

Leaching Behavior of “AGREMAX” Collected from a Coal-Fired Power Plant in Puerto Rico

A.C. Garrabrants¹, D.S. Kosson¹, R. DeLapp¹ and Peter Kariher²

¹ Vanderbilt University
Civil and Environmental Engineering
Nashville, TN 37215

² ARCADIS-US, Inc.
Research Triangle Park, NC 27711

Category III / Applied Research

Contract No. EP-C-09-027
Work Assignment No. 3-07

Prepared for

Susan A. Thorne

U.S. Environmental Protection Agency
Office of Research and Development
National Risk Management Research Laboratory
Air Pollution Prevention and Control Division
Research Triangle Park, NC 27711

Notice

The U.S. Environmental Protection Agency through its Office of Research and Development partially funded and managed the research described here under Contract Number EP-C-09-27 coordinated by ARCADIS-US, Inc. Prior to publication, this report will be subjected to the Agency's peer and administrative review to be approved for publication as an EPA document.

For Review Purposes Only - Do Not Quote or Cite

ABSTRACT

"AGREMAX" is a partially solidified mixture of coal combustion fly ash and bottom ash. Under the request of the United States Environmental Protection Agency (US EPA) Region 2, the US EPA Office of Research and Development (ORD) has performed two of the leaching tests contained in the Leaching Environmental Assessment Framework (LEAF) on subsamples of AGREMAX sampled from the AES Puerto Rico L.P. coal-fired power plant in Guayama, Puerto Rico. Subsamples of AGREMAX were collected and shipped under chain of custody to ARCADIS-US, Inc. (ARCADIS) serving as the on-site contractor for the EPA ORD Air Pollution Prevention and Control Division. Homogenized samples of the solid material were tested to characterize leaching behavior of AGREMAX during pH-dependent leaching using Method 1313 and liquid-to-solid ratio (L/S)-dependent leaching in a percolation column using Method 1314. Method 1313 was conducted at EPA's Research Triangle Park Laboratories by ARCADIS while Method 1314 was performed at Vanderbilt University (VU). Leaching test eluates were analyzed for nearly 40 target analytes including major, minor and trace constituents.

This document presents the results of LEAF-based leaching tests. Eluate concentrations from these leaching methods were compared to the minimum of indicator values derived from US EPA Region 9 regional screening levels (RSLs) for tapwater or US national drinking water regulations (NDWRs) as requested by US EPA Region 2 to put eluate concentrations into perspective. The comparison was based on an indicator ratio defined as the maximum applicable eluate concentration divided by the minimum indicator value. The maximum applicable eluate concentration was considered to be the maximum eluate concentration within an applicable pH range of pH 6.5 to 11.5 for Method 1313 or the maximum eluate concentration over the entire L/S range between 0.2 and 10 mL/g-dry for Method 1314.

Indicator ratios greater than 100 were observed for arsenic, boron, chloride and chromium based on results from both leaching tests. For fluoride, lithium and molybdenum, indicator ratios for Method 1314 test results were greater than 100 while indicator ratios for Method 1313 test results were between 10 and 100. Other analytes with indicator ratios between 10 and 100 include selenium, sulfate and thallium in both pH- and L/S-dependent leaching tests.

ABBREVIATIONS

ARCADIS	ARCADIS-US, Inc., Research Triangle Park, NC
CCR(s)	coal combustion residue(s)
DIC	dissolved inorganic carbon
DOC	dissolved organic carbon
DQI(s)	data quality indicator(s)
DWEL(s)	drinking water equivalent level(s)
IC	ion chromatography
ICP-MS	inductively coupled plasma-mass spectrometry
ICP-OES	inductively coupled plasma-optical emission spectroscopy
LEAF	Leaching Environmental Assessment Framework
L/S	cumulative liquid-to-solid ratio [L/kg, dry basis]
L/S _i	liquid-to-solid ratio through end of interval or test fraction <i>i</i> [mL/g, dry basis]
LSP	liquid-solid partitioning
MCL(s)	maximum concentration level(s)
MDL	method detection limit
ML	minimum level of quantitation
NDWR(s)	national drinking water regulation(s)
ORD	Office of Research and Development (US EPA)
QAPP	quality assurance project plan
QA/QC	quality assurance/quality control
RCRA	Resource Conservation and Recovery Act
RSL(s)	regional screening level(s), US EPA Region 9
TCLP	toxicity characteristic leaching procedure, US EPA Method 1311
US EPA	United States Environmental Protection Agency
VU	Vanderbilt University, Nashville, TN

Table of Contents

ABSTRACT	II
ABBREVIATIONS.....	III
INTRODUCTION	1
CHARACTERIZATION APPROACH	1
MATERIAL DESCRIPTION	2
CHARACTERIZATION METHODS	2
<i>Method 1313: Liquid-Solid Partitioning as a Function of Eluate pH using a Parallel Batch Extraction Procedure.....</i>	<i>2</i>
<i>Method 1314: Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio using an Up-Flow Percolation Column Procedure.....</i>	<i>3</i>
ELUATE CHEMICAL ANALYSIS.....	4
<i>Inductively-Coupled Plasma – Optical Emissions Spectrometry.....</i>	<i>4</i>
<i>Inductively-Coupled Plasma – Mass Spectrometry.....</i>	<i>4</i>
<i>Ion Chromatograph.....</i>	<i>4</i>
<i>Total Carbon Analysis.....</i>	<i>4</i>
<i>Quality Assurance/Quality Control.....</i>	<i>5</i>
INDICATOR VALUES.....	5
RESULTS AND DISCUSSION	8
TITRATION CURVE AND ELUATE pH.....	8
ELUATE CONCENTRATIONS AS FUNCTIONS OF PH AND L/S.....	8
DATA QUALITY EVALUATION.....	10
<i>Analytical QA/QC Results.....</i>	<i>10</i>
<i>Method Blank Analysis Results.....</i>	<i>11</i>
COMPARISON OF LEACHING RESULTS TO INDICATOR VALUES.....	11
SUMMARY	13
REFERENCES	14

APPENDIX A Graphical Leaching Test Data

APPENDIX B Detailed QA/QC Report

INTRODUCTION

Historically, the results of leaching tests have been the basis for environmental performance assessment of solid materials (e.g., contaminated materials, industrial wastes). For example, the United States Environmental Protection Agency (US EPA) uses the Toxicity Characteristic Leaching Procedure (TCLP; EPA Method 1311) as a regulatory test to classify wastes as “hazardous” or “non-hazardous” (i.e., wastes to be managed under Subtitle C or Subtitle D of RCRA, respectively). TCLP is a single batch extraction procedure using acetic acid that is considered to produce an eluate that estimates the leachate resulting from co-disposal of the test material with municipal solid waste. The single extraction of TCLP allows for a simple evaluation process by comparison of eluate concentration results to allowable concentration limits for selected constituents (see 40 CFR 261.24). Although the results of any scenario-simulation test should not be used outside of the intended simulation test conditions, the results of TCLP have been broadly applied beyond their intended use in waste classification (SAB, 2003).

More recently, the US EPA has been in the process of developing, validating, and adopting the leaching tests of the Leaching Environmental Assessment Framework (LEAF) for inclusion into its analytical chemistry testing guidance, SW-846, with intended use in situations where TCLP is not required or best-suited (Garrabrants et al., 2010; 2012a; 2012b). The LEAF testing and assessment approach has been developed to provide a comprehensive testing regime through multi-point leach testing over a range of test conditions considered suitable for complex environmental evaluations and material characterizations. The LEAF testing approach, test methods and assessment methodology were developed in parallel with European leach test development efforts. A primary difference between the LEAF methods and TCLP is that the LEAF methods are intended to characterize leaching from a material over a broad range of test conditions that may occur in the environment (e.g., varying values for pH, liquid-to-solid ratio, waste form, etc.) rather than at a single set of test conditions. Testing over a range of conditions provides assessment flexibility in that the results can be used to compare potential release under various environmental conditions or between material treatments. The results of the four leaching tests in LEAF may be used individually or in combinations to characterize the leaching behavior of a material for many plausible field scenarios. The LEAF testing results are intended to provide a source term for subsequent constituent transport and fate evaluation, recognizing that dilution and attenuation of constituents often will occur from the point of release to the point of exposure or compliance evaluation. The LEAF testing methodologies have been applied extensively to coal combustion residues (CCRs) as part of US EPA research on the potential impacts of land disposal or from engineering and commercial applications using fly ash and other secondary materials (Sanchez et al., 2006; 2008; Kosson et al., 2009; Thorneloe, 2010).

US EPA Office of Research and Development (ORD) was asked by US EPA Region 2 to determine the leaching properties of a blend of coal combustion fly ash and bottom ash (“AGREMAX”) using the LEAF leach testing approach. This report presents the results of LEAF testing of AGREMAX samples collected from the AES Puerto Rico L.P. coal-fired power plant in Guayama, Puerto Rico.

CHARACTERIZATION APPROACH

Leaching tests for determining the liquid-solid partitioning (LSP) of AGREMAX as a function of pH using Method 1313 and as a function of liquid-to-solid ratio (L/S) using EPA Method 1314. Batch pH-dependence testing (Method 1313) was conducted by ARCADIS-US, Inc. (ARCADIS) serving as an on-site contractor for the EPA ORD Air Pollution Prevention and Control Division at the National Risk Management and Research Laboratory in the Research Triangle Park, North Carolina. Percolation column testing using Method 1314 was conducted at Vanderbilt University (VU). The remaining LEAF test methods (i.e., EPA Method 1315 and

EPA Method 1316) were not conducted because Method 1315 was not considered appropriate based on the granular nature of the AGREMAX while Method 1316 is somewhat redundant in that it yields similar leaching data as provided by Method 1314. All leaching tests were performed between May and July 2012 and were completed within three months of receipt of the field samples from Region 2. Eluate pH and conductivity were measured directly by the performing laboratory (i.e., ARCADIS for Method 1313 or VU for Method 1314) and eluate chemical analysis for analytes of concern was conducted at VU. All chemical analyses were conducted in accordance with an on-going quality assurance project plan (QAPP) for characterization of CCRs approved by US EPA ORD (Kosson et al., 2009).

In order to put the results of Method 1313 and Method 1314 into perspective with environmental reference concentrations, this report compares leaching test eluate concentrations to a set of indicator values recommended by US EPA Region 2 (Grossman, personal communications, 19 July 2012; 20 July 2012) for this application. The indicator values were derived from US national drinking water regulations (NDWRs) based on a combination of primary and secondary drinking water standards (US EPA, 2012a) and on US EPA Region 9 regional screening levels (RSLs) for residential tapwater (US EPA, 2012b). A comparison was made between leaching data and the environmental reference concentrations for 29 analytes including Al, As, B, Ba, Be, Cd, Cl, Cr, Co, Cu, F, Fe, Li, Mn, Mo, Ni, NO₂, NO₃, Pb, PO₄, Sb, Se, SO₄, Sr, Tl, Sn, U, V and Zn.

MATERIAL DESCRIPTION

AGREMAX is generated by the AES Puerto Rico, LP coal-fired power plant in Guayama, Puerto Rico, and is a combination of 80% coal combustion fly ash and 20% bottom ash by weight. The component materials are mixed together and hydrated. The combined material is transported by conveyor belt to a yard where it is laid out to cure for 7 to 14 days. The cured solidified material is broken into manageable pieces using heavy machinery and transferred to a crushing machine for particle size reduction (Rivera, personal communication, 16 June 2011).

Two 5-gallon plastic buckets of cured and crushed AGREMAX were collected from the AES Puerto Rico L.P. coal-fired power plant in Guayama, Puerto Rico and sent under chain of custody by US EPA Region 2 to ARCADIS for homogenization prior to testing. The samples were designated with the material code "AES" and a replicate letter based on the sample location (e.g., "AES-A" was collected as one location while "AES-B" was collected at the other sampling location). The two buckets from each location were homogenized by cone and quartering (ASTM, 2003). After homogenization, samples were returned to the 5-gallon buckets and stored at ARCADIS for archiving and pH-dependent leach testing using Method 1313. Subsamples from each sample location – enough to fill two 500-mL high density polyethylene bottles – were sent to VU for percolation leach testing using Method 1314.

CHARACTERIZATION METHODS

Method 1313: Liquid-Solid Partitioning as a Function of Eluate pH using a Parallel Batch Extraction Procedure

Method 1313 is an equilibrium-based leaching test designed to provide eluate solutions representing the LSP curve of constituents as a function of eluate pH (Garraabrants et al., 2010; 2012a; 2012b). The procedure consists of nine parallel batch extractions at targeted pH values and one extraction at the natural pH of the

material.¹ The solid material may require particle size reduction by crushing in order to facilitate the approach to solid-liquid equilibrium within a reasonable extraction timeframe. Dilute acid or base in deionized water is added to each extraction according to a pre-test titration in order to achieve final extract pH values at specified target values ranging between 2 and 13 at an L/S of 10 mL/g-dry. The extraction contact time ranges from 24 to 72 hours based on the grain size of the “as tested” material (i.e., the material after any particle size reduction or air drying required to improve the handling of the “as received” material). The pH and conductivity of the final extract solution are recorded and vacuum- or pressure-assisted filtration is used to separate the liquid and solid phases prior to chemical analysis of the eluate. Eluate concentrations for constituents of interest are plotted as a function of eluate pH allowing for comparison to quality control and assessment limits. Eluate concentrations may also be interpolated to the target pH values to provide a uniform basis for comparison of results as the recorded eluate pH is likely to differ slightly from target values within specified pH tolerances.

Method 1314: Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio using an Up-Flow Percolation Column Procedure

Method 1314 is an up-flow percolation column procedure used to evaluate the release of constituents from solid materials as a function of cumulative L/S (Garraabrants et al., 2010; 2012a; 2012b).² In the context of the column test, L/S is defined as the volume of liquid passing through the column relative to the dry equivalent mass of test material in the column bed and is expressed in units of mL/g-dry. As it relates to conditions in the field, L/S can be considered a surrogate measure for time when infiltration rates are considered. Approximately 300-600 grams of “as is” or air-dried solid material (moisture content less than ca. 20% dry basis) is packed under moderate compactive effort into a 5-cm diameter x 30-cm long column. The test material is packed in five “lifts” with each lift hand tamped using a 1-cm diameter rod. Layers of clean silica sand are used at the top and bottom of the column to provide flow regulation on the inlet side and coarse filtration at the outlet. Leaching solution (eluent) is pumped upward through the material and eluate is collected as nine discrete volume fractions of the continuous elution volume. The up-flow percolation mode is intended to minimize air entrapment and flow channeling. The pump flow rate is adjusted to provide a volume of eluent equivalent to 0.75 ± 0.25 L/S per day. For primarily inorganic materials, deionized water is used as the eluent for testing; however, a 1 mM solution of CaCl_2 may be used when testing certain materials (e.g., organic soils, clayey materials) where deflocculation of clay layers or dissolution of organic carbon may be a concern. The collection bottle is placed at a height of 6 to 12 inches above the column to provide sufficient hydraulic head to ensure flow while avoiding siphoning as well as to minimize backpressure which can cause leaks in the system. The nine eluate fractions are collected at specified L/S values of 0.2, 0.5, 1.0, 1.5, 2.0, 4.5, 5.0, 9.5, and 10 mL/g-dry. Analytical sample compositing is allowed depending on the intended use of the test results. The eluate pH, conductivity and (optionally) oxidation-reduction potential are recorded for each fraction prior to filtration through a $0.45\text{-}\mu\text{m}$ membrane and preservation of an analytical sample. After chemical analysis of analytical samples, cumulative release from the column at the specified L/S values is calculated from eluate concentrations and interval liquid-solid ratios (L/S_i). The outputs of Method 1314 include graphs of eluate concentration and cumulative release plotted as a function of L/S, which are intended to illustrate changes in leaching that develop as percolation progresses and L/S increases.

¹ The natural pH (also referred to as “own pH”) is the final eluate pH response of a deionized water extraction (i.e., no acid or base added) of a solid material conducted at an L/S 10 mL/g-dry.

² For the purposes of this report, both batch liquid-to-solid ratio and column test cumulative liquid-to-solid ratio will be denoted as L/S whereas the liquid-to-solid ratio for individual test fractions from the column will be denoted as L/S_i where the value i represents the endpoint L/S. For example, $\text{L}/\text{S}_{0.2}$ refers to first fraction of Method 1314 starting at $\text{L}/\text{S}=0$ mL/g-dry and ending at an $\text{L}/\text{S}=0.2$ mL/g-dry while L/S_{10} refers to the last fraction of the test starting at $\text{L}/\text{S}=9.5$ mL/g-dry ending at $\text{L}/\text{S}=10$ mL/g-dry.

Eluate Chemical Analysis

All eluate solutions were analyzed at VU to minimize variability in the results associated with individual analytical methods and quality assurance/quality control (QA/QC) assessments. A suite of 40 analytes were measured for the purposes of preparing data for any future potential chemical speciation or other modeling.

Inductively-Coupled Plasma – Optical Emissions Spectrometry

The concentrations of a suite of analytes were measured by inductively coupled plasma – optical emissions spectrometry (ICP-OES) on a Varian ICP Model 720-ES (Agilent Technologies, Santa Clara, CA) following SW-846 Method 6010 (US EPA, 2007b). An ICP-OES analytical range of approximately 0.1 mg/L to 25 mg/L for trace metals was calibrated using five-point standard curves and the analytical range was extended for major components to a maximum of 500 mg/L using seven-point standard curves. During analysis of solutions, blanks and calibration check standards (~0.5 mg/L) were monitored every 10 to 20 samples with the requirement that recoveries be within 20% of the specified value. Analyses were performed on undiluted samples when possible and, when necessary, samples were diluted gravimetrically to within the targeted analytical range using 1% (v/v) Optima grade nitric acid (Fisher Scientific) in deionized water. Yttrium at 10 mg/L was used as an internal standard. One analytical matrix spike was checked for each replicate set of analytical samples of a leaching test (i.e., approximately one spike for every ten analytical samples). For each 5 mL sample aliquot, spikes were prepared by addition of 100 µL of a 10 µg/L standard solution for trace species or 100 µL of a 1,000 µg/L standard solution for major species.

Inductively-Coupled Plasma – Mass Spectrometry

Inductively-coupled plasma – mass spectrometry (ICP-MS) was used to determine eluate concentrations for analytes where the detection limit for ICP-OES was above the screening criteria (i.e., As, Be, Sb, Se, Tl) and for analytes not well-quantified by ICP-OES (i.e., Cs, Re, Sn). ICP-MS was conducted following SW-846 Method 6020 (US EPA, 2007c) using a Perkin Elmer model ELAN DRC II (Perkin Elmer, Waltham, MA). A seven point calibration curve was created by dilution of a bulk standard and used to calibrate the analytical range for low-level analytes (0.01 to 100 µg/L) and mid-level analytes (1 to 250 µg/L). ICP-MS was conducted initially on 10:1 volumetric dilutions (water-to-sample) of eluate solutions to minimize total dissolved solids loading to the instrument. If the concentrations of the 10:1 dilutions exceeded the calibration range, additional dilutions at 100:1 and 1000:1 were analyzed. If analysis of the diluted sample resulted in concentrations below the ML, the analysis is repeated with full strength analytical sample. Internal standards consisting of 50 µL of each a 10 mg/L indium solution and a 10 mg/L bismuth solution were added to each 10 mL diluted sample aliquot prior to analysis. Internal standards of indium and bismuth are used to adjust for the concentration response for analytes with mass range below and above 150, respectively.

Ion Chromatograph

The concentrations of anions (i.e., Br, Cl, F, NO₂, NO₃, PO₄, SO₂) analytical samples were determined by ion chromatography (IC) using a Dionex DX600 chromatograph (Dionex Corporation, Sunnyvale, CA) with a conductivity detector following SW-846 Method 9056A (US EPA, 2007d). A five-point standard curve was created by dilution of a seven-anion bulk standard and used to calibrate the analytical range which varied based on the relative concentrations of analytes in the bulk standard. Analytical blanks and calibration check standards at approximately 1 mg/L were measured after every ten analytical samples and compared to acceptable QA/QC criteria (i.e., calibration standard recoveries were required to be 85-115% and concentrations in analytical blanks required to be non-detectable).

Total Carbon Analysis

Total carbon analysis following SW-846 Method 9060 (US EPA, 2004) for all eluates was performed on a Shimadzu model TOC-V CPH/CPN (Shimadzu Scientific Instruments Inc., Columbia, MD) to determine the concentrations of dissolved inorganic carbon (DIC) and dissolved organic carbon (DOC) in analytical samples. Five-point calibration curves for the analytical range between 1 and 100 mg/L were generated for both DIC and non-purgeable DOC analyses. The value for a minimum acceptable standard curve correlation coefficient was set at 0.995. An analytical blank and a calibration check standard at approximately 1 mg/L were measured after every ten analytical solutions and the results compared to acceptable QA/QC criteria (i.e., the recovery of calibration checks were required to be 85-115% and concentrations in analytical blanks were required to be non-detectable).

Quality Assurance/Quality Control

A series of QA/QC assessments, including method and analytical blanks, spikes, duplicates, and detection limits evaluations, were used to ensure that chemical analysis of eluate solutions were of high quality and that test results were not influenced by external sources of target analytes. Data quality indicators (DQIs), or the applicable criteria established in the project QAPP for ensuring adequate data quality, were reviewed and accepted by US EPA ORD prior to the start of characterization.

Chemical Analysis

For each analyte, the method of chemical analysis, method detection limit (MDL), and minimum level of quantitation (ML) are presented in Table 1. MDL and ML values were determined by VU following guidance in the *Technical Support Document for the Assessment of Detection and Quantitation Approaches* (US EPA, 2003). These values were used while assessing the quality control of eluate concentrations as well as in the interpretation and reporting of results. Concentrations measured at values less than the MDL were reported as “non-detected” in tabular formats, but were set to a value of ½ the MDL value for plotting and data analysis. Concentrations measured at less than the ML but greater than the MDL were reported as “estimated values” and were maintained as measured for plotting and data analysis.

Method Blank Analysis

Each of the LEAF methods includes requirements for the preparation and collection of method blanks which are analytical samples used to assess the potential for interferences in the test results due to contamination from reagent sources or equipment surfaces. Method blanks typically are extractions conducted using the same procedure as the test method, but excluding the solid test material. For Method 1314 where the only reagent is deionized water or 1mM CaCl₂ solution used as an eluant, the method requires that a sample of the eluant is collected as a method blank. For Method 1313 where test reagents include aliquots of acid and base added to deionized water, the three specified method blanks include samples of (i) deionized water, (ii) the extraction fluid at the highest level of acid addition and (iii) the extraction fluid at the highest level of base addition. Using an algorithm developed for validation of the LEAF test methods, the analyte concentration result of a eluate solution (i.e., Method 1313 extraction or Method 1314 eluent fraction) is considered to be influenced by external sources of contaminants if the analyte concentration in the method blank associated with the eluate solution has a valid detectable concentration greater than 20% of the concentration in the eluate solution (Garraabrants et al., 2012a; 2012b). Method blanks with analyte concentrations below the method limit of quantitation are considered not to influence eluate concentrations.

Environmental Reference Concentrations and Indicator Ratios

In order to place leaching test results into context with environmental reference concentrations, the results of leach testing (i.e., eluate concentrations for Method 1313 and Method 1314) were compared to the set of environmental reference concentrations shown in Table 1. The appropriate sources of environmental reference concentrations designated by US EPA Region 2 were the US EPA Region 9 residential tapwater RSLs

and the NDWRs. The NDWRs consist of a combination of primary maximum contaminant levels (MCLs), and treatment technology action levels, which are enforceable regulatory standards, and secondary MCLs and drinking water equivalent levels (DWELs) which are guidance. Based on guidance from US EPA Region 2, From these two sources of reference concentrations, a final set of environmental reference concentrations for comparison with leaching test results was compiled from these two sources by selecting the lower of the two environmental reference concentrations (RSL and NDWR) for each analyte.³ The selection methodology and final set of environmental reference concentrations were reviewed and approved by US EPA Region 2 prior to use in this project (Grossman, personal communication, 20 July 2012). Indicator ratios were calculated as the maximum eluate concentration from leaching test results (i.e., the maximum concentration within the applicable pH range of 6.5-11.5 or the maximum concentration within the range of L/S-dependent leaching) divided by the selected environmental reference concentration for each analyte.

³ The NDWRs and RSLs were developed for different purposes and so the values for any constituent can differ substantially in some cases. The chromium and arsenic MCL values in particular are several orders of magnitude above the RSLs. As enforceable drinking water standards, MCLs result from rulemaking that considers not only risk, but the feasibility of the regulation on a national basis. The risks corresponding to MCLs may therefore range up to an estimated cancer risk of one per 10,000 and occasionally somewhat higher. MCLs also may not reflect the very latest science, as rulemaking is expensive and time consuming, and so MCLs are not updated as easily or frequently as RSLs. RSLs are non-regulatory guidance values used in assessing Superfund sites, and uniformly represent cancers risks of one per million, or a Hazard Quotient of one for non-carcinogens (G. Helms, personal communication, 1 November 2012).

Table 1. Analytical Methods with MDLs and MLs Compared to Environmental Reference Concentrations. The final set of Environmental Reference Concentrations used for comparison to leaching tests results is indicated in bold.

Analyte	Symbol	Environmental Reference Concentrations		Analytical Method	MDL (µg/L)	ML (µg/L)
		NDWR (µg/L)	RSLs (µg/L)			
Aluminum	Al	50-200 ^{N1}	16,000	ICP-OES	1	5
Antimony	Sb	6	6	ICP-MS	0.08	0.2
Arsenic*	As	10	0.045	ICP-MS	0.64	2
Barium	Ba	2,000	2,900	ICP-OES	1	5
Beryllium	Be	4	16	ICP-MS	0.64	2
Boron	B	7 ^{N2}	3,100	ICP-OES	1	5
Bromide	Br			IC	17	50
Cadmium	Cd	5	6.9	ICP-MS	0.17	0.5
Calcium	Ca			ICP-OES	2.6	10
Cesium	Cs			ICP-MS	0.49	2
Chloride	Cl	250,000 ^{N1}	1,600	IC	6.5	20
Chromium*	Cr	100	0.031 ^{R1}	ICP-MS	0.5	2
Cobalt	Co		4.7	ICP-OES	1.8	5
Copper	Cu	1,300 ^{N3}	620	ICP-OES	3.7	10
Dissolved Inorganic Carbon	DIC			TOC	130	500
Dissolved Organic Carbon	DOC			TOC	170	500
Fluoride	F	4,000	620	IC	7	20
Iron	Fe	300 ^{N1}	11,000	ICP-OES	2	10
Lead	Pb	15 ^{N3}		ICP-MS	0.23	1
Lithium	Li		31	ICP-OES	1.9	10
Magnesium	Mg			ICP-OES	1.1	5
Manganese	Mn	1,600 ^{N2}	320	ICP-OES	2	10
Molybdenum	Mo	200 ^{N2}	78	ICP-OES	1.2	5
Nickel	Ni	700 ^{N2}	300	ICP-OES	1.8	5
Nitrate	NO ₃	10,000	25,000	IC	26	100
Nitrite	NO ₂	1,000	1,600	IC	18	50
Phosphate	PO ₄		760,000	IC	24	100
Phosphorus	P			ICP-OES	3.7	10
Potassium	K			ICP-OES	1.6	5
Selenium	Se	50	78	ICP-MS	0.52	2
Silicon	Si			ICP-OES	1.1	5
Sodium	Na			ICP-OES	2.5	10
Strontium	Sr	20,000 ^{N2}	9,300	ICP-OES	1	5
Sulfate	SO ₄	250,000 ^{N1}		IC	21	100
Sulfur	S			ICP-OES	6.8	20
Thallium*	Tl	2	0.16	ICP-MS	0.51	2
Tin	Sn		9,300	ICP-MS	0.7	2
Titanium	Ti			ICP-OES	2	10
Uranium	U	30	47	ICP-MS	0.3	1
Vanadium	V		78	ICP-OES	1.5	5
Zinc	Zn	10 ^{N2}	4,700	ICP-OES	1	5

Notes: RSLs = Residential Tapwater Regional Screening Levels for US EPA Region 9 (US EPA, 2012b)

^{R1} Chromium RSL based on Cr(VI)

NDWRs = National Primary Drinking Water Regulations (US EPA, 2012a) unless noted:

^{N1} National Secondary Drinking Water Regulations - non-enforceable guideline

^{N2} Drinking Water Equivalent Level (DWEL)

^{N3} Treatment Technique Action Level

* MDL greater than minimum indicator value.

RESULTS AND DISCUSSION

In the following section, a brief presentation of pH- and L/S-dependent leaching results for selected analytes is shown to illustrate the general results presentation and compare testing results from Method 1313 and Method 1314. Full presentation of leaching results for all tests and analytes is provided in Appendix A.

Titration Curve and Eluate pH

Figure 1 shows the titration curve, i.e., eluate pH versus acid addition from Method 1313 (left) and the eluate pH recorded in test fractions of Method 1314 (right).

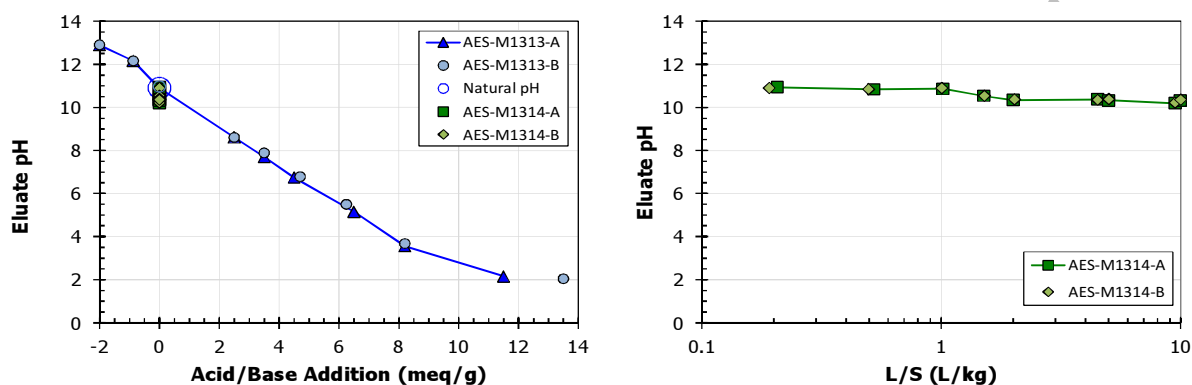


Figure 1. Eluate pH results for Method 1313 (left) and Method 1314 (right) testing of AGREMAX.

Each figure shows results for two test replicates (e.g., AES-M1313-A and AES-M1313-B) corresponding to the two material replicates. For the Method 1313 results, the natural pH of AES is 10.9 as indicated by the circled data in the Method 1313 figure for the pH response when no acid or base is added. The range of pH for Method 1314 eluates is 10.9 at low L/S to 10.2 at high L/S. The eluate pH range from the column test is consistent with the natural pH measured from the pH-dependent leaching test.

Eluate Concentrations as Functions of pH and L/S

The concentrations of arsenic, boron, cadmium and selenium resulting from testing AGREMAX using Method 1313 and Method 1314 are shown in Figure 2. Eluate concentrations are presented for both tests as a function of pH (left panel) and for only Method 1314 as a function of L/S (right panel). Plotting L/S-dependent data as a function of pH allows for comparison of eluate concentrations at low and high L/S values relative to the M1313 LSP curve. The scale of the vertical axes of the two panels has been coordinated so that the progression of L/S-dependent data from low to high L/S can be transferred to the pH-dependent leaching graph using the eluate concentrations values.

In the pH-dependent leaching panel (left) gold, dashed vertical lines represent the bounds of an applicable pH range for pH-dependent data. The vertical lines focus the assessment toward the range of potential pH values anticipated. In the case of AGREMAX, the applicable pH range was determined by US EPA Region 2 to be bounded at low pH by the minimum value in the NDWRs (i.e., pH 6.5) and at the upper pH by a pH value $\frac{1}{2}$ unit above the natural pH of the AES sample (i.e., pH 11.5).

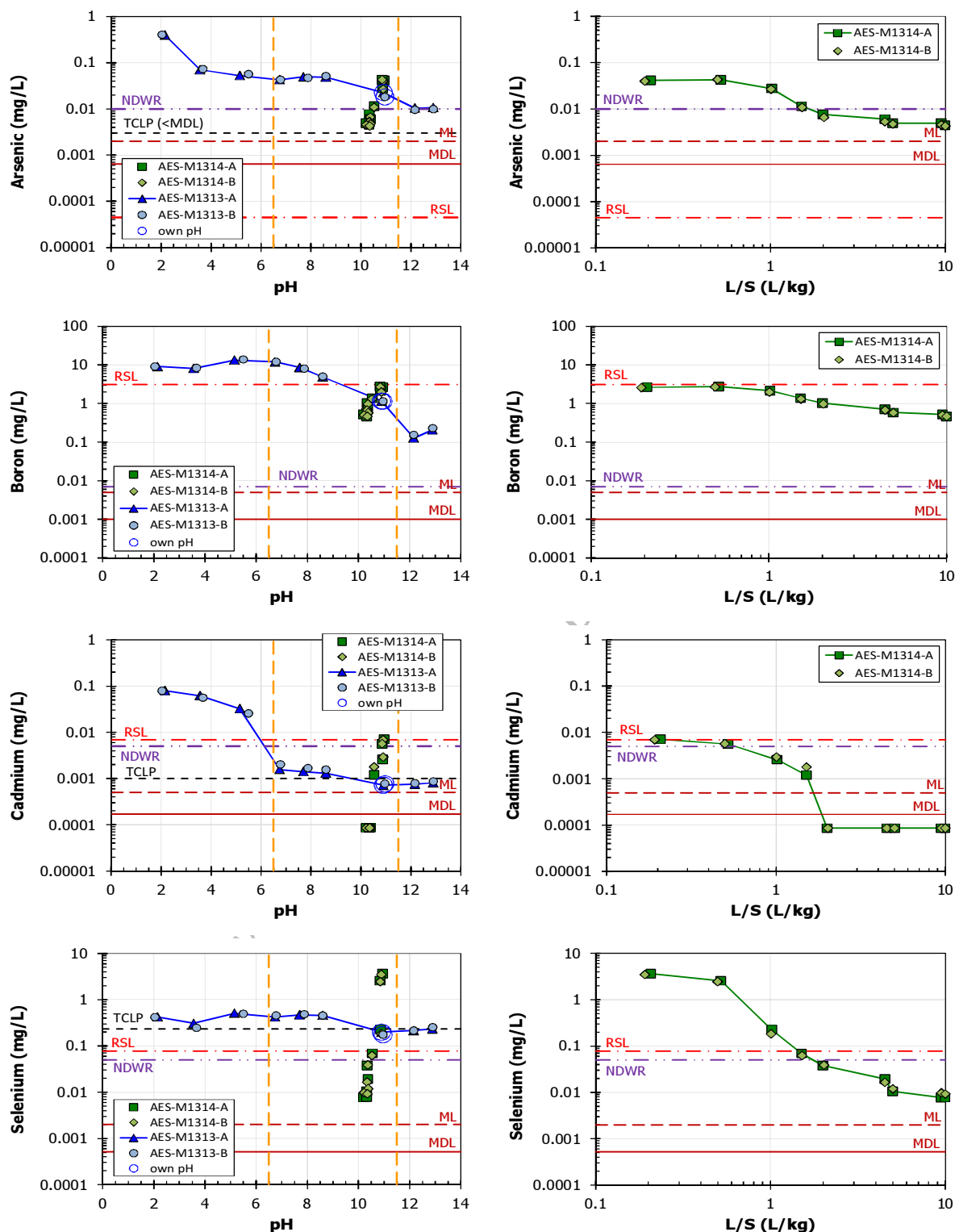


Figure 2. Eluate concentrations for arsenic boron cadmium and selenium from Method 1313 and Method 1314 testing of AGREMAX presented as functions of pH (left) and L/S (right) compared to NDWR and RSL values (the lower of each serving as the environmental reference concentration) shown as horizontal lines. TCLP values from earlier testing of AGREMAX are indicated as gray horizontal lines when available.

Also shown in each panel of Figure 2 are red and purple horizontal lines representing, respectively, the RSLs and NDWRs values presented in Table 1. In addition, TCLP concentration values published by AES Puerto Rico L.P. from independent testing of AGREMAX (AES, 2009) are shown as a black horizontal dashed line spanning the entire pH range.⁴

In Figure 2, the column test results (right) show that several analytes “washout” by a cumulative L/S value of 2 L/kg, resulting in eluate concentrations near or below quantitation limits (i.e., ML values). This washout effect is clearly illustrated for cadmium and to a lesser degree for arsenic and selenium. Method 1314 eluate concentrations measured at low L/S between 0.2 and 0.5 L/kg are commonly higher than Method 1313 eluate concentrations which are measured at L/S of 10 L/kg due to the influence of ionic strength on constituent solubility. When Method 1314 eluate concentrations are plotted as a function of pH and compared to Method 1313 data, the column data for more soluble species with strong L/S-dependence are shown to have a wide dynamic range (i.e., difference between high and low concentrations) with the trend line “cutting across” the pH-dependence curve. In Figure 2, strong L/S-dependence is illustrated for cadmium and selenium, while the L/S effect is less strong for arsenic and boron.

Data Quality Evaluation

Evaluation of data quality for this project include review of the analytical QC/QC relative to DQI goals established in the project QAPP and the assessment of potential interferences on low-level eluate concentrations through method blank analysis. A detailed analysis of analytic data quality is presented in Appendix B.

Analytical QA/QC Results

The established DQI goals for chemical analysis and the resulting DQIs from this project are shown in Table 2. The precision results are based on the overall mean relative standard deviation for parameters with more than two analytical replicates (i.e., ICP-OES, ICP-MS, IC, TOC analyses and moisture content). No precision value is provided for IC analysis as only a single analytical replicate is measured. Completeness was determined as the percentage of valid measurements (i.e., excluding analytical non-detects) meeting DQI goals out of the total number of measurements. The overall results show that the DQI goals established in the project QAPP were achieved for all eluate measurements in this study.

Table 2. Data Quality Indicator Goals and Overall Results.

Measurement	Method	DQI Goals		Overall Results	
		Precision	Completeness	Precision	Completeness
ICP-OES – cations	EPA Method 6010	±10%	>90%	1.9%	100%
ICP-MS – cations	EPA Method 6020	±10%	>90%	2.8%	100%
IC – anions	EPA Method 6056A	±10%	>90%	NA	100%
Carbon – DIC, DOC	EPA Method 9060	±10%	>90%	2.0	100%
pH, conductivity	Electrode	±2%	100%	2%	100%
moisture content	ASTM D2216-05	±10%	100%	0.2%	100%

Note: NA = “not available” - RSD as IC analysis is based on a single measurement.

⁴ TCLP does not require measurement and recording of the final extract pH (i.e., after tumbling). If, however, the final pH had been documented, the TCLP concentration values could be plotted as a function of pH which would provide better perspective on how TCLP data correspond to LEAF testing results.

In order to ensure that the analytical techniques accurately account for the target analytes in the analytical solution, matrix spikes (i.e., samples of the analytical solution with addition of a known mass of target analytes) were measured and the recovered spiked mass calculated. The criterion for acceptable spike recovery was that the mass recovered should be between 80 and 120% of the mass added to the sample. Table 3 presents a summary of the spike recovery results for each analytical technique (e.g., ICP-OES, ICP-MS, etc.) and leaching method (i.e., Method 1313 and Method 1314) as a percentage of the mass added to analytical sample. The table shows the mean spike recovery as well as the minimum and maximum spike recovery for all analytes measured by that analytical method. No matrix spikes were conducted for IC in accordance with the QAPP approved by the EPA QA personnel. All spike recoveries for ICP-OES, ICP-MS and carbon analyses were well within the 80 to 120% criteria.

Table 3. Analytical Matrix Spike Recovery Summary.

Measurement	Method	Method 1313			Method 1314		
		Mean	Min	Max	Mean	Min	Max
ICP-OES – cations	EPA Method 6010	99%	89%	115%	100%	85%	113%
ICP-MS – cations	EPA Method 6020	99%	94%	104%	98%	92%	102%
IC – anions	EPA Method 6056A	NA	NA	NA	NA	NA	NA
Carbon – DIC, DOC	EPA Method 9060	92%	91%	94%	93%	91%	96%

Note: NA = “not available” – Matrix spikes were not conducted for IC analysis.

Method Blank Analysis Results

Method blank analysis for both Method 1313 and Method 1314 showed that no method blanks had analyte concentrations above the ML values with the exception of those Method 1313 method blanks stemming from addition of acid or base to deionized water (e.g., potassium from KOH addition in the Method 1313 base method blank, or nitrate from HNO₃ addition in the Method 1313 acid method blank). Therefore, method blanks were not considered to influence eluate concentrations for any of the eluate concentrations from either leaching test.

Comparison of Leaching Results to Environmental Reference Concentrations

In order to facilitate the comparison of eluate concentrations to the selected environmental reference concentrations, the indicator ratio was defined as the maximum eluate concentration (i.e., the maximum concentration within the applicable pH range of 6.5-11.5 or the maximum concentration within the scope of L/S-dependent leaching) divided by the selected environmental reference concentration. The indicator ratio is intended to place leaching test eluate concentrations for various analytes into perspective. However, the indicator ratio does not consider any dilution or attenuation that may occur from the point of release (i.e., leaching source term) to the point of potential exposure or compliance.

The resulting indicator ratios for Method 1313 and Method 1314 test results (rounded to whole numbers) are shown in Table 4 for each analyte. The indicator ratios for arsenic, boron, chloride, chromium, fluoride (Method 1314), lithium (Method 1314), and molybdenum (Method 1314) for both leaching tests are greater than 100 as shown in red bold text with a thick red outline. Indicator ratios for aluminum (Method 1314), fluoride (Method 1313), lithium (Method 1313), molybdenum (Method 1313), nitrate (Method 1314), selenium, sulfate and thallium are between 10 and 100 as shown in orange bold text with a thin orange outline.

Table 4. Environmental Reference Concentrations, Maximum Leaching Test Eluate Concentrations, and Indicator Ratios for pH-dependent Leaching (Method 1313) and L/S-dependent Leaching (Method 1314). Ratios between 10 and 100 are shown in bold orange while ratios greater than 100 are shown in bold red.

Analyte	Symbol	Environmental Reference Conc. (mg/L)	Maximum M1313* (mg/L)	M1313 Indicator Ratio	Maximum M1314 (mg/L)	M1314 Indicator Ratio
Aluminum	Al	0.05	0.35	7	0.66	13
Antimony	Sb	0.006	0.053	9	0.019	3
Arsenic	As	0.000045	0.051	1,100	0.043	950
Barium	Ba	2	2.1	1	0.061	-
Beryllium	Be	0.004	0.00032	-	0.00032	-
Boron	B	0.007	12	1,700	2.7	390
Cadmium	Cd	0.005	0.0020	-	0.0071	1
Chloride	Cl	1.6	780	490	12,000	7,600
Chromium	Cr	0.000031	0.015	470	0.28	9,000
Cobalt	Co	0.0047	0.013	3	0.0064	1
Copper	Cu	0.62	0.0072	-	0.018	-
Fluoride	F	0.62	40	65	92	150
Iron	Fe	0.3	0.014	-	0.001	-
Lead	Pb	0.015	0.00012	-	0.0058	-
Lithium	Li	0.031	1.1	36	3.9	120
Manganese	Mn	0.32	0.61	2	0.001	-
Molybdenum	Mo	0.078	0.99	13	13	160
Nickel	Ni	0.3	0.081	-	0.027	-
Nitrate	NO ₃	10	41	4	560	56
Nitrite	NO ₂	1	0.009	-	0.009	-
Phosphate	PO ₄	760	0.012	-	0.012	-
Selenium	Se	0.05	0.51	10	3.6	73
Strontium	Sr	9.3	46	5	15	2
Sulfate	SO ₄	250	2,700	11	21,000	84
Thallium	Tl	0.00016	0.0050	31	0.0023	14
Tin	Sn	9.3	0.00035	-	0.0021	-
Uranium	U	0.03	0.059	2	0.0015	-
Vanadium	V	0.078	0.43	6	0.65	8
Zinc	Zn	0.01	0.0043	-	0.030	3

Notes: * Maximum M1313 concentration between pH 6.5 (lower range of NDWRs) and pH 11.5 (1/2 unit above natural pH)

"-" indicates that the ratio is less than 1 (i.e., the maximum LEAF test result was less than the environmental reference concentration)

SUMMARY

This document reports on the results of LEAF-based leaching assessment of AGREMAX sampled from the AES Puerto Rico L.P. coal-fired power plant in Guayama, Puerto Rico. AGREMAX is a partially solidified mixture of coal combustion fly ash and bottom ash. Subsamples of AGREMAX were collected and shipped to ARCADIS under chain of custody for testing. Homogenized samples of the solid material were subjected to pH-dependent leaching conducted by ARCADIS using Method 1313 and percolation column leaching conducted by VU using Method 1314. Test eluates were analyzed for a range of nearly 40 target analytes including major, minor and trace constituents. The leaching results for a selection of analytes were presented to illustrate how pH-dependence and percolation are related. In order to put eluate concentrations into perspective, the eluate concentrations from these leaching methods were compared to the minimum of indicator values derived from RSLs for tapwater or NDWRs.

Based on the above test results, the following observations were made regard the indicator ratios calculated as the maximum applicable eluate concentration from each leaching test⁵ divided by the selected environmental reference concentration:

pH-dependent Leaching

- Indicator Ratio greater than 100 for arsenic, boron, chloride and chromium, and
- Indicator Ratio between 10 and 100 for fluoride, lithium, molybdenum selenium, sulfate, and thallium,

L/S-dependent Leaching (maximum concentrations at L/S < 2 L/kg)

- Indicator Ratio greater than 100 for arsenic, boron, chloride, chromium, fluoride, lithium, and molybdenum, and
- Indicator Ratio between 10 and 100 for aluminum, nitrate, selenium, sulfate, and thallium.

Comparisons of the eluate concentrations to environmental reference concentrations are used to put the leachate concentrations from Method 1313 and Method 1314 into perspective with some regulatory and non-regulatory risk estimate values. The leaching test concentrations provided in this report do not consider dilution and attenuation factors that may be associated with different disposal or use scenarios. However, the LEAF method data can appropriately be used as a source term release estimate for groundwater fate and transport modeling that reflects the range of environmental conditions that AGREMAX is anticipated to encounter in the environment over time.

⁵ For Method 1313, maximum eluate concentration was determined within the applicable pH range established by US EPA Region 2 to be 6.5 to 11.5. For Method 1314, maximum eluate concentration was the determined to be the maximum concentration in any test fraction between L/S 0.2 and L/S 10 mL/g-dry.

REFERENCES

- AES (2009) "TCLP – Agremax Sep 15.09," AES Puerto Rico, LP, <http://www.agremax.com>, accessed 9/27/12.
- AES (2012) "Uses and Applications," AES Puerto Rico, LP, <http://www.agremax.com/#>, accessed 9/27/12, (in Spanish, translated to English using the online version of Babylon 9, <http://translation.babylon.com/spanish/to-english/>).
- ASTM (2009) D6323-98 "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities," ASTM International, West Conshohocken, PA.
- CDC (2003) Method 5040, Diesel Particulate Matter (as Elemental Carbon), Centers for Disease Control, NIOSH Manual of Analytical Method (NMAM), 4th edition, 2003.
- Garrabrants, A.C., D.S. Kosson, H.A. van der Sloot, F. Sanchez and O. Hjelm (2010) "Background Information for the Leaching Environmental Assessment Framework (LEAF) Test Methods," EPA-600/R-10/170, U.S. Environmental Protection Agency, Air Pollution Prevention and Control Division, December 2010.
- Garrabrants, A.C., D.S. Kosson, L. Stefanski, R. DeLapp, P.F.A.B. Seignette, H.A. van der Sloot, P. Kariher and M. Baldwin (2012a) "Interlaboratory Validation of the Leaching Environmental Assessment Framework (LEAF) Method 1313 and Method 1316," EPA-600/R-12/623, U.S. Environmental Protection Agency, Air Pollution Prevention and Control Division, September 2012.
- Garrabrants, A.C., D.S. Kosson, R. DeLapp, P. Kariher, P.F.A.B. Seignette, H.A. van der Sloot, L. Stefanski, and M. Baldwin (2012b) "Interlaboratory Validation of the Leaching Environmental Assessment Framework (LEAF) Method 1314 and Method 1315," EPA-600/R-12/624, U.S. Environmental Protection Agency, Air Pollution Prevention and Control Division, September 2012.
- Grossman, L. (US EPA Region 2), "Reference Value for LEAF Report," Email to S. Thorneloe (US EPA ORD), cc-ing N. Kraft and G. Meyer, 19 July 2012.
- Grossman, L. (US EPA Region 2), "Screening Values for Puerto Rico Leaching Tests," Email to S. Thorneloe (US EPA ORD), cc-ing N. Kraft, 20 July 2012.
- Helms, G. (US EPA Office of Solid Waste), "Footnote on Comparison of MCLs and RSLs," Edit submitted upon draft review to S. Thorneloe (US EPA ORD), 1 November 2012.
- Kosson, D.S., F. Sanchez, P. Kariher, L.H. Turner, R. DeLapp and P. Seignette (2009) "Characterization of Coal Combustion Residues from Electric Utilities – Leaching and Characterization Data," EPA-600/R-09/151, U.S. Environmental Protection Agency, Air Pollution Prevention and Control Division, December 2009.
- Rivera, R. (AES Corporation), "AGREMAX Information", Email to L. Grossman (US EPA Region 2), 16 June 2011.
- SAB (2003) Consultation Summary in *EPA Science Advisory Board, Environmental Engineering Committee Consultation with U.S. Environmental Protection Agency*, US EPA Science Advisory Board, Environmental Engineering Committee, Washington, DC (available as Attachment A of Sanchez et al., 2006).
- Sanchez, F., R. Keeney, D.S. Kosson and R. DeLapp (2006) "Characterization of Mercury-enriched Coal Combustion Residues from Electric Utilities using Enhanced Sorbents for Mercury Control," EPA-600/R-06/008, U.S. Environmental Protection Agency, Air Pollution Prevention and Control Division, February 2006.
- Sanchez, F., D.S. Kosson, R. Keeney, R. DeLapp, L. Turner and P. Kariher (2008) "Characterization of Coal Combustion Residues from Electric Utilities using Wet Scrubbers for Multi-pollutant Control," EPA-600/R-08/077, U.S. Environmental Protection Agency, Air Pollution Prevention and Control Division, July 2008.
- Thorneloe, S.A., D.S. Kosson, F. Sanchez, A.C. Garrabrants and G. Helms (2010) "Evaluating the fate of metals in air pollution control residues from coal-fired power plants," *Environmental Science and Technology*, 44(19), 7351-7356.
- US EPA (1996) SW-846 Method 3052 "Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. Environmental Protection Agency, <http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3052.pdf>.
- US EPA (1998) SW-846 Method 7473 "Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. Environmental Protection Agency, <http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/7473.pdf>.
- US EPA (2003) "Technical Support Document for the Assessment of Detection and Quantitation Approaches," U.S. Environmental Protection Agency, Office of Water, February 2003.

US EPA (2004) SW-846 Method 9060 "Total Organic Carbon," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. Environmental Protection Agency,
<http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/9060a.pdf>.

US EPA (2007a) SW-846 Method 3051 "Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. Environmental Protection Agency,
<http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3051.pdf>.

US EPA (2007b) SW-846 Method 6010 "Inductively Coupled Plasma-Atomic Emission Spectroscopy," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. Environmental Protection Agency,
<http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/6010c.pdf>.

US EPA (2007c) SW-846 Method 6020 "Inductively Coupled Plasma-Mass Spectroscopy," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. Environmental Protection Agency,
<http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/6020a.pdf>.

US EPA (2007d) SW-846 Method 9056 "Determination of Inorganic Anions by Ion Chromatography," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. Environmental Protection Agency,
<http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/9056a.pdf>.

US EPA (2010) MOP 5003.5 "Miscellaneous Operating Procedure 5003.5, Procedure for Phillips PW 2404 XRF Monthly Drift Correction, Quality Control Stability Check, and QC Chart Update, Revision 5," Internal EPA document located at L:\lab\NRML_Public\APPCD Facility Manuals ARCHIVED\X-ray\XRF Ops\XRF Quality System, December 2010.

US EPA (2012a) "2012 Edition of the Drinking Water Standard and Health Advisories", Office of Water - United States Environmental Protection Agency, EPA 822-S-12-001, Washington DC, April 2012.

US EPA (2012b) "Summary Table" Pacific Southwest, Region 9 Regional Screening Levels, updated May 2012,
<http://www.epa.gov/region9/superfund/prg/> (accessed 22 August 2012).

For Review Purposes Only - Do Not Cite

APPENDIX A. GRAPHICAL LEACHING TEST DATA

For Review Purposes Only - Do Not Quote or Cite

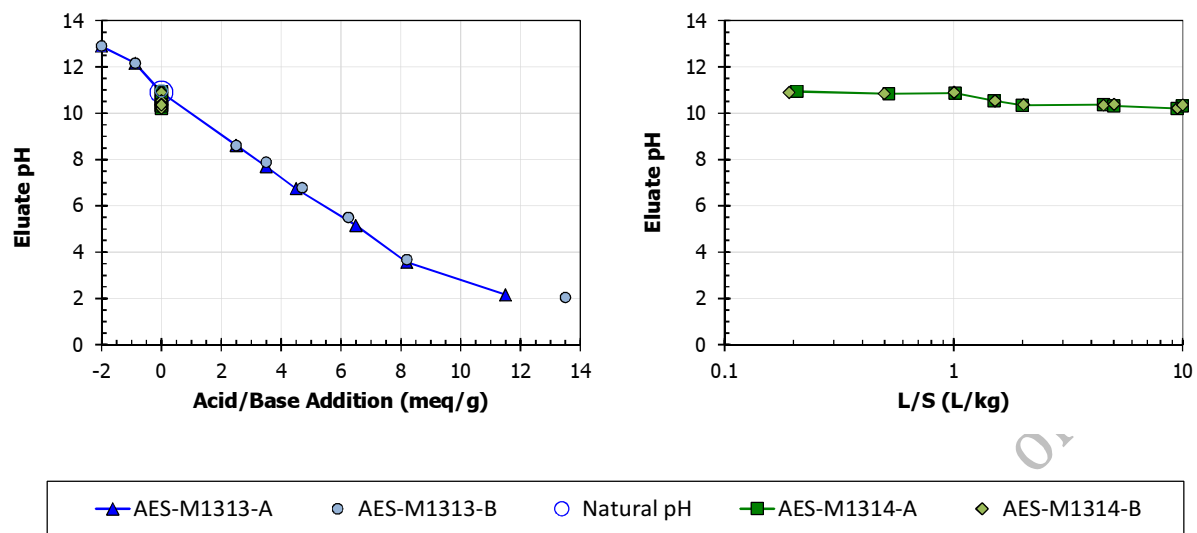


Figure A-1. Eluate pH measured for pH-dependence leaching (Method 1313) and percolation column leaching (Method 1314).

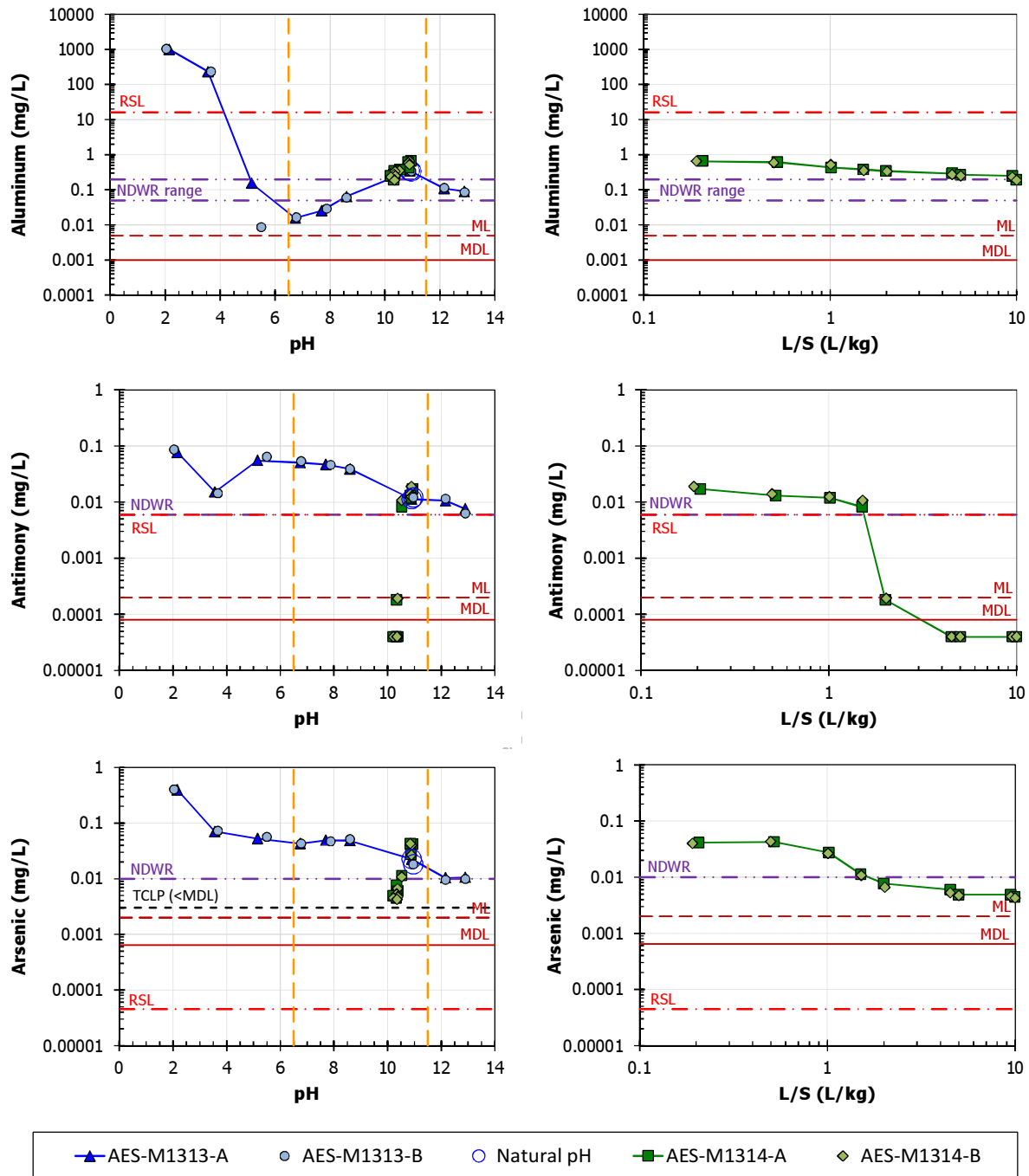


Figure A-2. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

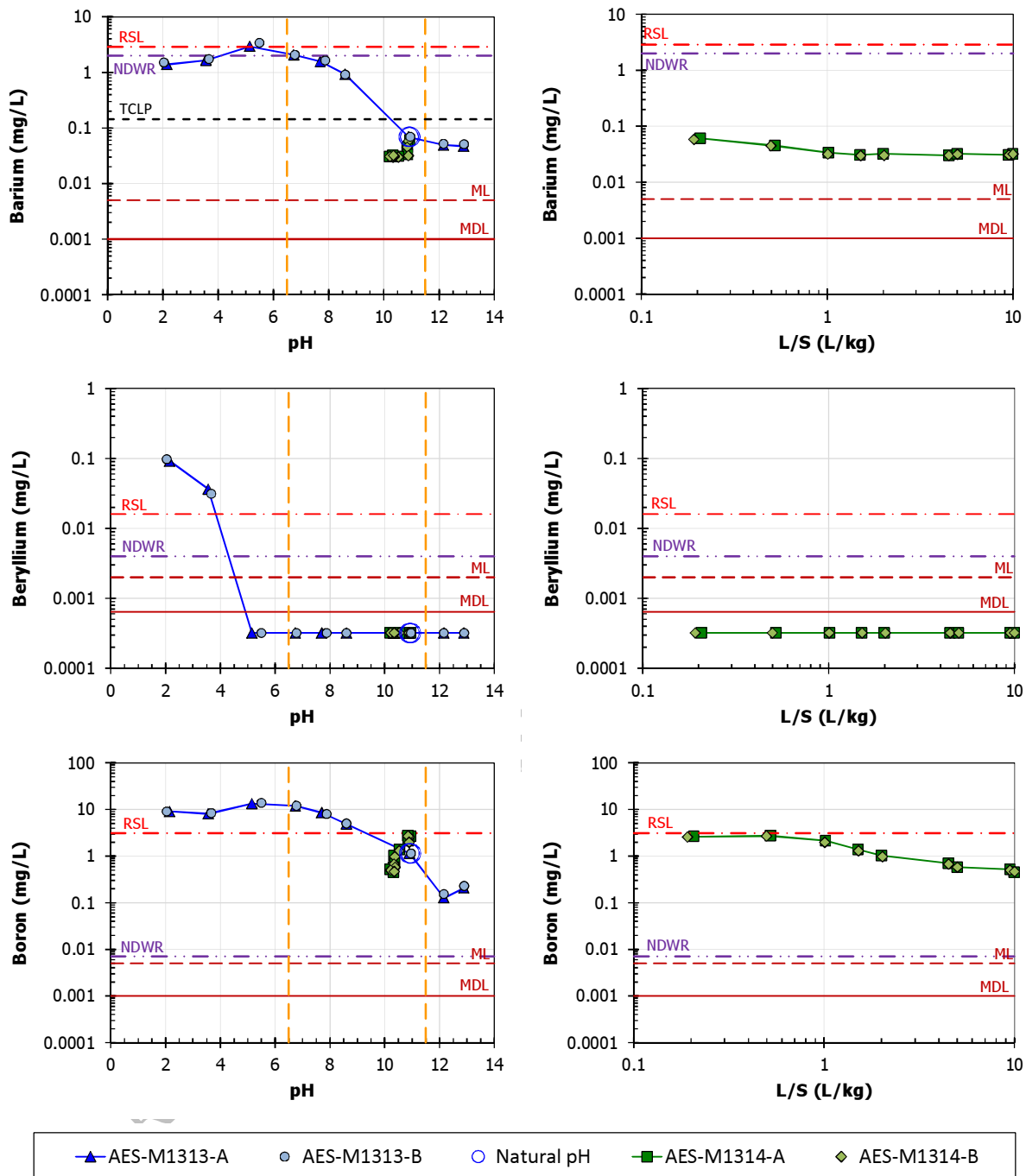


Figure A-3. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

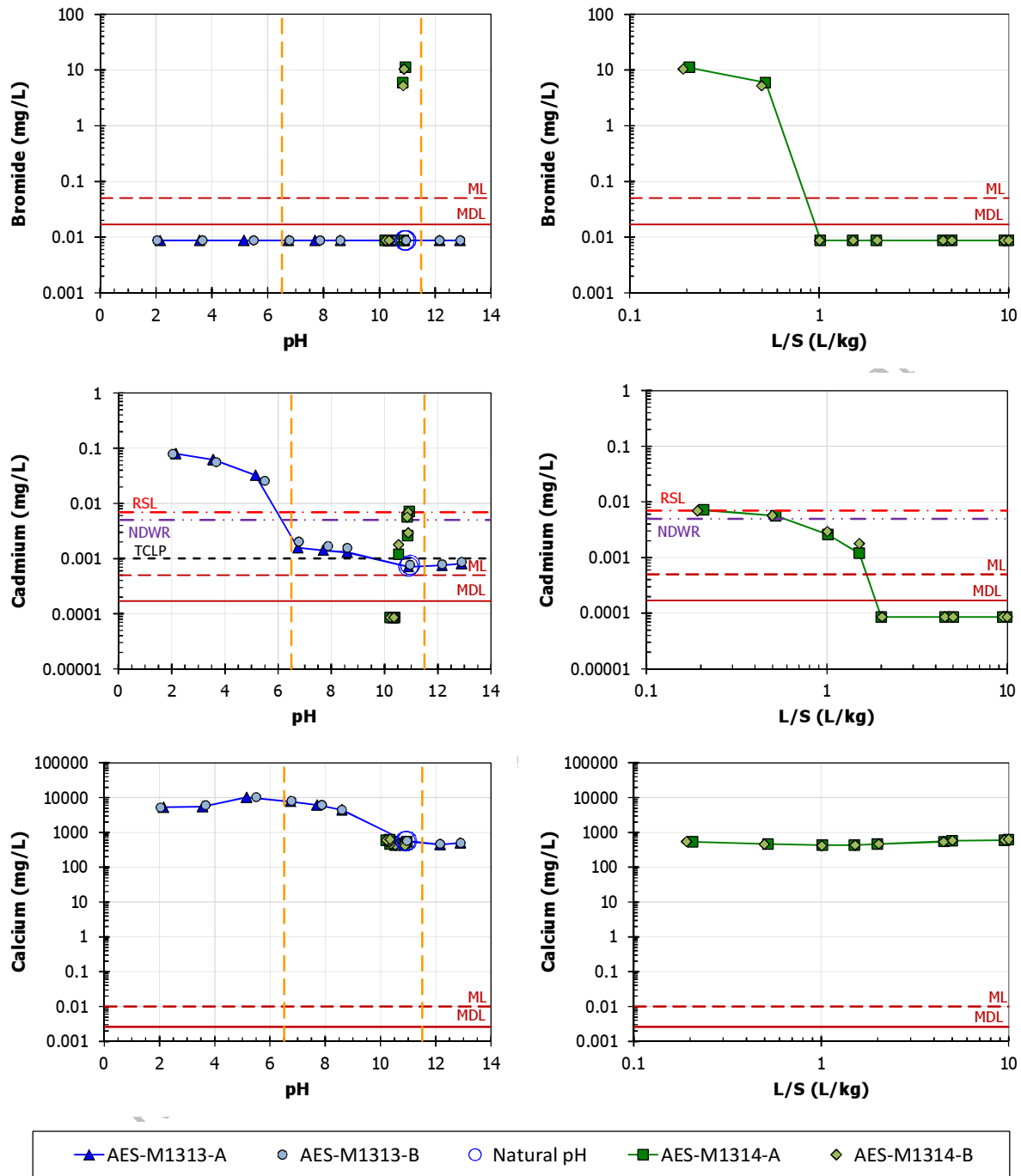


Figure A-4. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

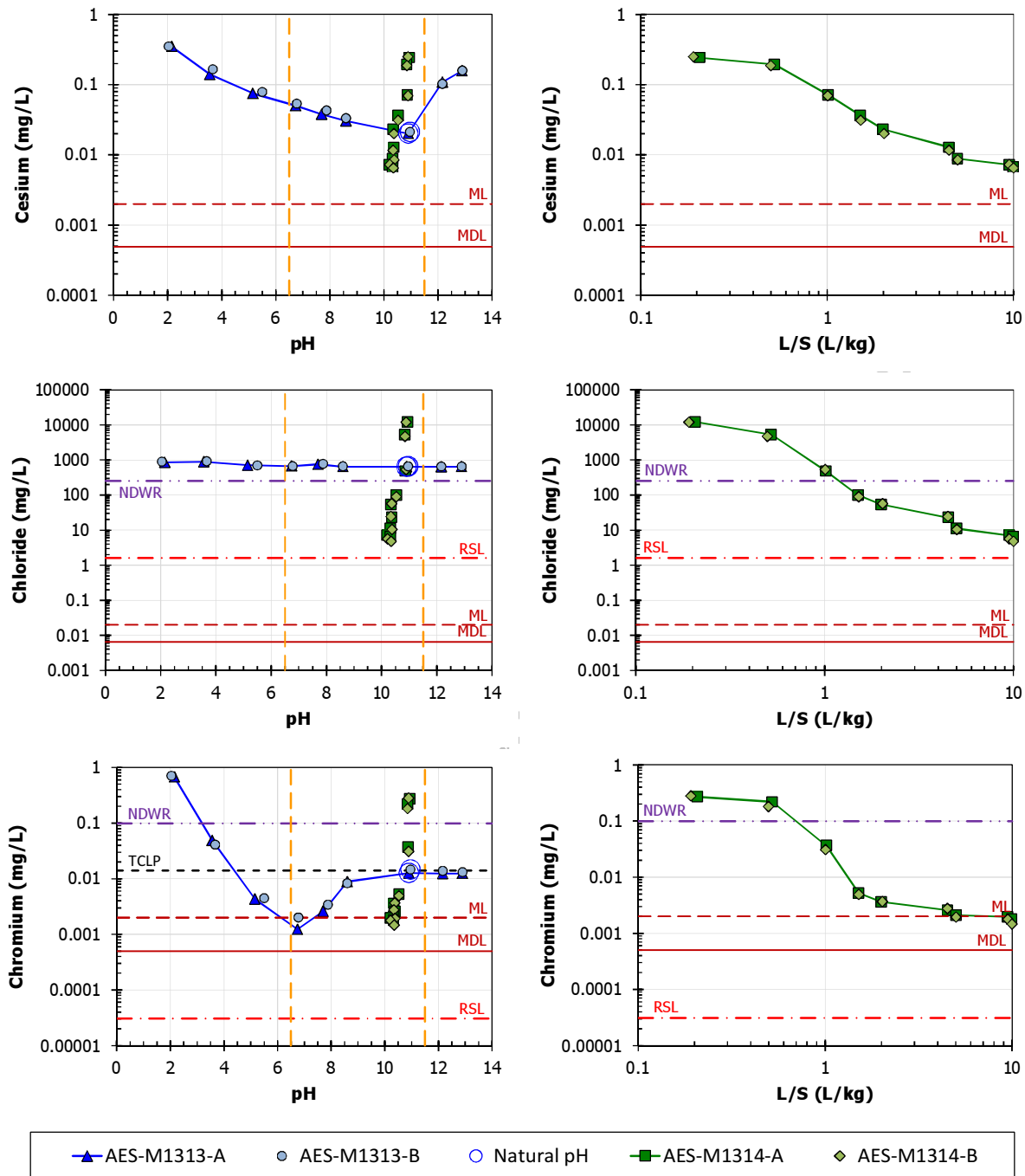


Figure A-5. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
 RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
 TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

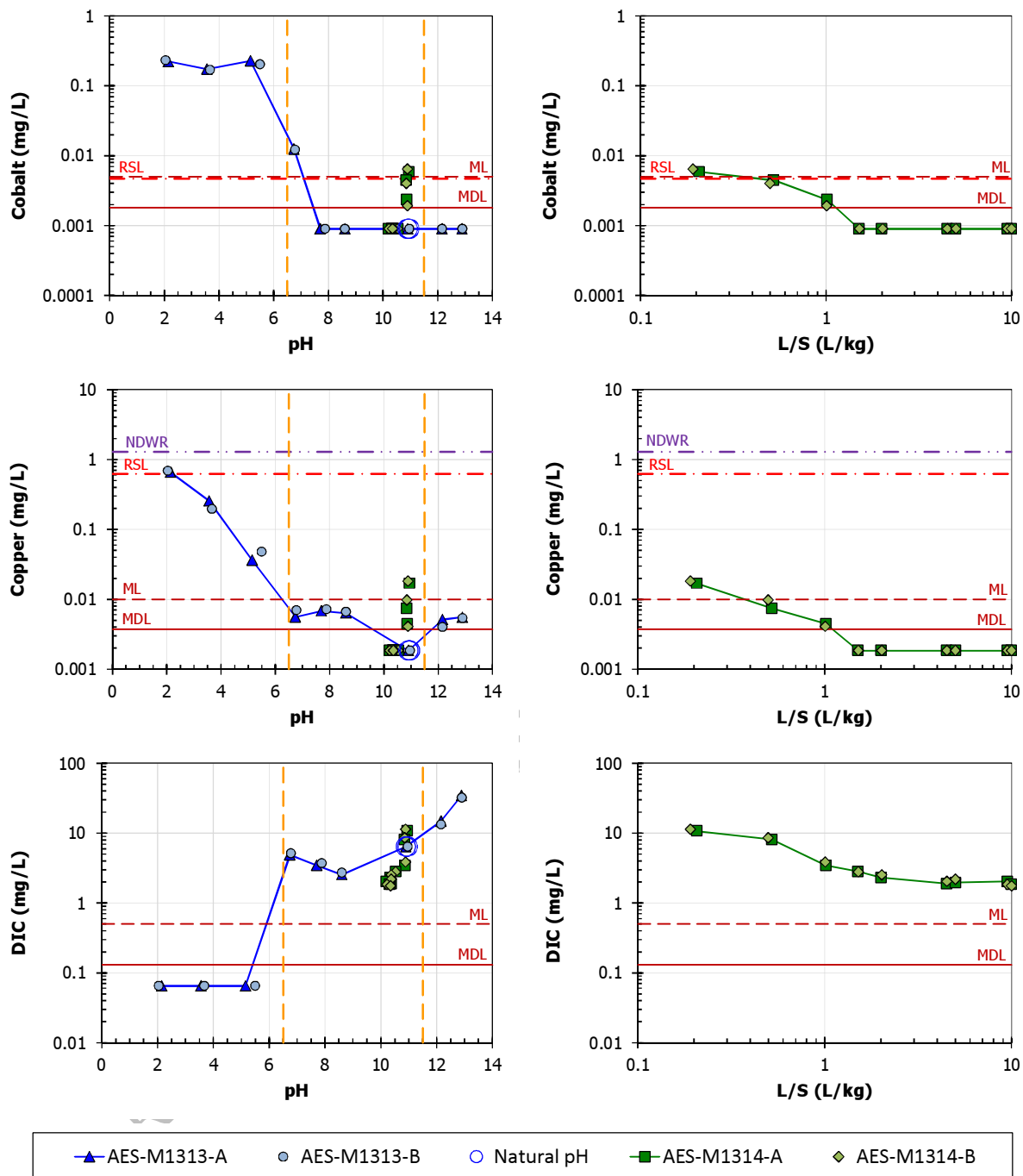


Figure A-6. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

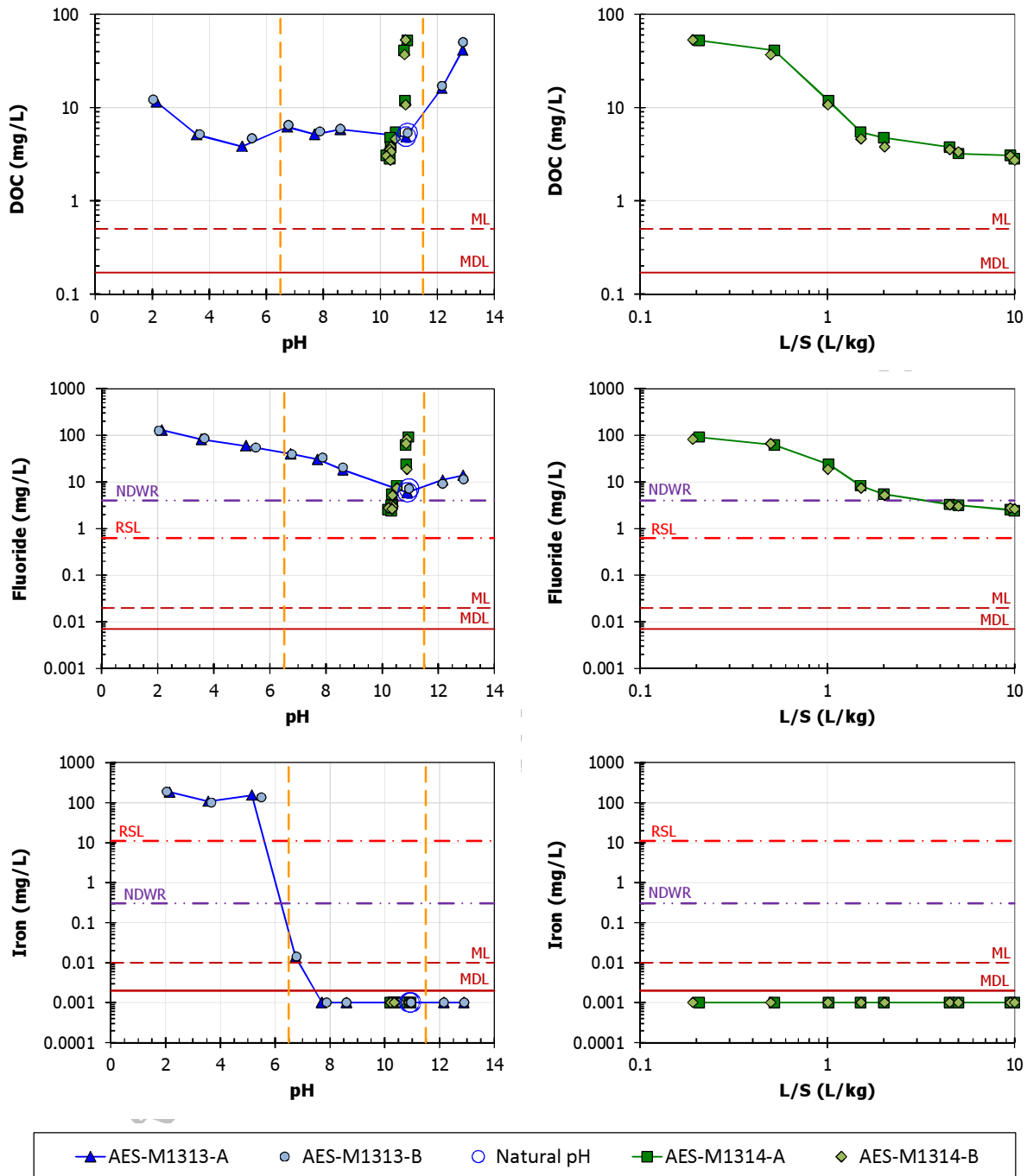


Figure A-7. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
 RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
 TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

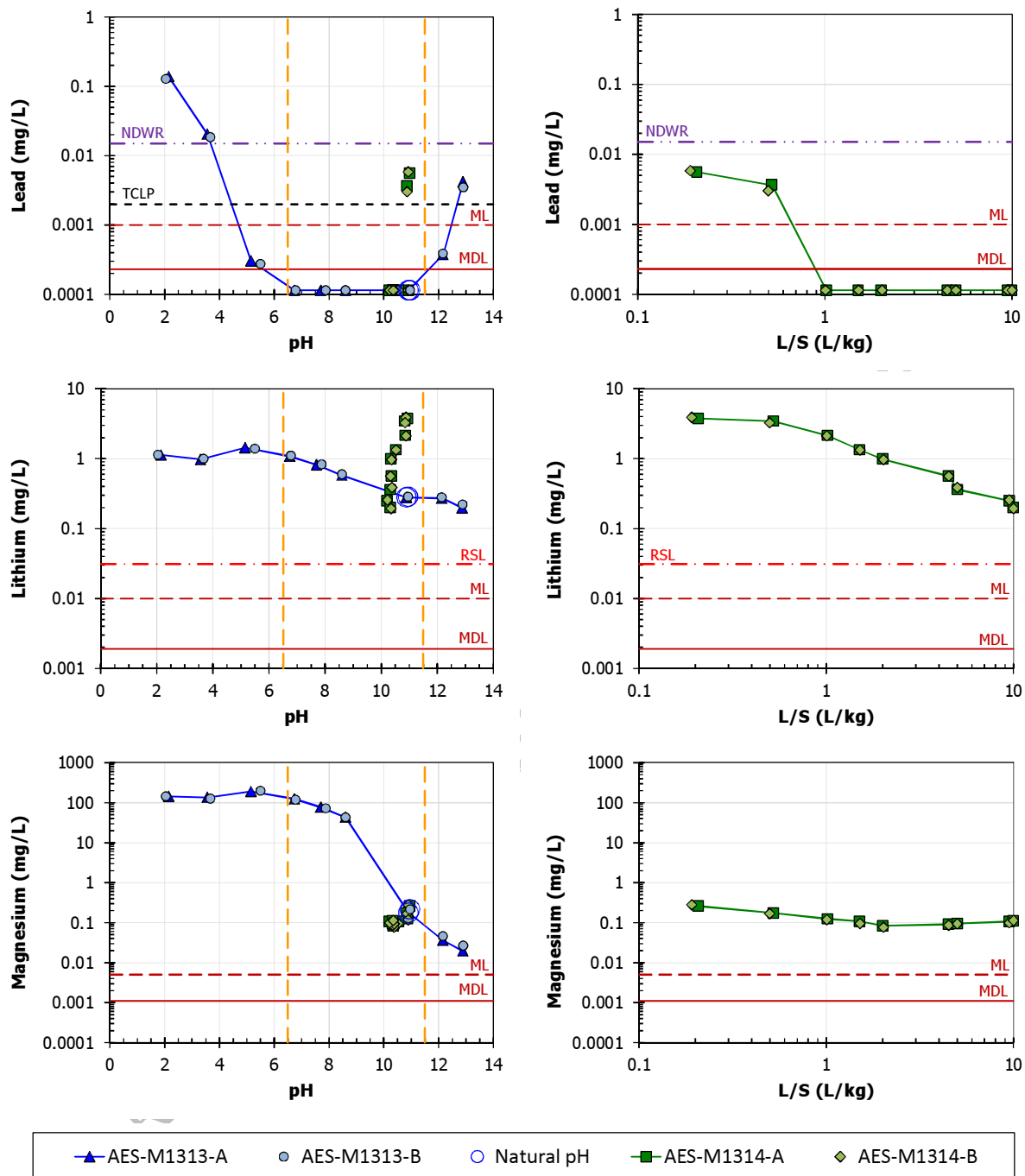


Figure A-8. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

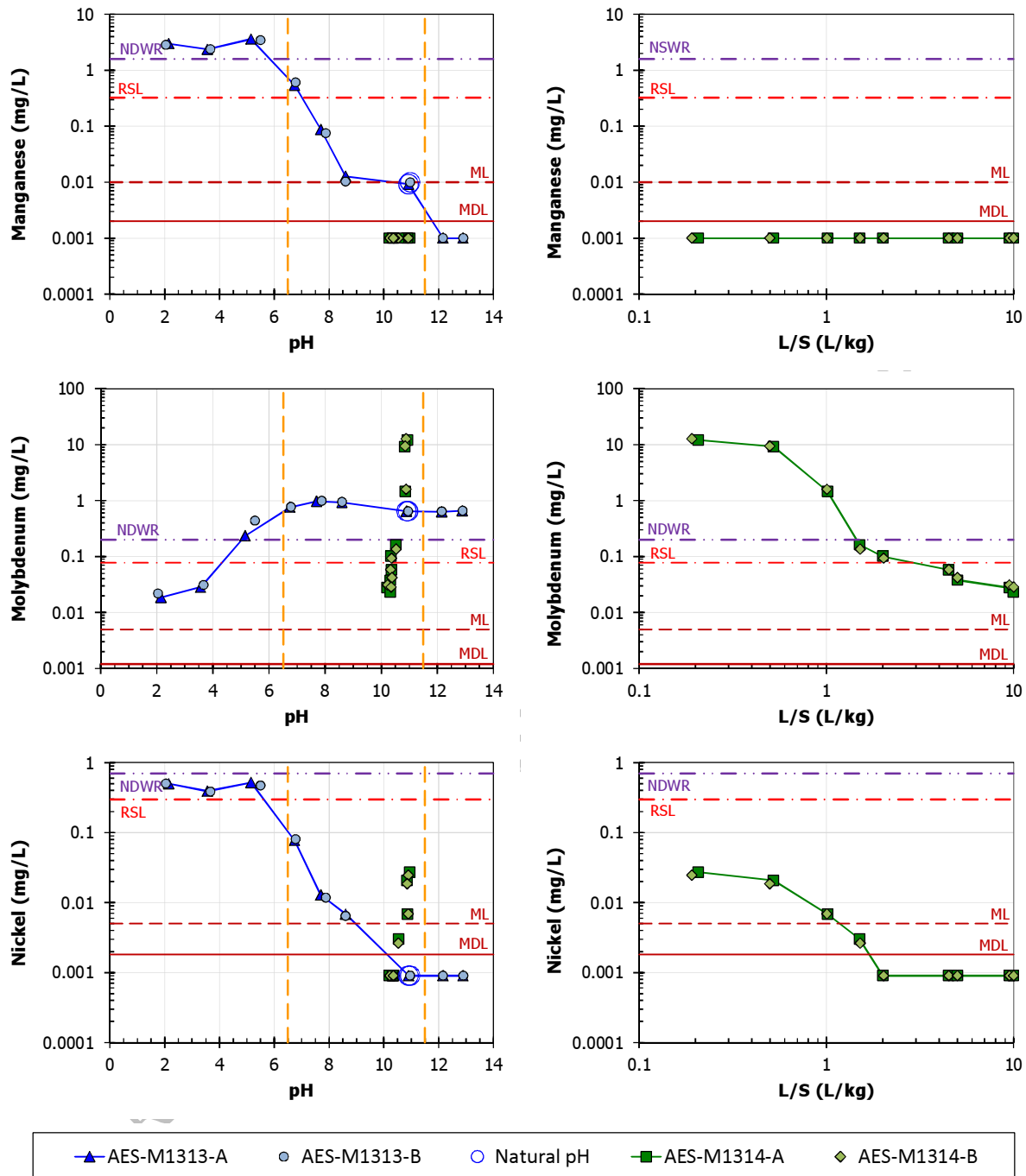


Figure A-9. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

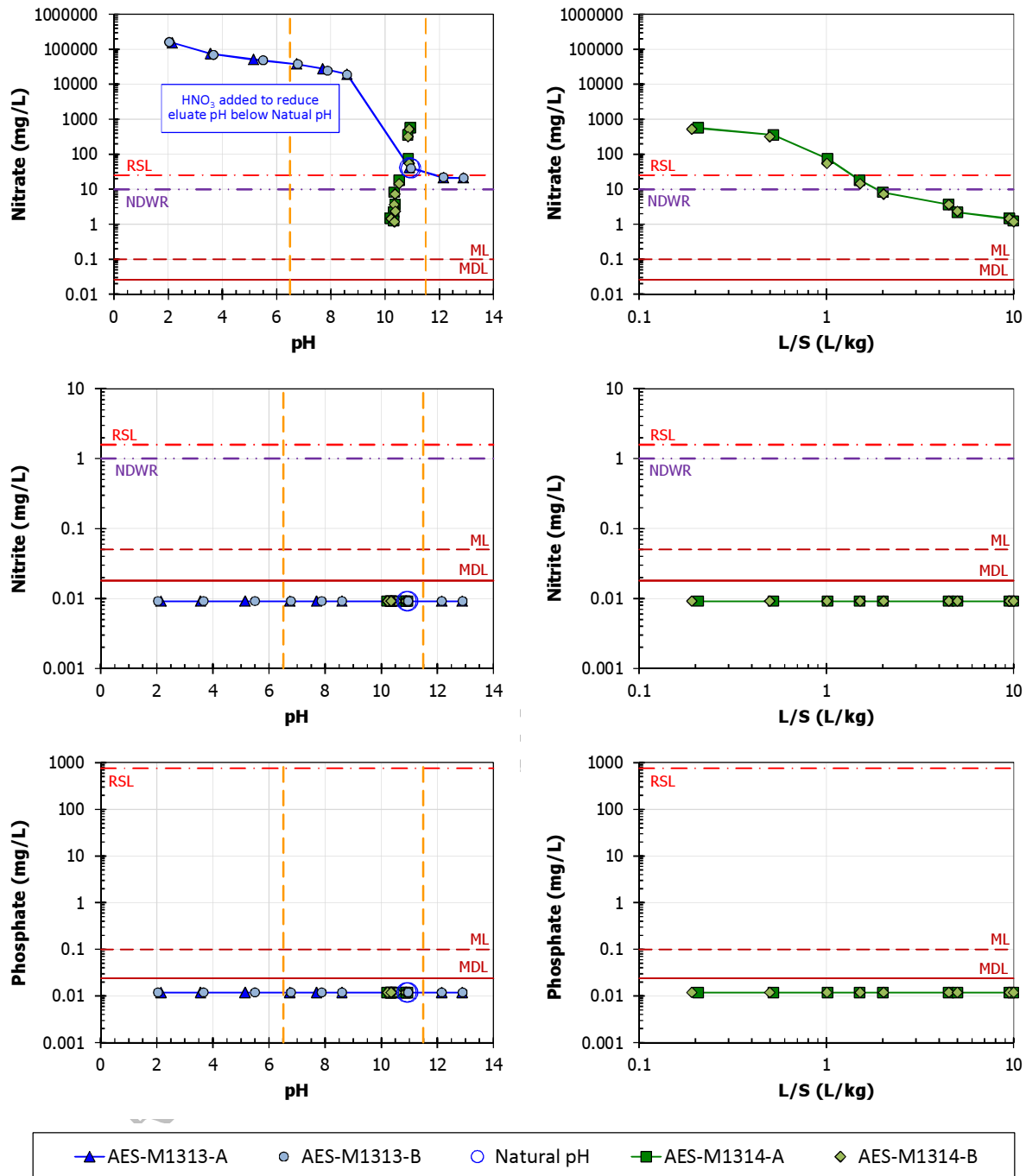


Figure A-10. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
 RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
 TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

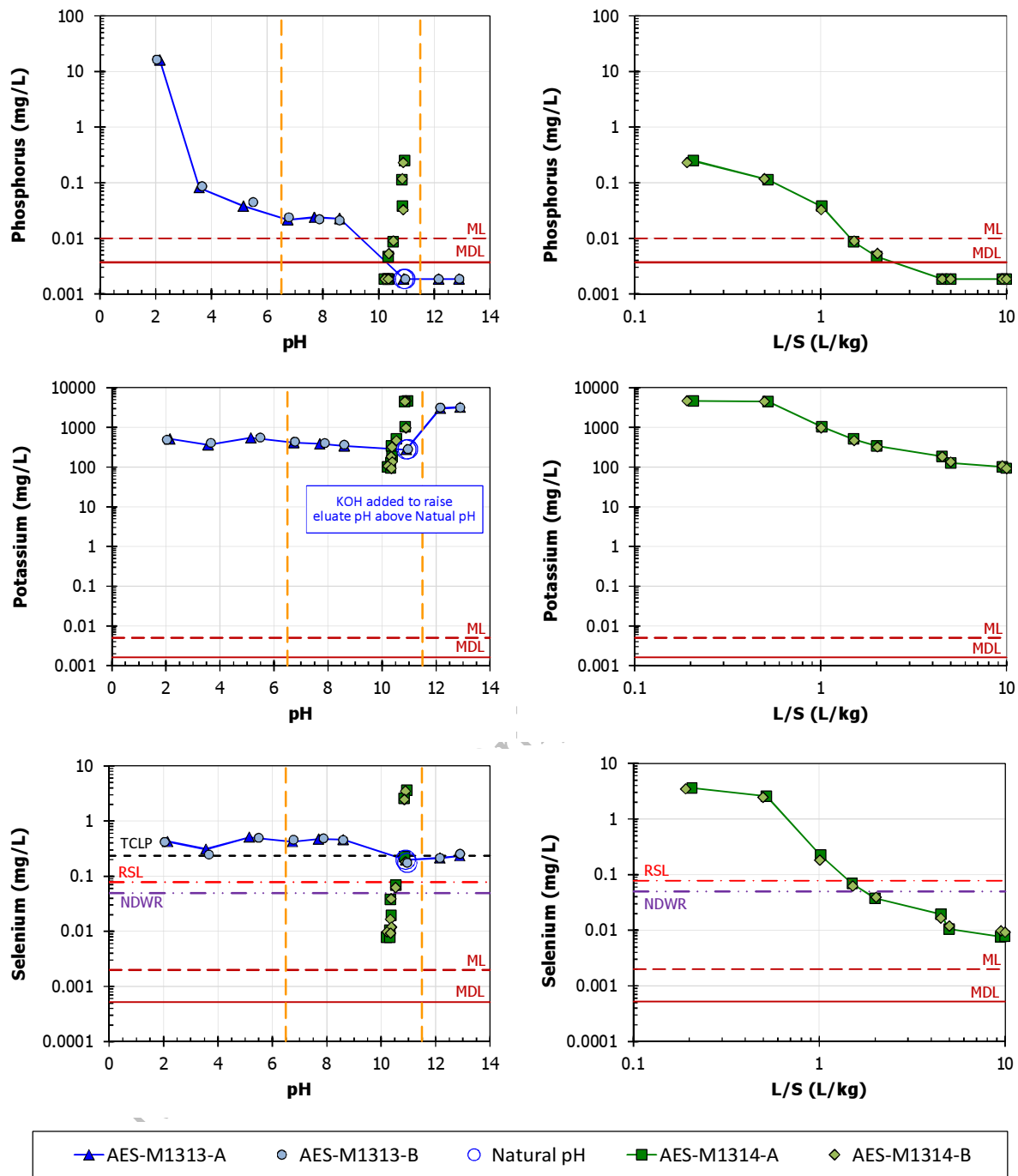


Figure A-11. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
 RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
 TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

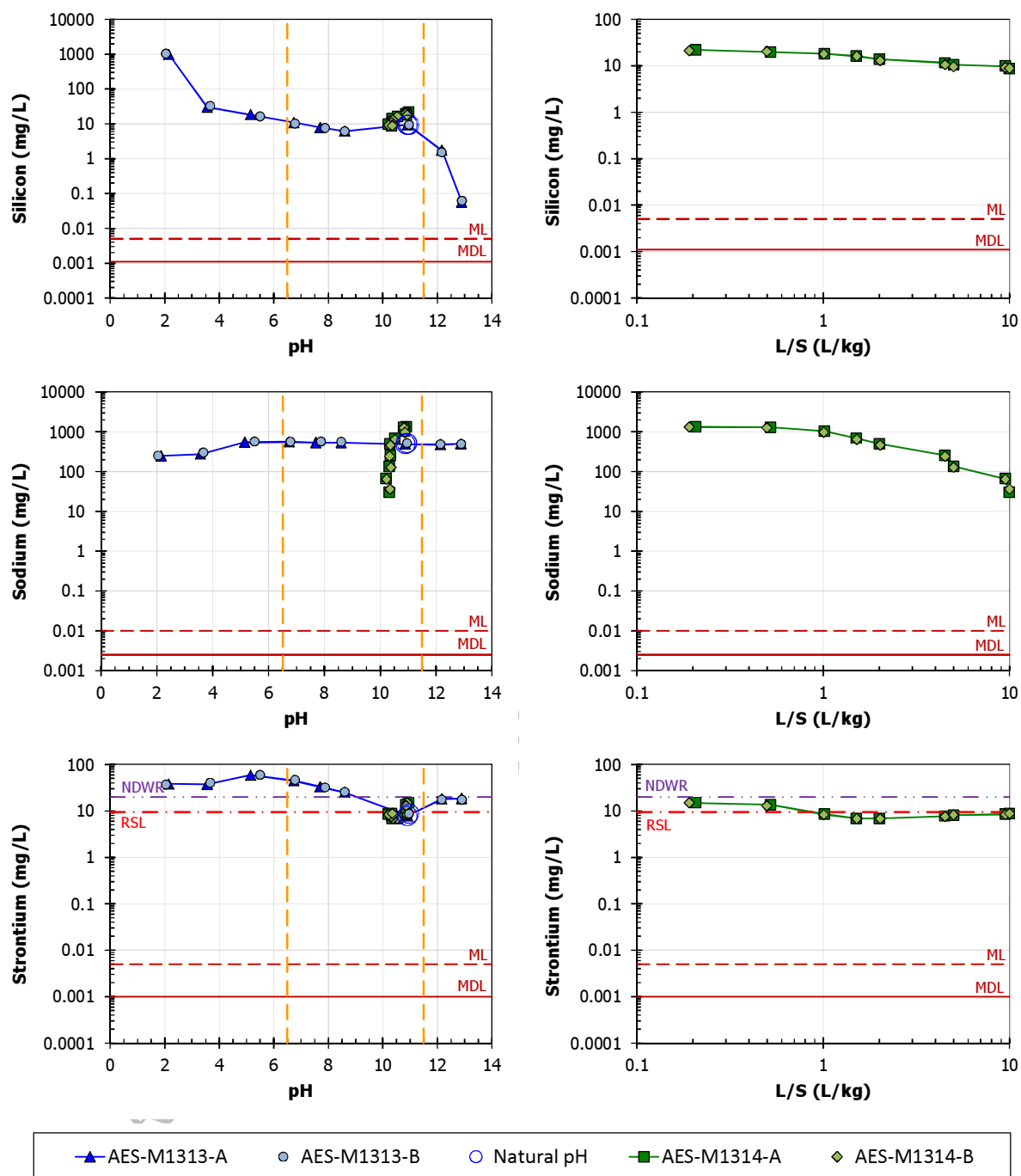


Figure A-12. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
 RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
 TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

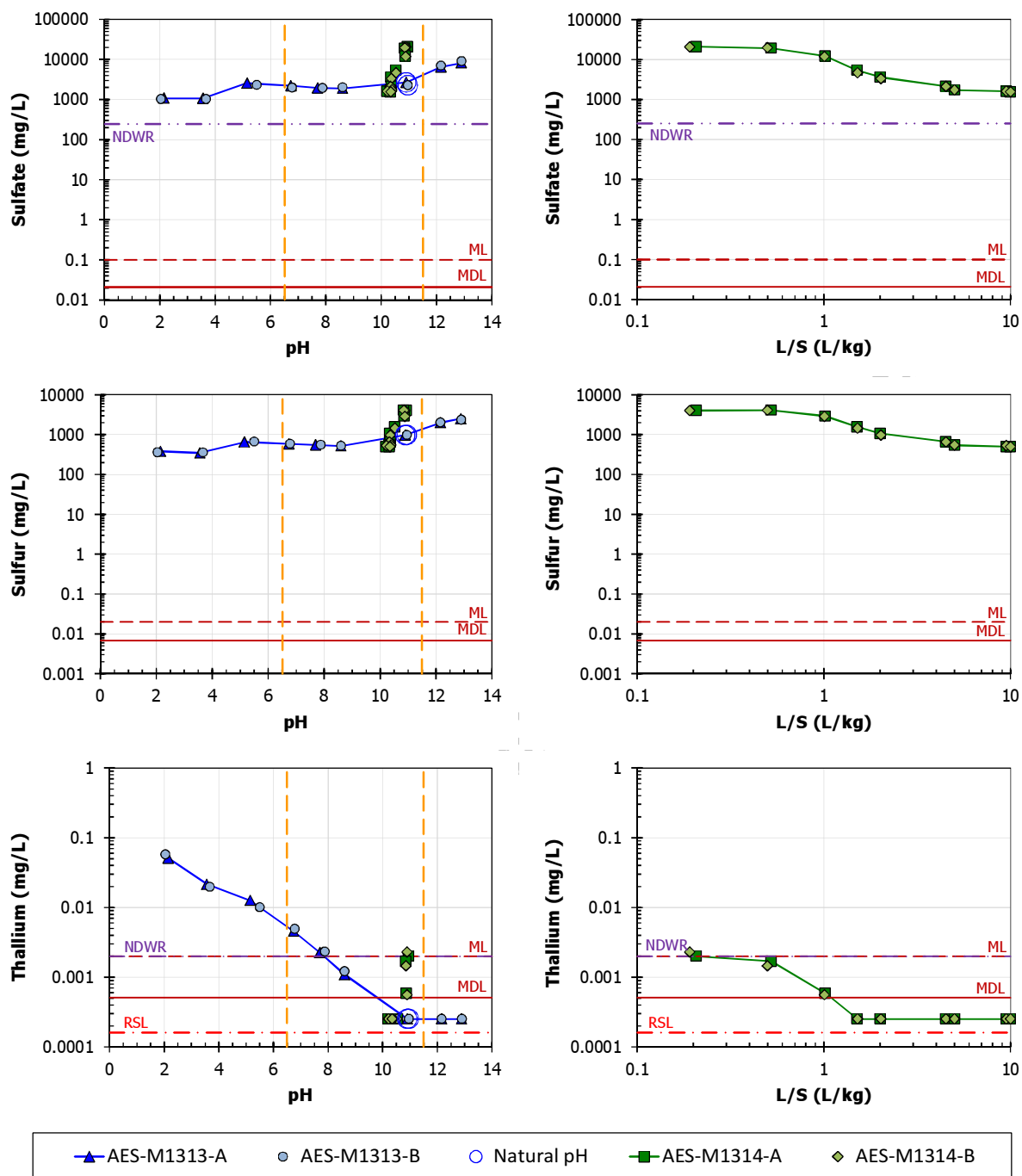


Figure A-13. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

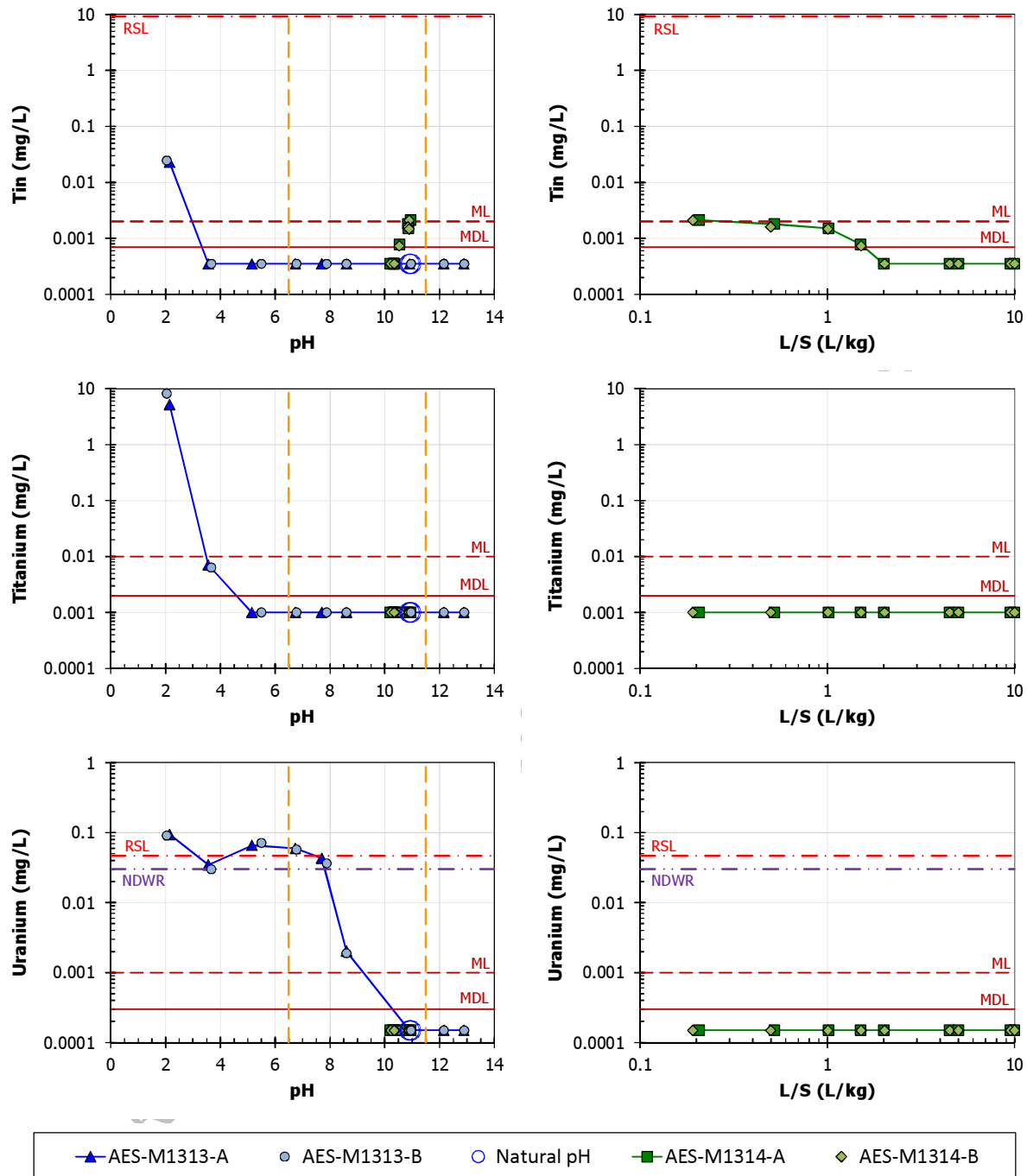


Figure A-14. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

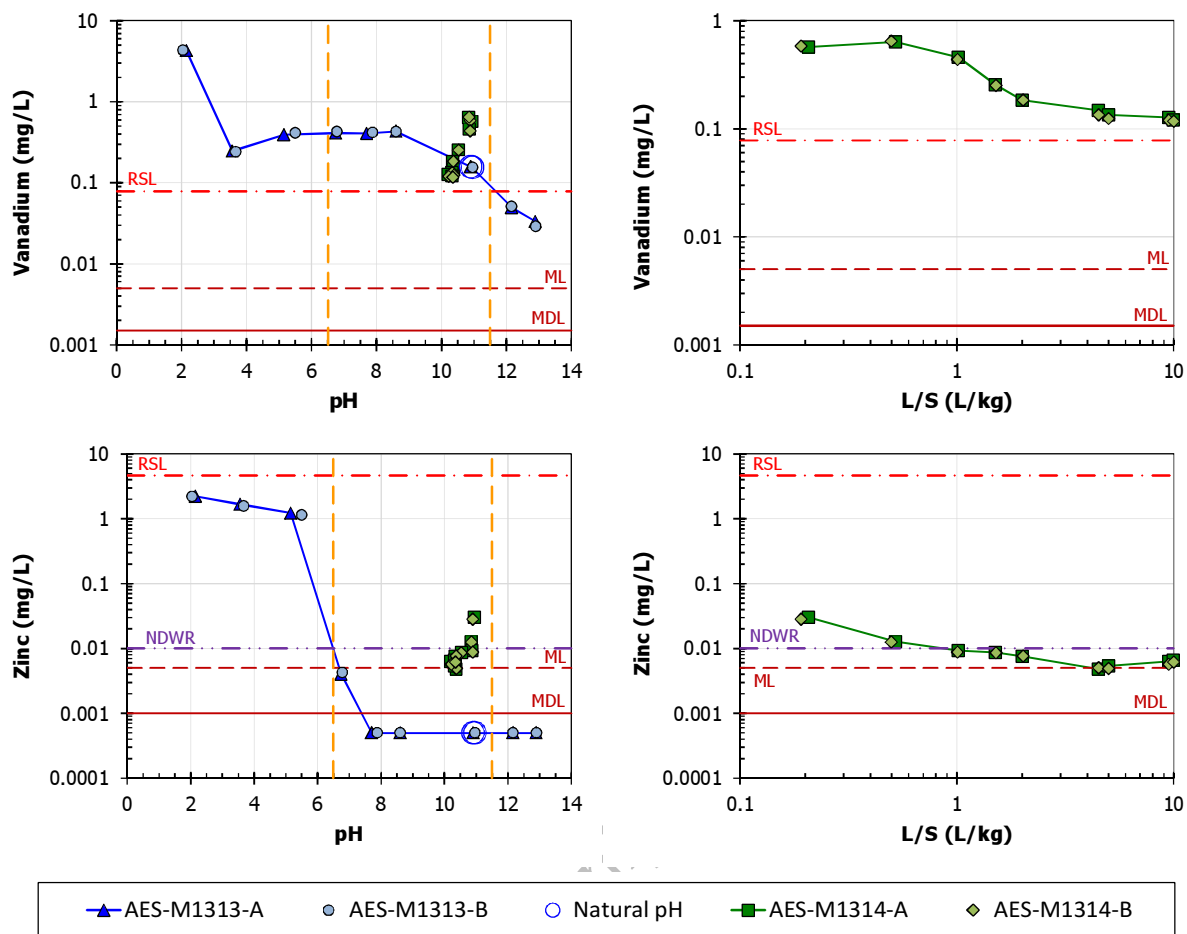


Figure A-15. Eluate concentration from pH-dependent leaching (Method 1313) and percolation column leaching (Method 1314) compared to indicator lines.

Notes: NDWR = National Drinking Water Regulations (US EPA, 2012a)
RSL = Residential Tapwater Regional Screening Levels for Region 9 (US EPA, 2012b)
TCLP = Toxicity Characteristic Leaching Procedure results from September 2009 (AES, 2009)

APPENDIX B. DETAILED QA/QC REPORT

TABLE OF CONTENTS	B-i
CHAIN OF CUSTODY FORMS	B-1
ANALYTICAL QA/QC.....	B-7
Introduction	B-7
Organization of the QA/QC Tables.....	B-7
Columns in the Analytical QA/QC Tables	B-7
Analytical QA/QC Report Summary	B-10
Method 1313 QA/QC Tables.....	B-14
Method 1314 QA/QC Tables.....	B-55

Solid Material Transport – AES Material from Region 2 to ARCADIS-US, Inc. (ARCADIS)

Page 1 of 1 pages

SURVEY NAME & LOCALITY PROGRAM: SF ☐ : Decision ☐ RCRA ☒ RCRA ENF ☐ NPDES ☐ SDWA ☐ AM ☐ CAA ☐ Unit Code Y206 D210 D307 B304 C215 B224 A305		PROJECT LEADER Bob Morrell PROGRAM RESULTS CODE _____ TSCA ☐ OD ☐ FIFRA ☐ CRIMINAL ENF ☐ L306 B253	
Permit #: LAB ID/ FIELD ID	CENTERS # OF MATRIX CHECK IF SPLIT SAMPLE DESCRIPTION & INSTRUCTIONS INCLUDING LOCATION, ESTIMATED CONCENTRATIONS, SPECIAL REPORTING LIMITS.	Res CL Checked Preservative (circle)	Collection Time (24hr clock) Begin End Collection Date mm/dd/yy
Manufactured Aggregate Composite	2 J 2 5-gallon plastic buckets for LEAF	☐ 012345678910 ☐ 012345678910 ☐ 012345678910 ☐ 012345678910 ☐ 012345678910 ☐ 012345678910 ☐ 012345678910 ☐ 012345678910 ☐ 012345678910 ☐ 012345678910	1145/1345 03/13/12
COMMENTS & SPECIAL REQUIREMENTS:		Preservative Added & Checked 0=ice 7=FAS 1=H2SO4 pH<2 8=ZnAc 2=HNO3 pH<2 9=NaOH pH>12 3=HCl pH<2 10=NH4Cl 4=Na2S2O3 5=NaOH pH>9 6=Ascorbic Acid	
Matrix: A=aqueous F=multiphasic B=aqueous (chlorinated) G=solvent C=soil H=biota D=sediment I=oil E=sludge J=other		Relinquished By: Relinquished By: Relinquished By:	Person Assuming Responsibility for Sample(s): Received By: Received By:
Survey Complete? Y ☒ N ☐		Time Date 1245 3/13/12 10:26 3-16-12	

B-1

[illegible]

Analytical Sample Transport – Method 1313 Analytical Samples from ARCADIS to VU

Arcadis Geraghty and Miller
4915 Prospectus Drive
Durham, NC 27713
(919) 544-4535

CHAIN OF CUSTODY RECORD

Project Description: Coal Combustion Residue Study
Arcadis G&M charge #: R0990273.0007.00001
Responsible EPA Party: Susan Thorneloe
Responsible Arcadis Party: Peter Kariher

Page 2 of 2

Sample ID	Date Collected	Sample Description	Number of Containers	Analyses Required
1313-AES PR-T01-B	04/23/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T02-B	04/23/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T03-B	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T04-B	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T05-B	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T06-B	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T07-B	03/29/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T08-B	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T09-B	04/02/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-B01-B	04/02/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-B02-B	04/02/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-B03-B	04/23/12	1313 pH/TOC sample 50mL	1	

Sample ID	Special Instructions
	All samples 1% nitric acid

Report results to: Peter Kariher

(phone) 919-541-5740

(fax) 919-544-5690

Samples shipped to: Rossane Delapp

Jacobs Hall Rm 162a

400 24th Ave South

Nashville, TN 37235

615-322-3189

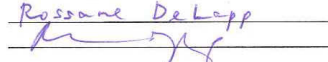
Samples collected by: Eric Morris
(signature) 

Date: 8/27/2012

Date shipped: 8/27/2012

Samples relinquished by: Eric Morris
(signature) 

Date: 8/27/2012

Samples received by: Rossane DeLapp
(signature) 

Date: 8/27/12

EC

Arcadis Geraghty and Miller

4915 Prospectus Drive
Durham, NC 27713
(919) 544-4535

CHAIN OF CUSTODY RECORD

Project Description: Coal Combustion Residue Study
Arcadis G&M charge #: RN990273.0007.00001
Responsible EPA Party: Susan Thorneioe
Responsible Arcadis Party: Peter Kariher

Page 1 of 2

Sample ID	Date Collected	Sample Description	Number of Containers	Analyses Required
1313-AES PR-T01A	04/23/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T02A	04/23/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T03A	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T04A	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T05A	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T06A	03/29/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T07A	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T08A	03/26/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-T09A	03/29/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-B01-A	04/02/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-B02-A	04/02/12	1313 pH/TOC sample 50mL	1	
1313-AES PR-B03-A	04/23/12	1313 pH/TOC sample 50mL	1	

Sample ID	Special Instructions

Report results to: Peter Kariher(phone) 919-541-5740(fax) 919-544-5690Samples shipped to: Rossane DelappJacobs Hall Rm 162a400 24th Ave SouthNashville, TN 37235615-322-3189Samples collected by: Eric Morris

(signature)

Date: 8/28/2012Date shipped: 8/28/2012Samples relinquished by: Eric Morris

(signature)

Date: 8/28/2012Samples received by: Rossane DeLapp

(signature)

Date: 8/29/12

For

4915 Prospectus Drive
Durham, NC 27713
(919) 544-4535

Project Description:	Coal Combustion Residue Study
Arcadis G&M charge #:	RN990273.0007.00001
Responsible EPA Party:	Susan Thorneloe
Responsible Arcadis Party:	Peter Karher

Page 1 of 2

Sample ID	Date Collected	Sample Description	Responsible Arcadis Party:	Number of Containers	Peter Karther	
					Analyses Required	
1313-AES PR-T01A	04/23/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-T02A	04/23/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-T03A	03/26/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-T04A	03/26/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-T05A	03/26/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-T06A	03/26/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-T07A	03/26/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-T08A	03/26/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-T09A	03/29/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-B01-A	04/02/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-B02-A	04/02/12	1313 pH/TOC sample	50mL	1		
1313-AES PR-B03-A	04/23/12	1313 pH/TOC sample	50mL	1		

[illegible]

Report results to: Peter Kariher

(phone) 919-541-5740

(fax) 919-544-5690

Samples shipped to: Rossane Delapp

Jacobs Hall Rm 162a

400 24th Ave South

Nashville, TN 37235

615-322-3189

Samples collected by:

Eric Morris

(signature)

Date: 8/28/2012

Date shipped:

8/28/2012

Samples relinquished by:

Eric Morris

(signature)

Date: 8/28/2012

Samples received by:

Rossane De La po

(signature)

Date: 8/29/12

For

ANALYTICAL QA/QC

Introduction

The tables in this appendix present the quality assurance/quality control results for the analytical measurement of eluate concentrations from the preserved eluates submitted by each of the validation laboratories. The data shown in this appendix is applicable only to the analytical solutions measured by inductively coupled plasma – optical emissions spectrometry (ICP-OES) performed by Vanderbilt University (VU). The analytical methods, instrument calibration, and analytical QA/QC program followed throughout this project are detailed in the main report starting on Page 10.

Organization of the QA/QC Tables

Each table in the appendix represents the final eluate solution concentrations for a single element from a single laboratory. The tables are ordered alphabetically by element symbol (e.g., “Al” for aluminum or “Sb” for antimony) within each the leaching method (i.e., Method 1313 versus Method 1314). The information within each table is ordered by the test position number within each leaching method.

Columns in the Analytical QA/QC Tables

The following is a description of the columns in each table in the appendix.

Analyte (1st Column)

Element or ion symbol based on the periodic table used to identify the analyte.

Sample ID (2nd Column)

Coded identifier for each sample comprised of the following items separated by dashes:

- method code (i.e., “1313” for Method 1313 or “1314” for Method 1314)
- material code (i.e., “AES”)
- test position number (e.g., “T01” for first eluate of the test)
- replicate letter (e.g., “A” for first test run)

Dilution Factor (3rd Column)

The dilution factor for the analytical sample measured in the instrument or the multiplier associated with calculating final eluate concentrations from analytical sample concentration. Typically for ICP-OES, full strength analytical samples are measured and the resultant dilution factor is “1”. However, dilution factors of at least “10” are common for inductively coupled plasma – mass spectrometry (ICP-MS) analysis in order to minimize interferences from analytical solutions with high sodium and potassium content.

Sample Conc. in $\mu\text{g/L}$ (4th Column)

The concentration of the analytical sample in $\mu\text{g/L}$ as measured by the instrument specified in the 14th Column.

Qualifier (5th Column)

A quality control qualifier used to flag analytical samples that are below the quantification or detection limits. The following table of qualifiers was used.

Qualifier	Meaning
E	Estimated value between the MDL and ML values
U	Non-detected values below the MDL
U1/2	Non-detected values below the MDL set to ½ the MDL for graphing purposes

Instrument Conc. in µg/L (6th Column)

The final eluate concentration used in the validation study determined by multiplying the analytical sample concentration (4th Column) by the dilution factor (3rd Column).

RSD (7th Column)

Relative standard deviation of the three analytical measurements of the analytical sample calculated automatically through instrument software. The RSD does not represent the relative standard deviation of replicate analysis using triplicate samples.

MDL (8th Column)

The method detection limit (MDL) in µg/L for the analysis using the instrument specified in the 14th Column. The value is calculated by the VU laboratory manager based on the standard deviation of analysis of seven low-level standard solutions measured on seven non-consecutive days and the Student's *t*-distribution value for the seven replicates at the 99% confidence level (EPA, 2003).

$$MDL = S_{(n=7)} \times t_{(n-1=6, 1-\alpha=0.99)}$$

where $S_{(n=7)}$ is the standard deviation of seven non-consecutive measurements (µg/L), and $t_{(n-1=6, 1-\alpha=0.99)}$ is the Student *t*-distribution value for seven replicates at the 99% confidence level (3.14267).

ML (9th Column)

The estimated minimum level of quantification (ML) in µg/L for the analysis using the instrument specified in the 14th Column. The value is calculated by the VU laboratory manager based on the previously calculated MDL (EPA, 2003).

$$ML = 3.18 \times MDL$$

Spike Solution Conc. (10th Column) and Spike Volume (11th Column)

The concentration in µg/L of the standard solution used to spike analytical samples and the volume of the standard solution added to each analytical sample. When multiplied, these values provide the theoretical mass of an element that is spiked into an analytical sample.

$$M_{QA/QC\ spike} = C_{std} \times \left(V_{std} \times \frac{L}{10^6 \mu L} \right)$$

where $M_{QA/QC\ spike}$ is the theoretical mass of spiked into the analytical sample (μg),
 C_{std} is the concentration of the standard solution ($\mu g/L$), and
 V_{std} is the volume of the standard solution added to the analytical sample (μL).

Spike Conc. (12th Column)

The concentration of the spiked analytical solution in $\mu g/L$ as measured by the instrument specified in the 14th Column.

Spike Recovery (13th Column)

The percentage of spiked mass recovered during analysis of the spiked solution. The spike recovery is calculated by the VU laboratory manager using the following formula:

$$\frac{(C_{QA/QC\ spike} - C_{sample}) \times V_{QA/QC\ spike}}{M_{QA/QC\ spike}} \times 100\%$$

where $C_{QA/QC\ spike}$ is the concentration of the spiked analytical sample ($\mu g/L$),
 C_{sample} is the concentration of the analytical sample in the 4th Column ($\mu g/L$), and
 $V_{QA/QC\ spike}$ is the final volume of the spiked analytical sample (L).

Instrument (14th Column)

The instrument used for the analytical measurement (e.g., "VU ICP-OES" denotes that the instrument was the Vanderbilt University Varian 620 ICP-OES).

Date/Time (15th Column)

The date and time that the analytical sample measurement was recorded by the instrument software. This column is used to uniquely identify each analysis and does not reflect the data and time of spiked solution measurement.

Filename (16th Column)

The name of the instrument output file where the analytical data is stored.

Analytical QA/QC Summary – Method 1313

ICP-OES

Analyte	Symbol	Analytical Samples	Results > ML	RSDs Meeting DQI Goals	Mean RSD (%)	Completeness (%)
Aluminum	Al	18	18	18	3.6	100%
Boron	B	18	18	18	1.0	100%
Barium	Ba	18	18	18	3.2	100%
Calcium	Ca	18	18	18	0.8	100%
Cobalt	Co	18	8	8	2.0	100%
Copper	Cu	18	6	6	1.9	100%
Iron	Fe	18	8	8	2.1	100%
Lithium	Li	18	18	18	2.4	100%
Magnesium	Mg	18	18	18	1.3	100%
Manganese	Mn	18	12	12	1.1	100%
Molybdenum	Mo	18	18	18	1.5	100%
Nickel	Ni	18	12	12	1.9	100%
Phosphorus	P	18	12	12	3.1	100%
Potassium	K	18	18	18	2.2	100%
Silicon	Si	18	18	18	1.0	100%
Sodium	Na	18	18	18	0.7	100%
Strontium	Sr	18	18	18	1.3	100%
Sulfur	S	18	18	18	0.8	100%
Titanium	Ti	18	2	2	1.6	100%
Vanadium	V	18	18	18	1.2	100%
Zinc	Zn	18	6	6	1.2	100%
				Overall	1.7%	100%

ICP-OES

Analyte	Symbol	Analytical Samples	Results > ML	RSDs Meeting DQI Goals	Mean RSD (%)	Completeness (%)
Antimony	Sb	18	18	18	1.7	100%
Arsenic	As	18	18	18	2.7	100%
Beryllium	Be	18	4	4	2.8	100%
Cadmium	Cd	18	18	18	2.3	100%
Cesium	Cs	18	18	18	1.6	100%
Chromium	Cr	18	18	18	2.9	100%
Lead	Pb	18	6	6	1.1	100%
Selenium	Se	18	18	18	2.4	100%
Thallium	Tl	18	10	10	1.5	100%
Tin	Sn	18	2	2	3.4	100%
Uranium	U	18	12	12	7.8	100%
				Overall	2.7%	100%

Carbon Analysis

Analyte	Symbol	Analytical Samples	Results > ML	RSDs Meeting DQI Goals	Mean RSD (%)	Completeness (%)
Carbon, Inorganic	DIC	18	12	12	3.8	100%
Carbon, Organic	DOC	18	18	18	0.9	100%
Overall					2.3%	100%

IC

Analyte	Symbol	Analytical Samples	Results > ML	RSDs Meeting DQI Goals	Mean RSD (%)	Completeness (%)
Bromide	Br	18	0	0	RSD and Completeness are not available as IC analysis is based on a single measurement of an analytical sample.	
Chloride	Cl	18	18	18		
Fluoride	F	18	18	18		
Nitrate	NO3	18	18	18		
Nitrite	NO2	18	0	0		
Phosphate	PO4	18	0	0		
Sulfate	SO4	18	18	18		
Overall					NA	NA

Analytical QA/QC Summary – Method 1314

ICP-OES

Analyte	Symbol	Analytical Samples	Results > ML	RSDs Meeting DQI Goals	Mean RSD (%)	Completeness (%)
Aluminum	Al	18	18	18	1.7	100%
Boron	B	18	18	18	1.1	100%
Barium	Ba	18	18	18	1.0	100%
Calcium	Ca	18	18	18	1.0	100%
Cobalt	Co	18	2	2	1.7	100%
Copper	Cu	18	2	2	6.0	100%
Iron	Fe	18	0	0	NA	NA
Lithium	Li	18	18	18	2.0	100%
Magnesium	Mg	18	18	18	3.9	100%
Manganese	Mn	18	0	0	NA	NA
Molybdenum	Mo	18	18	18	1.8	100%
Nickel	Ni	18	6	6	4.9	100%
Phosphorus	P	18	6	6	2.7	100%
Potassium	K	18	18	18	1.1	100%
Silicon	Si	18	18	18	1.1	100%
Sodium	Na	18	18	18	0.9	100%
Strontium	Sr	18	18	18	1.4	100%
Sulfur	S	18	18	18	1.0	100%
Titanium	Ti	18	0	0	NA	NA
Vanadium	V	18	18	18	1.3	100%
Zinc	Zn	18	16	16	4.4	100%
				Overall	2.2%	100%

ICP-OES

Analyte	Symbol	Analytical Samples	Results > ML	RSDs Meeting DQI Goals	Mean RSD (%)	Completeness (%)
Antimony	Sb	18	8	8	2.1	100%
Arsenic	As	18	18	18	2.6	100%
Beryllium	Be	18	0	0	NA	NA
Cadmium	Cd	18	8	8	2.9	100%
Cesium	Cs	18	18	18	1.8	100%
Chromium	Cr	18	13	13	2.1	100%
Lead	Pb	18	4	4	4.4	100%
Selenium	Se	18	18	18	2.1	100%
Thallium	Tl	18	2	2	4.5	100%
Tin	Sn	18	2	2	2.4	100%
Uranium	U	18	0	0	NA	NA
				Overall	2.8%	100%

Carbon Analysis

Analyte	Symbol	Analytical Samples	Results > ML	RSDs Meeting DQI Goals	Mean RSD (%)	Completeness (%)
Carbon, Inorganic	DIC	18	18	18	2.6	100%
Carbon, Organic	DOC	18	18	18	0.9	100%
Overall					1.7%	100%

IC

Analyte	Symbol	Analytical Samples	Results > ML	RSDs Meeting DQI Goals	Mean RSD (%)	Completeness (%)
Bromide	Br	18	4	4	RSD and Completeness are not available as IC analysis is based on a single measurement of an analytical sample.	
Chloride	Cl	18	18	18		
Fluoride	F	18	18	18		
Nitrate	NO3	18	18	18		
Nitrite	NO2	18	0	0		
Phosphate	PO4	18	0	0		
Sulfate	SO4	18	18	18		
Overall					NA	NA

Matrix Spike Recovery Summary – Method 1313

ICP-OES

Analyte	Symbol	Sample Conc (µg/L)	Spike Conc Calculated (g/L)	Spike Conc Measured (g/L)	Recovery (g/L)	Recovery (%)
Aluminum	Al	348.29	1,000	1,387.3	1,039.0	104%
Aluminum	Al	336.59	1,000	1,353.2	1,016.6	102%
Boron	B	1,150.93	1,000	2,225.8	1,074.9	107%
Boron	B	1,135.35	1,000	2,171.6	1,036.2	104%
Barium	Ba	68.81	1,000	1,008.3	939.4	94%
Barium	Ba	69.01	1,000	997.6	928.6	93%
Calcium	Ca	566,606.00	1,000	567,514.0	908.0	91%
Calcium	Ca	573,389.00	1,000	574,264.0	875.0	88%
Cobalt	Co	0.90	1,000	993.9	993.0	99%
Cobalt	Co	0.90	1,000	970.6	969.7	97%
Copper	Cu	1.85	1,000	1,030.2	1,028.3	103%
Copper	Cu	1.85	1,000	1,025.1	1,023.2	102%
Iron	Fe	1.00	1,000	1,061.1	1,060.1	106%
Iron	Fe	1.00	1,000	1,042.9	1,041.9	104%
Lithium	Li	278.99	1,000	1,256.2	977.2	98%
Lithium	Li	284.46	1,000	1,256.4	971.9	97%
Magnesium	Mg	9.18	1,000	1,036.1	1,026.9	103%
Magnesium	Mg	9.98	1,000	1,026.6	1,016.6	102%
Manganese	Mn	177.15	1,000	1,170.3	993.2	99%
Manganese	Mn	214.98	1,000	1,161.3	946.3	95%
Molybdenum	Mo	646.46	1,000	1,650.1	1,003.7	100%
Molybdenum	Mo	643.36	1,000	1,611.0	967.7	97%
Nickel	Ni	0.90	1,000	1,043.1	1,042.2	104%
Nickel	Ni	0.90	1,000	1,034.4	1,033.5	103%
Phosphorus	P	1.85	1,000	1,056.1	1,054.3	105%
Phosphorus	P	1.85	1,000	1,023.9	1,022.1	102%
Potassium	K	276,032.00	1,000	276,966.0	934.0	93%
Potassium	K	282,925.00	1,000	283,826.0	901.0	90%
Silicon	Si	9,420.70	1,000	10,372.1	951.4	95%
Silicon	Si	9,392.35	1,000	10,322.1	929.8	93%
Sodium	Na	491,942.00	1,000	492,837.0	895.0	90%
Sodium	Na	517,956.00	1,000	518,837.0	881.0	88%
Strontium	Sr	7,809.93	1,000	8,763.5	953.5	95%
Strontium	Sr	8,742.79	1,000	9,663.5	920.7	92%
Sulfur	S	973,267.00	5,000	978,043.0	4,776.0	96%
Sulfur	S	988,445.00	5,000	993,043.0	4,598.0	92%
Titanium	Ti	1.00	1,000	1,060.5	1,059.5	106%
Titanium	Ti	1.00	1,000	1,029.3	1,028.3	103%
Vanadium	V	158.34	1,000	1,163.8	1,005.4	101%
Vanadium	V	155.34	1,000	1,137.6	982.2	98%
Zinc	Zn	0.50	1,000	1,149.5	1,149.0	115%
Zinc	Zn	0.50	1,000	1,123.6	1,123.1	112%
Mean						99%

ICP-MS

Analyte	Symbol	Sample Conc (µg/L)	Spike Conc Calculated (g/L)	Spike Conc Measured (g/L)	Recovery (g/L)	Recovery (%)
Antimony	Sb	11.30	500	517.58	506.3	101%
Antimony	Sb	12.10	500	491.05	479.0	96%
Beryllium	Be	0.32	500	504.71	504.4	101%
Beryllium	Be	0.32	500	484.35	484.0	97%
Cadmium	Cd	0.71	500	516.38	515.7	103%
Cadmium	Cd	0.77	500	508.41	507.6	102%
Cesium	Cs	20.10	500	499.22	479.1	96%
Cesium	Cs	21.20	500	495.03	473.8	95%
Chromium	Cr	12.70	500	518.00	505.3	101%
Chromium	Cr	14.60	500	512.00	497.4	99%
Lead	Pb	0.11	500	488.03	487.9	98%
Lead	Pb	0.11	500	476.39	476.3	95%
Selenium	Se	199.00	500	705.96	507.0	101%
Selenium	Se	175.00	500	665.41	490.4	98%
Thallium	Tl	0.25	500	497.71	497.5	99%
Thallium	Tl	0.25	500	474.38	474.1	95%
Tin	Sn	0.35	500	492.10	491.8	98%
Tin	Sn	0.35	500	483.60	483.3	97%
Uranium	U	0.15	500	502.81	502.7	101%
Uranium	U	0.15	500	499.38	499.2	100%
Mean						99%

TC

Analyte	Symbol	Sample Conc (µg/L)	Spike Conc Calculated (g/L)	Spike Conc Measured (g/L)	Recovery (g/L)	Recovery (%)
Carbon, inorganic	DIC	6,413.00	500	6,877.00	464.0	93%
Carbon, inorganic	DIC	6,405.00	500	6,865.00	460.0	92%
Carbon, organic	DOC	4,873.00	500	5,341.00	468.0	94%
Carbon, organic	DOC	5,357.00	500	5,814.00	457.0	91%
Mean						92%

Matrix Spike Recovery Summary – Method 1314

ICP-OES

Analyte	Symbol	Sample Conc (µg/L)	Spike Conc Calculated (g/L)	Spike Conc Measured (g/L)	Recovery (g/L)	Recovery (%)
Aluminum	Al	435.79	1,000	1,461.8	1,026.0	103%
Aluminum	Al	520.39	1,000	1,546.5	1,026.1	103%
Boron	B	2,142.53	1,000	3,066.0	923.5	92%
Boron	B	1,964.58	1,000	2,848.3	883.7	88%
Barium	Ba	33.56	1,000	1,115.6	1,082.1	108%
Barium	Ba	31.73	1,000	1,075.0	1,043.2	104%
Calcium	Ca	427,401.00	1,000	428,358.0	957.0	96%
Calcium	Ca	416,730.00	1,000	417,658.0	928.0	93%
Cobalt	Co	2.36	1,000	1,058.3	1,055.9	106%
Cobalt	Co	1.92	1,000	995.4	993.5	99%
Copper	Cu	4.46	1,000	1,105.1	1,100.6	110%
Copper	Cu	4.10	1,000	1,037.9	1,033.8	103%
Iron	Fe	1.00	1,000	1,073.0	1,072.0	107%
Iron	Fe	1.00	1,000	1,006.3	1,005.3	101%
Lithium	Li	2,145.97	1,000	3,089.3	943.3	94%
Lithium	Li	2,106.65	1,000	3,002.7	896.0	90%
Magnesium	Mg	1.00	1,000	1,067.6	1,066.6	107%
Magnesium	Mg	1.00	1,000	1,009.3	1,008.3	101%
Manganese	Mn	124.48	1,000	1,151.8	1,027.3	103%
Manganese	Mn	117.86	1,000	1,061.1	943.2	94%
Molybdenum	Mo	1,447.32	1,000	2,469.7	1,022.4	102%
Molybdenum	Mo	1,590.47	1,000	2,543.7	953.2	95%
Nickel	Ni	6.82	1,000	1,039.5	1,032.6	103%
Nickel	Ni	6.87	1,000	986.4	979.5	98%
Phosphorus	P	37.56	1,000	1,098.4	1,060.9	106%
Phosphorus	P	32.54	1,000	1,083.5	1,051.0	105%
Potassium	K	1,034,420.00	5,000	1,039,150.0	4,730.0	95%
Potassium	K	967,993.00	5,000	972,570.0	4,577.0	92%
Silicon	Si	18,312.50	1,000	19,271.8	959.3	96%
Silicon	Si	18,063.10	1,000	18,988.6	925.5	93%
Sodium	Na	1,028,840.00	5,000	1,033,350.0	4,510.0	90%
Sodium	Na	967,339.00	5,000	971,630.0	4,291.0	86%
Strontium	Sr	8,486.42	1,000	9,592.1	1,105.7	111%
Strontium	Sr	8,278.21	1,000	9,333.4	1,055.2	106%
Sulfur	S	2,916,400.00	5,000	2,920,820.0	4,420.0	88%
Sulfur	S	2,842,140.00	5,000	2,846,400.0	4,260.0	85%
Titanium	Ti	1.00	1,000	1,132.3	1,131.3	113%
Titanium	Ti	1.00	1,000	1,064.7	1,063.7	106%
Vanadium	V	458.46	1,000	1,516.3	1,057.8	106%
Vanadium	V	437.57	1,000	1,448.4	1,010.9	101%
Zinc	Zn	9.19	1,000	1,093.8	1,084.6	108%
Zinc	Zn	8.76	1,000	1,106.7	1,097.9	110%
Mean						100%

ICP-MS

Analyte	Symbol	Sample Conc (µg/L)	Spike Conc Calculated (g/L)	Spike Conc Measured (g/L)	Recovery (g/L)	Recovery (%)
Antimony	Sb	11.90	500	512.15	500.3	100%
Antimony	Sb	12.40	500	504.71	492.3	98%
Beryllium	Be	0.32	500	506.09	505.8	101%
Beryllium	Be	0.32	500	499.38	499.1	100%
Cadmium	Cd	2.58	500	509.32	506.7	101%
Cadmium	Cd	2.92	500	503.02	500.1	100%
Cesium	Cs	71.30	500	543.82	472.5	95%
Cesium	Cs	69.40	500	528.78	459.4	92%
Chromium	Cr	36.80	500	517.18	480.4	96%
Chromium	Cr	30.90	500	504.22	473.3	95%
Lead	Pb	0.11	500	501.07	501.0	100%
Lead	Pb	0.11	500	489.26	489.1	98%
Selenium	Se	226.00	500	694.83	468.8	94%
Selenium	Se	182.00	500	661.07	479.1	96%
Thallium	Tl	0.59	500	491.23	490.6	98%
Thallium	Tl	0.56	500	485.95	485.4	97%
Tin	Sn	1.51	500	512.85	511.3	102%
Tin	Sn	1.46	500	500.37	498.9	100%
Uranium	U	0.15	500	496.28	496.1	99%
Uranium	U	0.15	500	488.44	488.3	98%
Mean						99%

TC

Analyte	Symbol	Sample Conc (µg/L)	Spike Conc Calculated (g/L)	Spike Conc Measured (g/L)	Recovery (g/L)	Recovery (%)
Carbon, inorganic	DIC	3,465.00	500	3,944.00	479.0	96%
Carbon, inorganic	DIC	3,848.00	500	4,308.00	460.0	92%
Carbon, organic	DOC	11,840.00	500	12,310.00	470.0	94%
Carbon, organic	DOC	10,678.00	500	11,132.00	454.0	91%
Mean						92%