</sup>	1.33(10) <sup>-05</sup>
Ca	OES	4.812	7.341	5.341	7.246
Cs	MS	1.65(10) <sup>-04</sup>	8.57(10) <sup>-05</sup>	1.70(10) <sup>-04</sup>	1.88(10) <sup>-04</sup>
Cr	OES	0.007	0.003	0.002	0.002
Co	MS	0.001	5.16(10) <sup>-04</sup>	3.32(10) <sup>-04</sup>	5.32(10) <sup>-04</sup>
Cu	MS	0.004	0.002	0.001	0.002
Fe	OES	1.377	0.758	1.478	1.253
Pb	MS	0.001	8.32(10) <sup>-04</sup>	0.002	0.001
Li	OES	0.004	0.003	0.002	0.003
Mg	OES	0.754	1.210	0.763	0.752
Mn	OES	0.033	0.056	0.044	0.045
Mo	MS	7.25(10) <sup>-04</sup>	3.49(10) <sup>-04</sup>	2.10(10) <sup>-04</sup>	2.68(10) <sup>-04</sup>
Ni	MS	0.003	0.002	9.31(10) <sup>-04</sup>	9.79(10) <sup>-04</sup>
P	OES	0.039	0.026	0.065	0.040
K	OES	0.413	0.272	1.628	1.883
Re	MS	4.76(10) <sup>-06</sup>	5.30(10) <sup>-06</sup>	4.93(10) <sup>-06</sup>	4.90(10) <sup>-06</sup>
Se	MS	0.002	0.001	0.001	0.002
Na	OES	0.038	0.066	0.672	0.523
Sr	OES	0.022	0.016	0.021	0.021
S	OES	0.214	0.329	0.203	0.280
Tl	MS	1.04(10) <sup>-04</sup>	6.33(10) <sup>-05</sup>	6.63(10) <sup>-05</sup>	6.63(10) <sup>-05</sup>
Sn	MS	0.011	3.15(10) <sup>-04</sup>	0.003	6.02(10) <sup>-04</sup>
Ti	OES	0.249	0.200	0.270	0.219
U	MS	3.64(10) <sup>-04</sup>	3.02(10) <sup>-04</sup>	2.63(10) <sup>-04</sup>	4.03(10) <sup>-04</sup>
V	OES	0.007	0.004	0.005	0.003
Zn	OES	0.005	0.004	0.004	0.006
SUM		11.306	12.766	15.106	16.373



Table 16: ICP-OES and ICP-MS analysis results for total content of acid digested component materials, in mass percents.

Symbol	Method	BFS	FAF	OPC	QS
Al	OES	3.778	13.690	2.401	0.837
Sb	MS	5.07(10) <sup>-05</sup>	8.55(10) <sup>-04</sup>	0.001	1.04(10) <sup>-04</sup>
As	MS	5.64(10) <sup>-05</sup>	0.008	0.001	4.74(10) <sup>-04</sup>
Ba	OES	0.043	0.090	0.020	0.003
Be	MS	0.001	0.002	4.29(10) <sup>-04</sup>	3.83(10) <sup>-04</sup>
Cd	MS	1.31(10) <sup>-05</sup>	5.92(10) <sup>-05</sup>	5.22(10) <sup>-05</sup>	1.75(10) <sup>-05</sup>
Ca	OES	26.660	0.832	39.821	0.079
Cs	MS	2.69(10) <sup>-05</sup>	9.88(10) <sup>-04</sup>	6.06(10) <sup>-05</sup>	1.82(10) <sup>-04</sup>
Cr	OES	0.006	0.017	0.007	7.10(10) <sup>-04</sup>
Co	MS	8.54(10) <sup>-05</sup>	0.006	0.001	5.57(10) <sup>-04</sup>
Cu	MS	1.55(10) <sup>-05</sup>	0.016	0.005	0.002
Fe	OES	0.228	4.769	2.137	0.095
Pb	MS	6.17(10) <sup>-05</sup>	0.007	0.001	0.001
Li	OES	0.004	0.015	0.002	5.79(10) <sup>-04</sup>
Mg	OES	7.462	0.576	0.580	0.010
Mn	OES	0.326	0.015	0.020	0.002
Mo	MS	2.93(10) <sup>-04</sup>	0.002	7.61(10) <sup>-04</sup>	6.22(10) <sup>-04</sup>
Ni	MS	4.51(10) <sup>-04</sup>	0.012	0.004	0.001
P	OES	7.23(10) <sup>-04</sup>	0.132	0.070	0.003
K	OES	0.502	2.156	0.470	0.054
Re	MS	5.30(10) <sup>-06</sup>	4.81(10) <sup>-06</sup>	4.81(10) <sup>-06</sup>	4.89(10) <sup>-06</sup>
Se	MS	0.002	0.007	9.84(10) <sup>-04</sup>	0.001
Na	OES	0.163	0.166	0.142	0.006
Sr	OES	0.033	0.079	0.070	6.59(10) <sup>-04</sup>
S	OES	1.272	0.048	1.606	0.062
Tl	MS	1.13(10) <sup>-05</sup>	4.49(10) <sup>-04</sup>	3.26(10) <sup>-05</sup>	7.70(10) <sup>-05</sup>
Sn	MS	3.67(10) <sup>-04</sup>	0.011	0.016	7.02(10) <sup>-04</sup>
Ti	OES	0.186	0.818	0.166	0.087
U	MS	4.71(10) <sup>-04</sup>	0.001	7.00(10) <sup>-04</sup>	4.01(10) <sup>-04</sup>
V	OES	0.004	0.032	0.008	7.86(10) <sup>-04</sup>
Zn	OES	0.003	0.012	0.043	9.38(10) <sup>-04</sup>
SUM		40.676	23.521	47.594	1.252

#### 4.2.3 Porosity (ASTM C 642)

Values of bulk dry density, bulk saturated density, and porosity for BGM and SVC are reported in Table 17 and Table 18, respectively.

Table 17: Mean (MEAN) and standard deviation (STD) values for density and porosity of BGM.

	Bulk density, dry [g/cm <sup>3</sup> ]	Bulk density, saturated [g/cm <sup>3</sup> ]	Porosity [cm <sup>3</sup> voids/cm <sup>3</sup> ]
MEAN	1.96	2.19	0.23
STD	0.02	0.02	0.01

Table 18: Mean (MEAN) and standard deviation (STD) values for density and porosity of SVC.

	Bulk density, dry [g/cm <sup>3</sup> ]	Bulk density, saturated [g/cm <sup>3</sup> ]	Porosity [cm <sup>3</sup> voids/cm <sup>3</sup> ]
MEAN	2.02	2.22	0.20
STD	0.02	0.02	0.01

## 5.0 REFERENCES

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