LEAF

Leaching Environmental Assessment Framework

The Leaching Environmental Assessment Framework as a Tool for Risk-informed, Science-based Regulation

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VANDERBILT SCHOOL OF ENGINEERING

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ECN Energy research Centre of the Netherlands

ARCADIS Infrastructure · Water · Environment · Buildings

EPA United States Environmental Protection Agency

Hans van der Sloot Consultancy

DHI

Presentation Outline

Introduction to Leaching Assessment

Overview of LEAF

- Leaching Tests
- Data Management

Overview of Interlaboratory Validation

Applicability to DOE Challenges

Conclusions

Materials Testing – Historically

1960s-1990s

Protection from hazardous wastes; waste minimization/conservation.

- Classification of “hazardous” waste (RCRA Subtitle C/D landfills)
- Acceptance criteria for disposal of treated wastes (Universal Treatment Standards)
- Best demonstrated available treatment (BDAT)

1990s – present

Move toward integrated materials management; balancing overall environmental performance with materials costs and long-term liability

- Global economic policy (resource costs, international trade)
- Risk-informed waste management practices
- Changing definition of waste materials (e.g., Dutch Building Materials Decree; U.S. definition of solid waste)
- Applications for waste delisting and alternative measures of treatment effectiveness
- Re-use of waste materials (mine reclamation, alternative construction materials)

What is Leaching?

Process by which constituents of a solid material are released into a contacting water phase

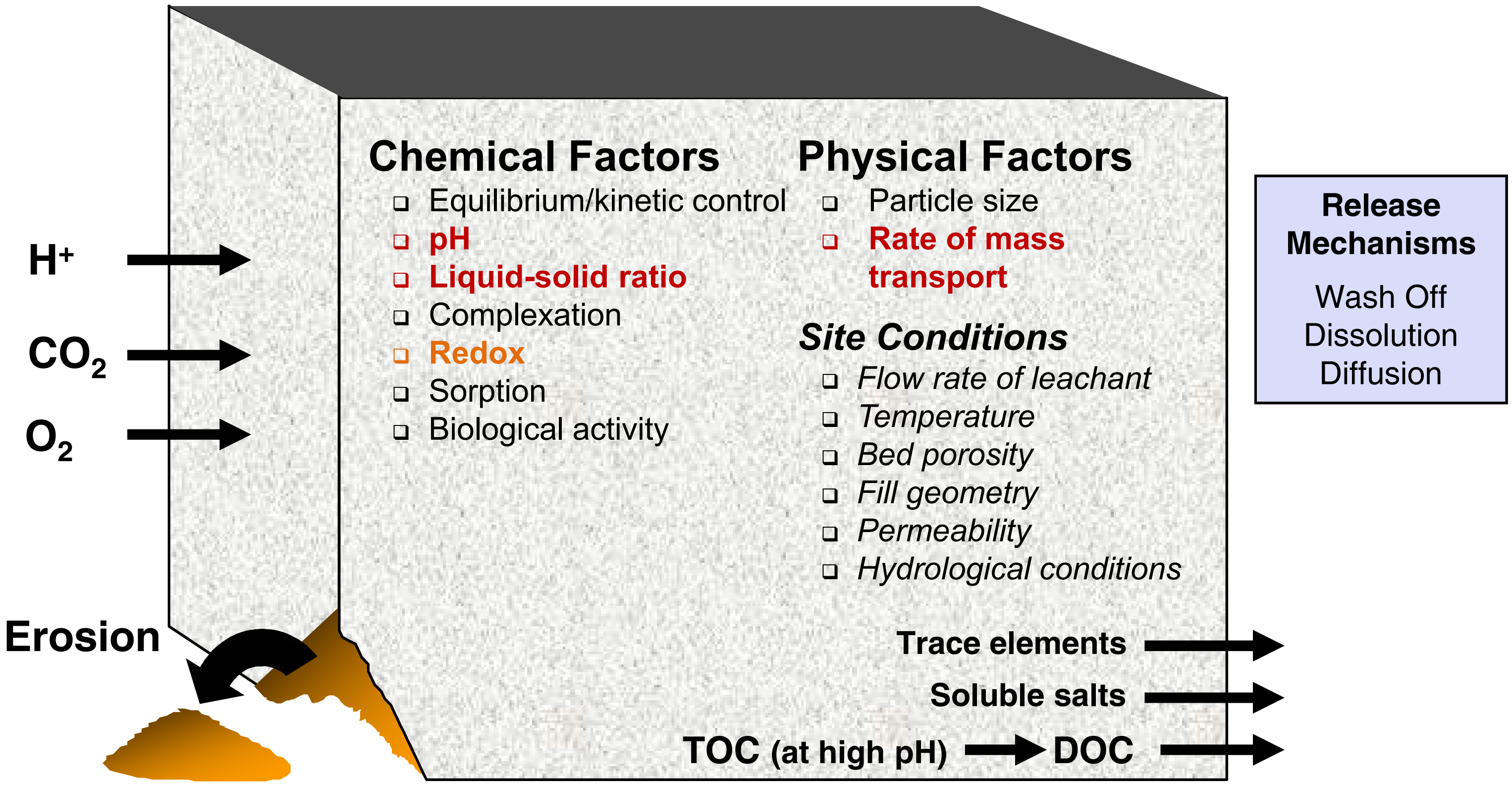
Percolation Release

- Water passes thru material
- Equilibrium
- High concentration

Mass Transfer Release

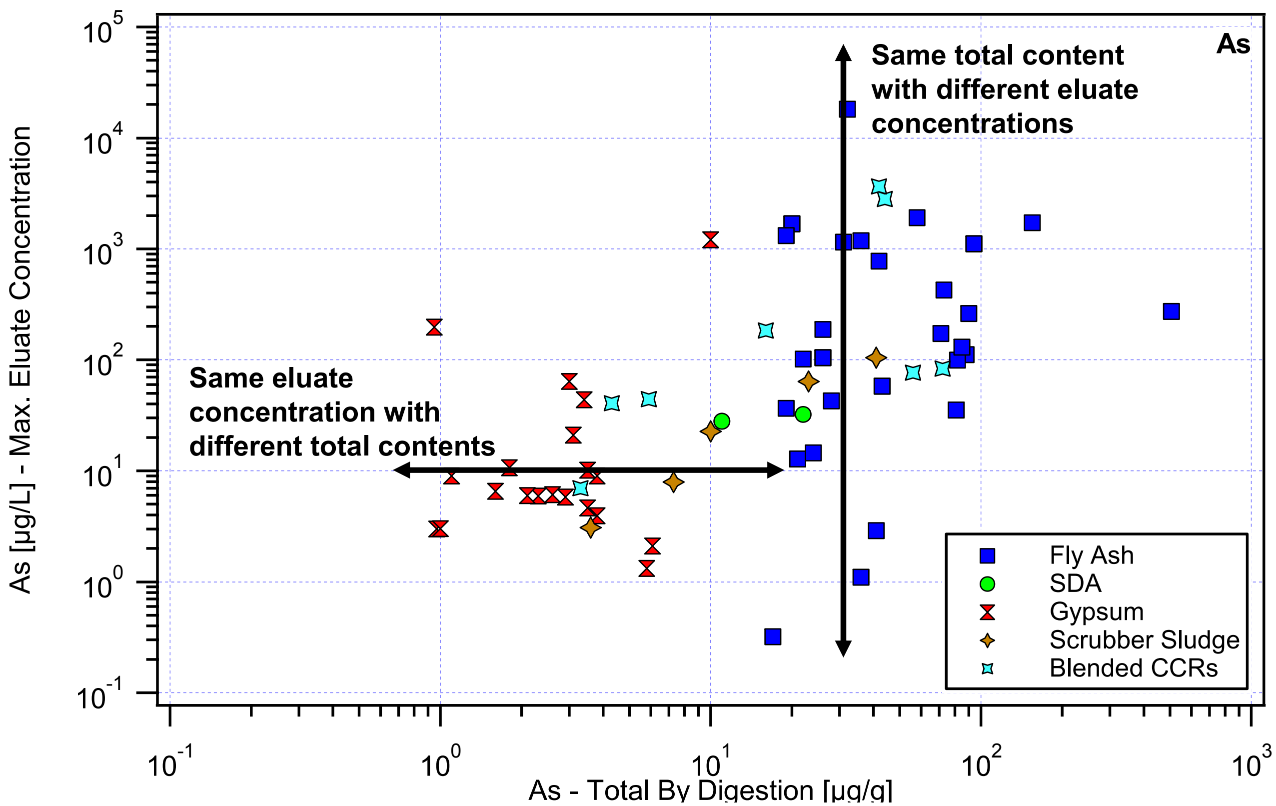
- Water flows around material
- Diffusion to material surface
- Lower concentration

Leaching - Controlling Factors

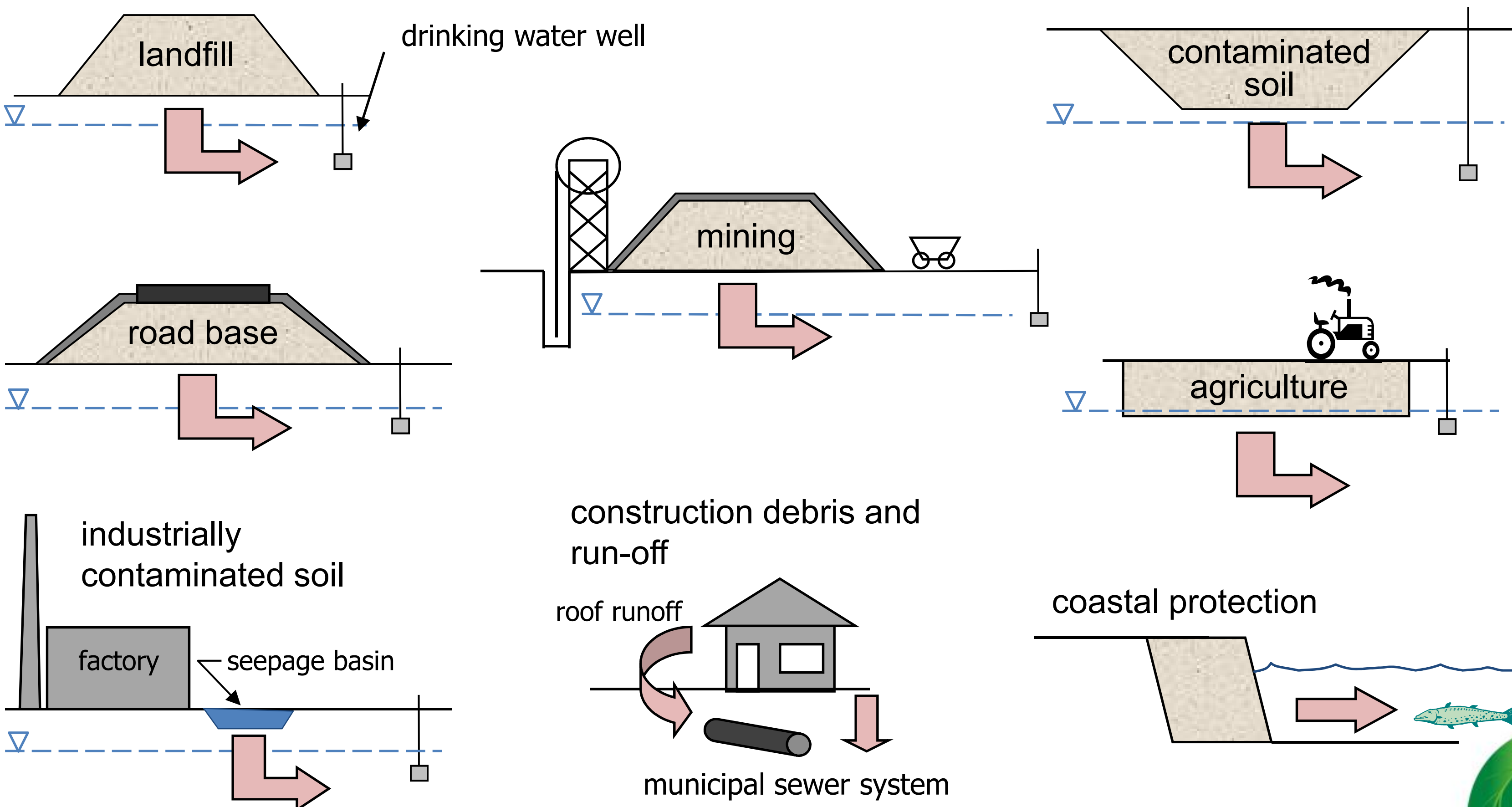


Total Content

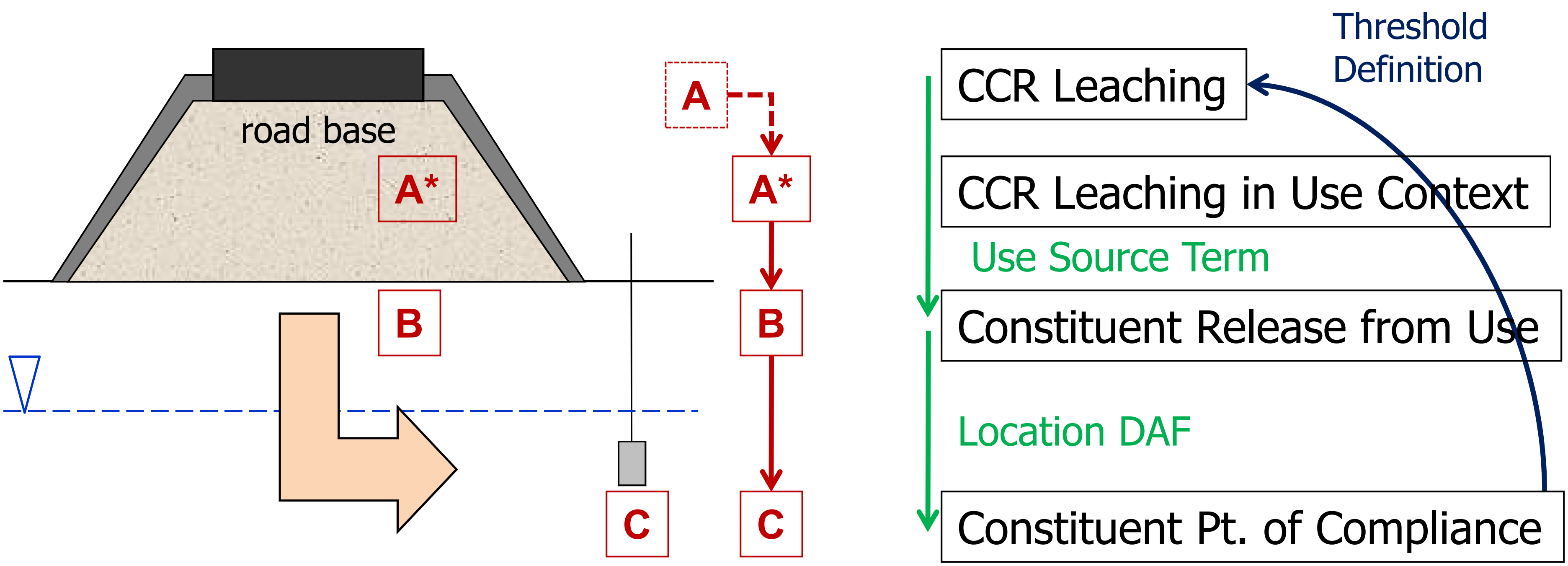
Total Content **Does Not** Correlate to Leaching



Many Leaching Scenarios …



Common Assessment Approach



Leaching Tests

Total Content

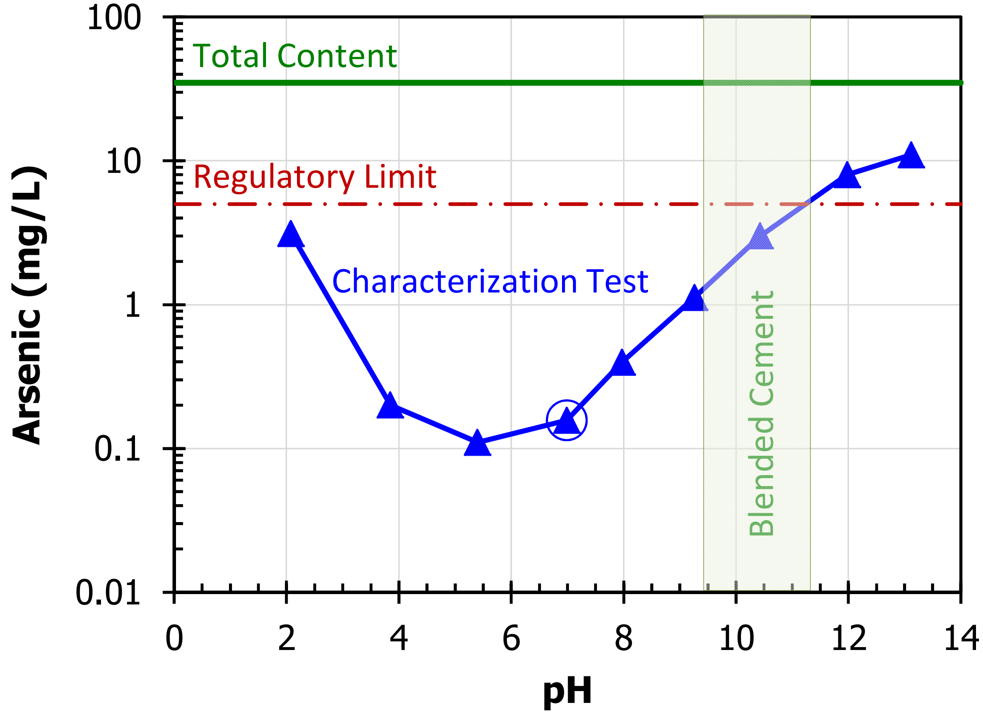
- Correlation to leaching?

Regulatory Tests

- Comparison to limits
- Does not consider
  - Release Scenario
  - Time (kinetics)
  - Mass Transport

Characterization Tests

- Range of conditions
- Comparisons between
  - Materials
  - Treatments
  - Scenarios



Leaching Method Development

Leaching characterization applied to anticipated release conditions resulting in improved accuracy and more reliable environmental decision making

"An Integrated Framework for Evaluating Leaching in Waste Management and Utilization of Secondary Materials," D.S. Kosson, H.A. van der Sloot, F. Sanchez, and A.C. Garrabrants, Environmental Engineering Science, 19(3): 159-204, 2002.

Parallel and coordinated methods development in the EU and US

Designed to address concerns of EPA Science Advisory Board

- Form of the material (e.g., monolithic)
- Leaching parameters (e.g., pH, liquid-solid ratio (L/S), release rate)

Intended for situations where TCLP is <u>not</u> required or best suited

- Assessment of materials for beneficial reuse
- Evaluating treatment effectiveness (determination of equivalent treatment)
- Characterizing potential release from high-volume materials
- Corrective action (remediation decisions)

Leaching Environmental Assessment Framework

LEAF is a collection of …

- Four leaching methods
- Data management tools
- Geochemical speciation and mass transfer modeling
- Quality assurance/quality control for materials production
- Integrated leaching assessment approaches

… designed to identify characteristic leaching behaviors for a wide range of materials and associated use and disposal scenarios.

LEAF facilitates integration of leaching methods which provides a material-specific “source term” release for support of material management decisions.

More information at http://www.vanderbilt.edu/leaching

LEAF Leaching Methods

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

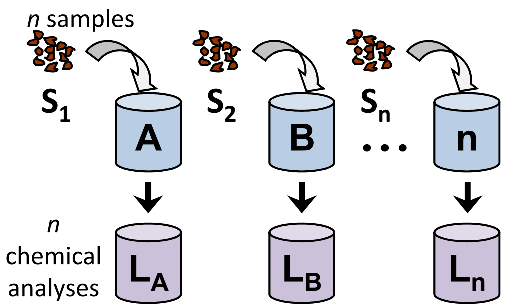
Method 1314 – Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio (L/S) using an Up-flow Percolation Column Procedure

Method 1315 – Mass Transfer Rates in Monolithic and Compacted Granular Materials using a Semi-dynamic Tank Leaching Procedure

Method 1316 – Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio using a Parallel Batch Procedure

Note: Incorporation into SW-846 is ongoing; method identification numbers are subject to change

Method 1313 Overview

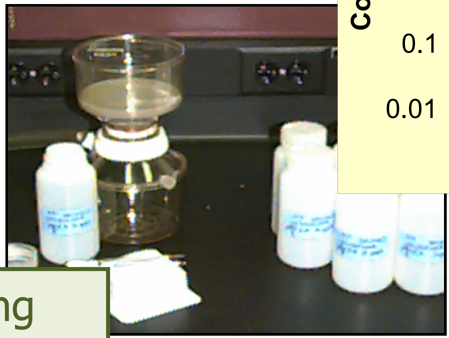


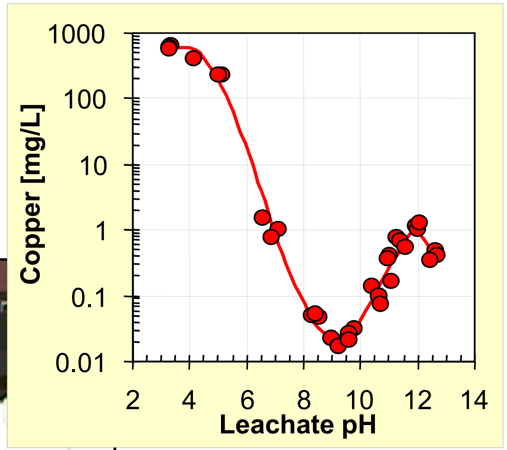
Equilibrium Leaching Test

- Parallel batch as function of pH

Test Specifications

- 9 specified target pH values plus natural conditions
- Size-reduced material
- L/S = 10 mL/g-dry
- Dilute HNO_3 or NaOH
- Contact time based on particle size
  - 18-72 hours
- Reported Data
  - Equivalents of acid/base added
  - Eluate pH and conductivity
  - Eluate constituent concentrations





Titration Curve and Liquid-solid Partitioning (LSP) Curve as Function of Eluate pH

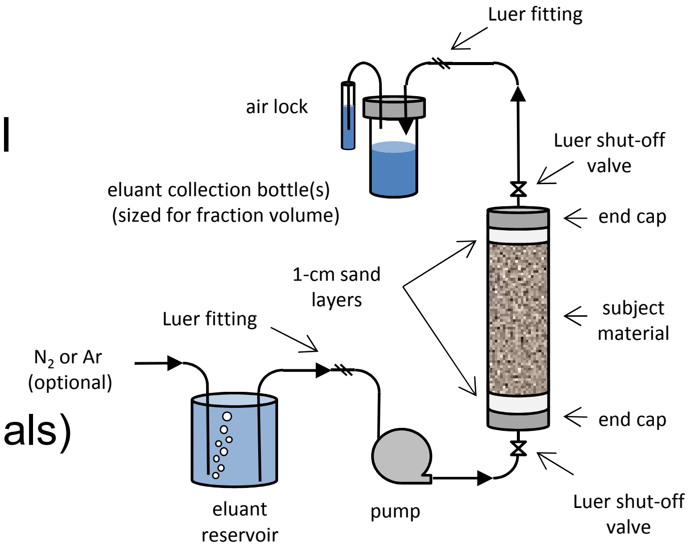
Method 1314 Overview

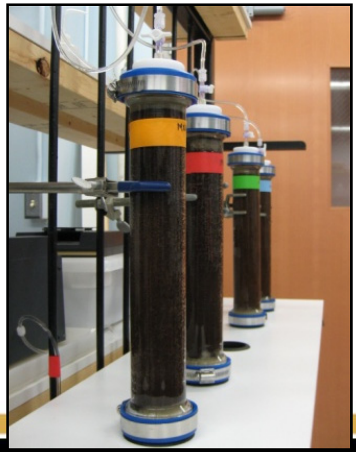
Equilibrium Leaching Test

- Percolation through loosely-packed material

Test Specifications

- 5-cm diameter x 30-cm high glass column
- Size-reduced material
- DI water or 1 mM $CaCl_2$ (clays, organic materials)
- Upward flow to minimize channeling
- Collect leachate at cumulative L/S
  - ❑ 0.2, 0.5, 1, 1.5, 2, 4.5, 5, 9.5, 10 mL/g-dry
- Reported Data
  - ❑ Eluate volume collected
  - ❑ Eluate pH and conductivity
  - ❑ Eluate constituent concentrations



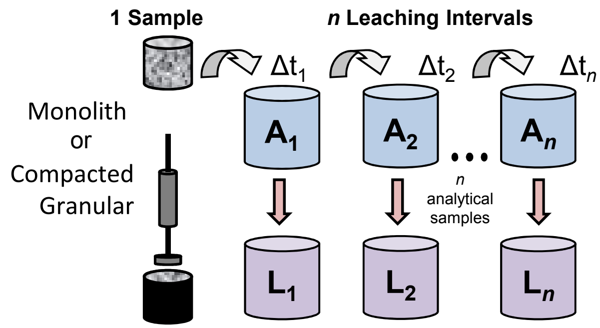


Liquid-solid Partitioning (LSP) Curve as Function of L/S; Estimate of Pore Water Concentration

Method 1315 Overview

Mass-Transfer Test

- Semi-dynamic tank leach test

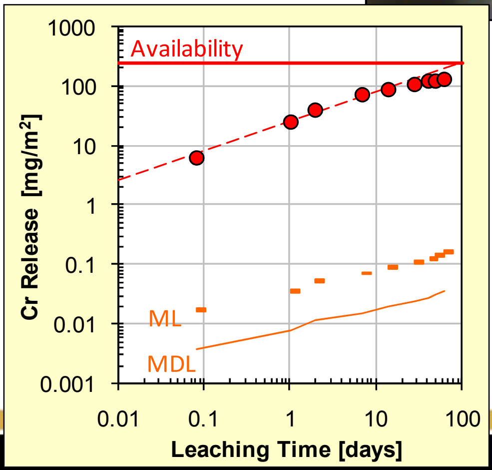


Monolithic

Granular

Test Specifications

- Material forms
  - ❑ monolithic (all faces exposed)
  - ❑ compacted granular (1 circular face exposed)
- DI water so that waste dictates pH
- Liquid-surface area ratio (L/A) of 9±1 mL/cm^2
- Refresh leaching solution at cumulative times
  - ❑ 2, 25, 48 hrs, 7, 14, 28, 42, 49, 63 days
- Reported Data
  - ❑ Refresh time
  - ❑ Eluate pH and conductivity
  - ❑ Eluate constituent concentrations

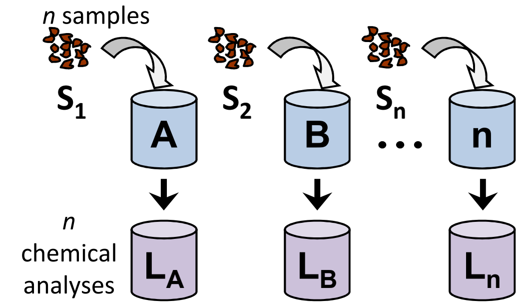


Flux and Cumulative Release as a Function of Leaching Time

Method 1316 Overview

Equilibrium Leaching Test

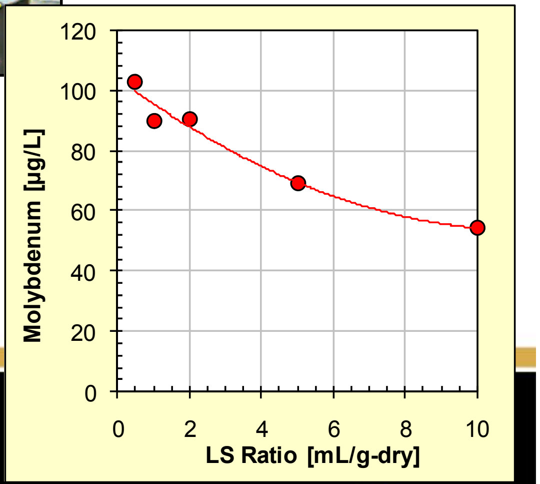
- Parallel batch as function of L/S



Test Specifications

- Five specified L/S values (±0.2 mL/g-dry)
  - ❑ 10.0, 5.0, 2.0, 1.0, 0.5 mL/g-dry
- Size-reduced material
- DI water (material dictates pH)
- Contact time based on particle size
  - ❑ 18-72 hours
- Reported Data
  - ❑ Eluate L/S
  - ❑ Eluate pH and conductivity
  - ❑ Eluate constituent concentrations

Liquid-solid Partitioning (LSP) Curve as a Function of L/S; Estimate of Pore Water Concentration



Data Management Tools

Data Templates

- Excel Spreadsheets for Each Method
  - Perform basic, required calculations (e.g, moisture content)
  - Record laboratory data
  - Archive analytical data with laboratory information
- Form the upload file to materials database

LeachXS (Leaching eXpert System) Lite

- Data management, visualization and processing program
- Compare Leaching Test Data
  - Between materials for a single constituent (e.g., As in two different CCRs)
  - Between constituents in a single material (e.g., Ba and SO_4 in cement)
  - To default or user-defined “indicator lines” (e.g., QA limits, threshold values)
- Export leaching data to Excel spreadsheets
- Freely available at http://www.vanderbilt.edu/leaching

Data Templates

DRAFT METHOD 1313 (Liquid-Solid Partitioning as a Function of pH) LAB DATA

1) Enter particle size and solids content

	Code	Description *(optional)*
Project	ABC	Example project
Material	XYZ	Exaple material
Replicate	A	

Test conducted by:

Solids Information

Maximum Particle Size	0.3	[mm]
Minimum Dry Equivalent Mass *	20.00	[g-dry]
Solids Content (default = 1)	0.901	[g-dry/g]
Mass of "As Tested" Material / Extraction	22.20	[g]

Extraction Information

LS Ratio	10	[mL/g-dry
Liquid Volume / Extraction	200	[mL]
Recommended Bottle Size *	250	[mL]

	Date	Time
Test Start	1/2/xx	2:00 PM
Test End	1/3/xx	1:45 PM
Required Contact Time *	23-25	[hr]

* Data based on Draft Method 1313 Table 1.

2) Enter acid/base type & normality

Nominal Reagent Information

Acid Type	HNO3	
Acid Normality	2.0	[meq/mL]
Base Type	NaOH	
Base Normality	1.0	[meq/mL]

4) Follow "set-up" recipe

Schedule of Acid and Base Addition

est Position	T01	T02	T03	T04	T05	T06	T07	T08	T09	B01	B02	B03	totals
"As Tested" Solid [g] (±0.05g)	22.20	22.20	22.20	22.20	22.20	22.20	22.20	22.20	22.20	no solid	no solid	no solid	199.8
Reagent Water [mL] (±5%)	147.80	167.80	185.80	197.80	195.80	193.80	189.80	185.80	178.80	200.00	181.00	150.00	2174.2
Acid Volume [mL] (±1%)	-	-	-	-	2.00	4.00	8.00	12.00	19.00	-	19.00	-	64.0
Base Volume [mL] (±1%)	50.00	30.00	12.00	-	-	-	-	-	-	-	-	50.00	142.0
Acid Normality [meq/mL]	-	-	-	-	2.0	2.0	2.0	2.0	2.0	-	2.0	-	
Base Normality [meq/mL]	1.0	1.0	1.0	-	-	-	-	-	-	-			

3) Enter target equivalents from titration curve

Target pH	13.0±0.5	12.0±0.5	10.5±0.5	natural	8.0±0.5	7.0±0.5	5.5±0.5	4.0±0.5	2.0±0.5			
Acid Addition [meq/g]	-2.5	-1.5	-0.6	0	0.2	0.4	0.8	1.2	1.9	Water	Acid	Base
Eluate pH	12.80	12.20	10.80	9.20	7.80	5.98	4.79	3.60	2.30			
te EC [mS/cm]												
luate Eh [mV]												
nter "a" or "r")	✔	✔	✔	✔	✔	✘	✘	✔	✔			
Notes						pH out of range	pH out of range					

5) Record pH, conductivity, Eh (optional)

6) Verify that final pH is in acceptable range

LeachXS Lite

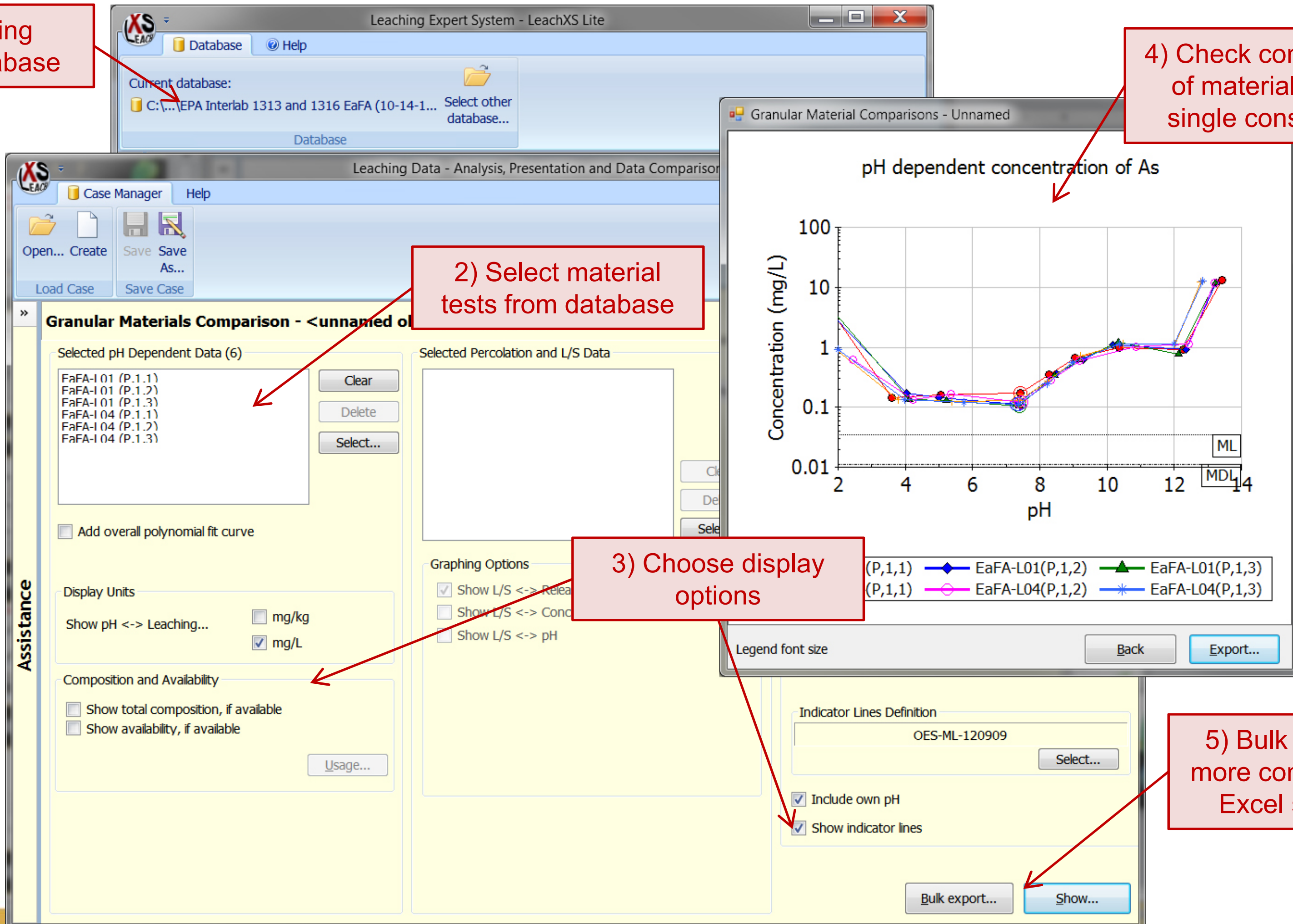
1) Set working materials database

2) Select material tests from database

3) Choose display options

4) Check comparison of materials for a single constituent

5) Bulk export one or more constituents to an Excel spreadsheet



LEAF Methods Validation

Study Materials

Coal Combustion Fly Ash

- Collected for EPA study
- Selected for validation of …
  - ❑ Method 1313
  - ❑ Method 1316

Solidified Waste Analog

- Created at Vanderbilt University
- Blast Furnace Slag, Class C Fly Ash, Type I/II Cement, Metal Salts
- Selected for validation of …
  - ❑ Method 1313
  - ❑ Method 1316
  - ❑ Method 1315

Contaminated Field Soil

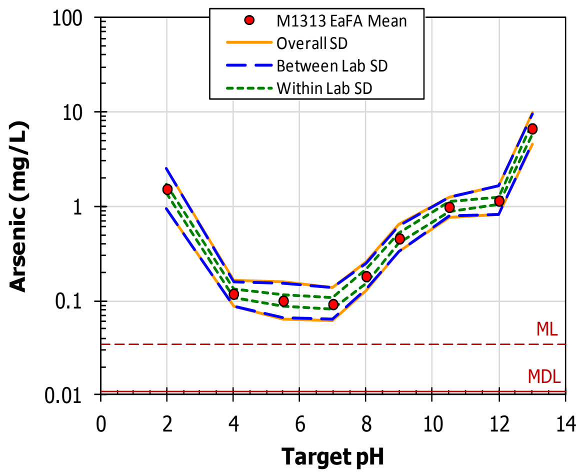
- Copper smelter site
- Selected for validation of…
  - ❑ Method 1313
  - ❑ Method 1316
  - ❑ Method 1315
  - ❑ Method 1314

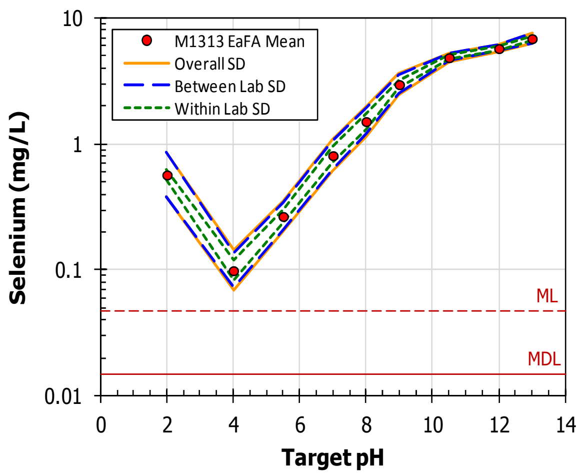
Brass Foundry Sand

- Selected for validation of …
  - ❑ Method 1315
  - ❑ Method 1314

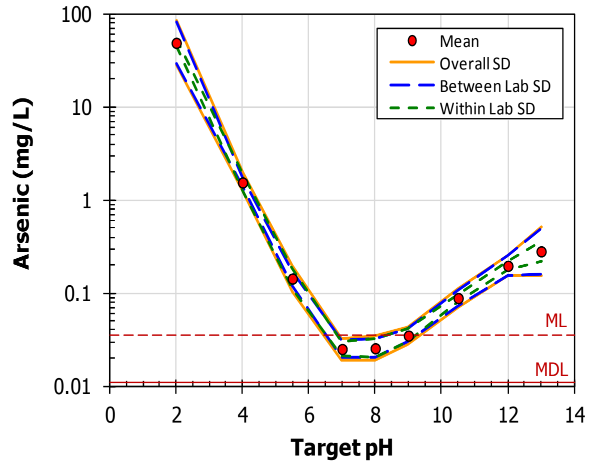
Method 1313 Validation

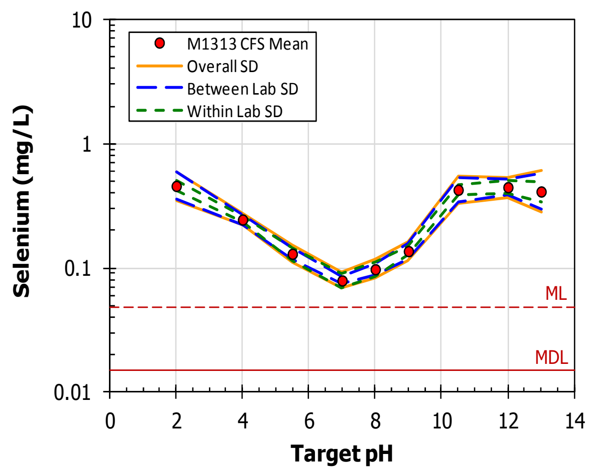
Coal Combustion Fly Ash



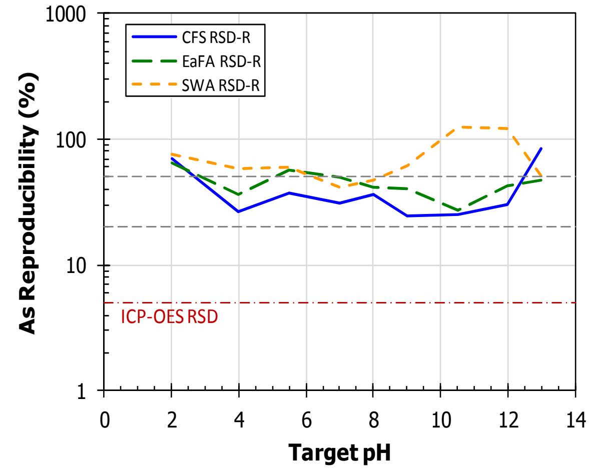


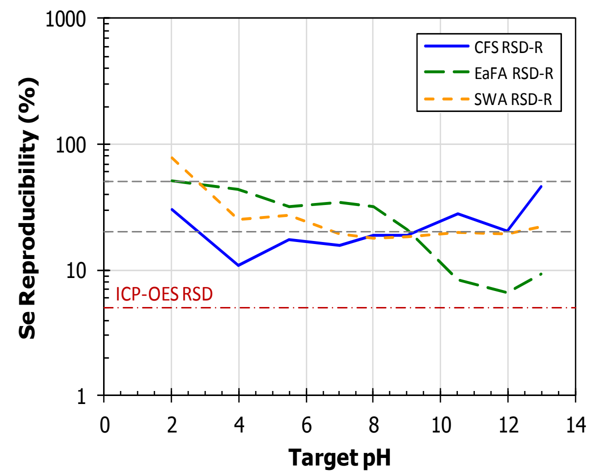
Contaminated Field Soil





Reproducibility





Data Processing

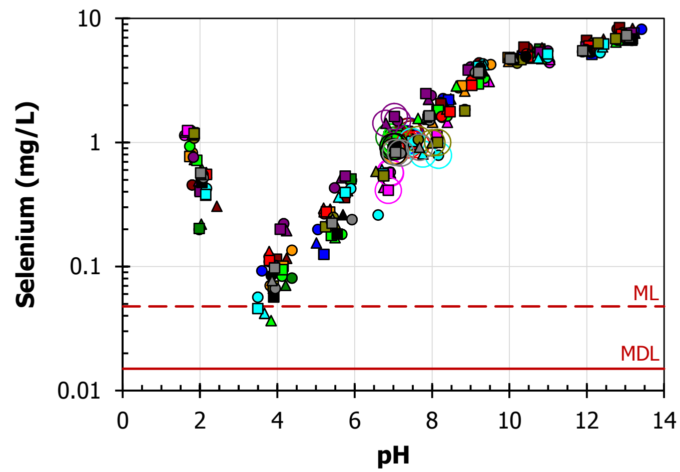
Log$_{10}$-Transform of Test Output

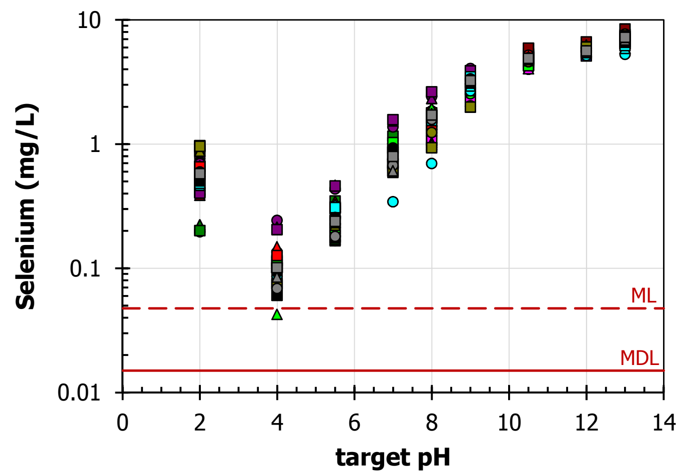
- Method 1313 – Eluate Concentration
- Method 1314 – Eluate Concentration, Cumulative Mass Release
- Method 1315 – Interval Mass Flux, Cumulative Mass Release
- Method 1316 – Eluate Concentration

Linear Interpolation and Extrapolation

- Collected Data Shows Variability
- Brings Data to Specified pH, L/S or Time
- Consistency in Comparisons

Implications for Compliance Standards



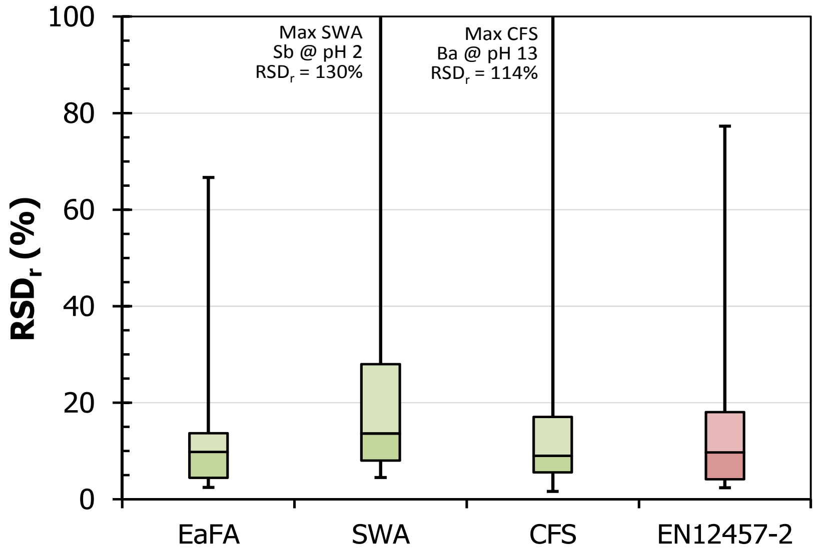


LEAF Method Precision

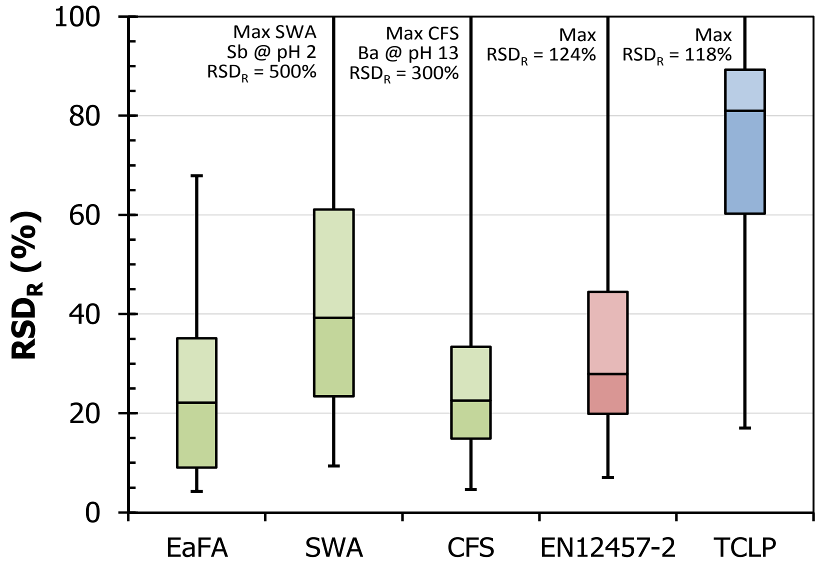
Method	Test Output	RSD_r (%)	RSD_R (%)
Method 1313	Eluate Concentration (average over pH range)	10	26
Method 1314	Eluate Concentration (9th fraction at L/S=10) Mass Release (cumulative to L/S=0.5) Mass Release (cumulative to L/S=10)	13 7 5	28 18 14
Method 1315	Interval Flux (average excluding wash-off) Mass Release (cumulative to 7-days) Mass Release (cumulative to 63-days)	11 9 6	28 19 23
Method 1316	Eluate Concentration (average over L/S range)	7	17

Precision Comparison
(pH-dependence Tests)

Repeatability



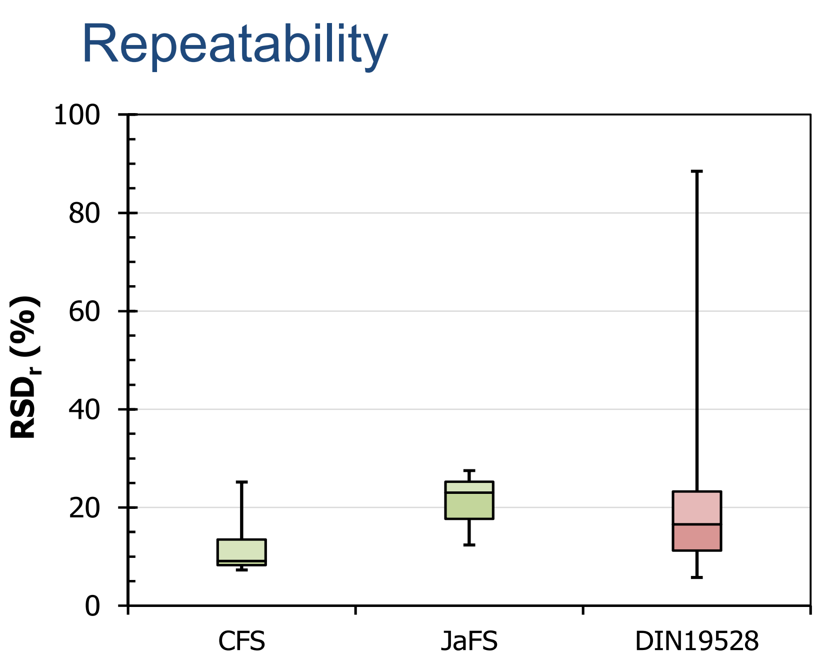
Reproducibility

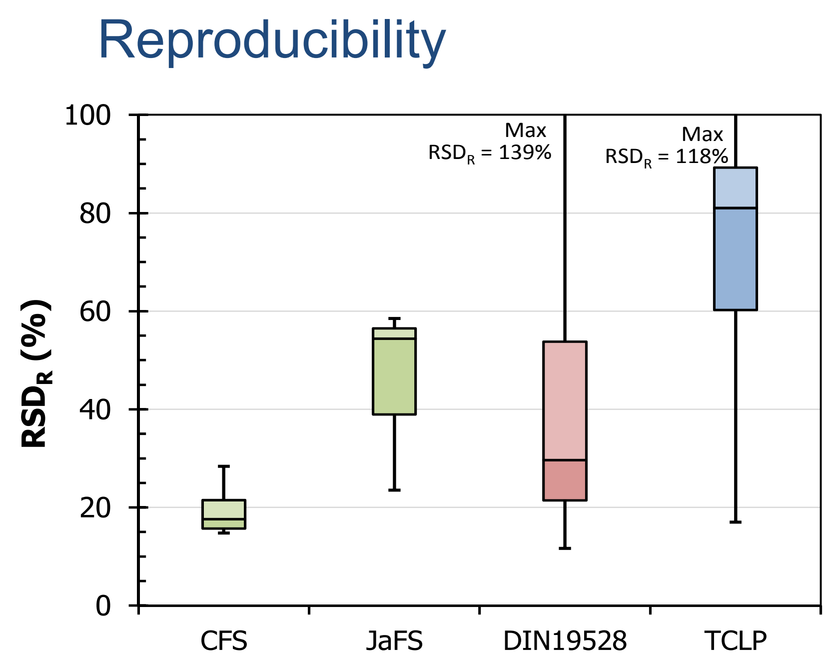


Method Precision

- Method 1314 Eluate Concentration ($2 \le pH \le 13$)
- EN12457 Eluate Concentration (natural pH)
- TCLP Eluate Concentration (acetic acid buffer)

Precision Comparison
(Percolation Tests)

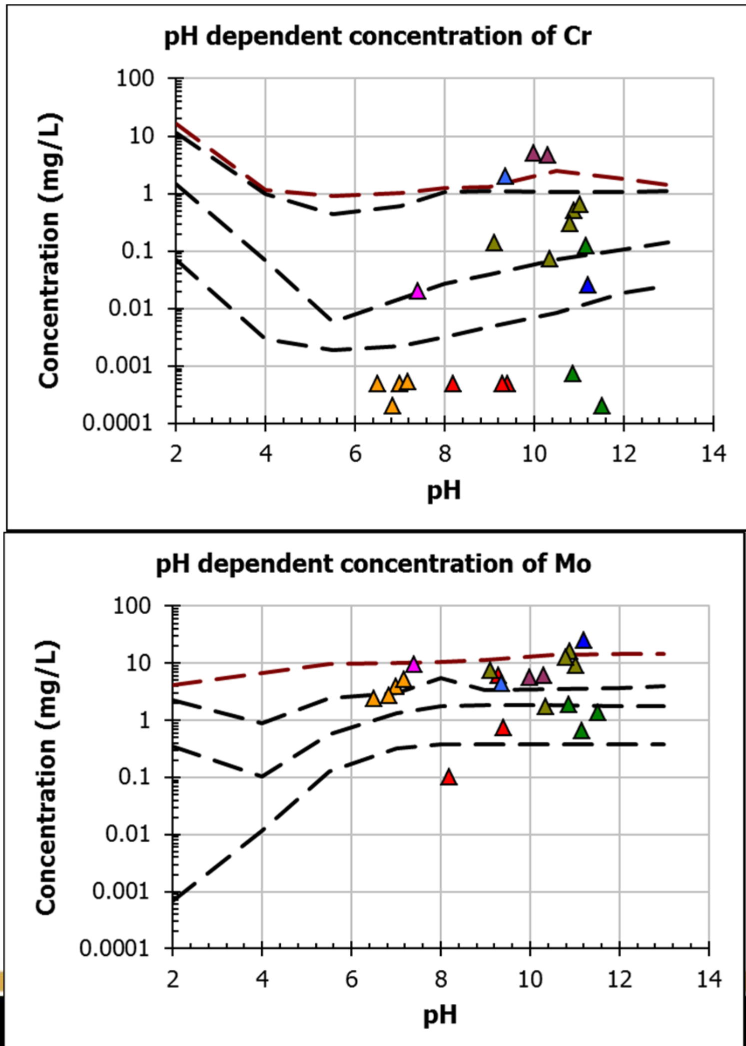


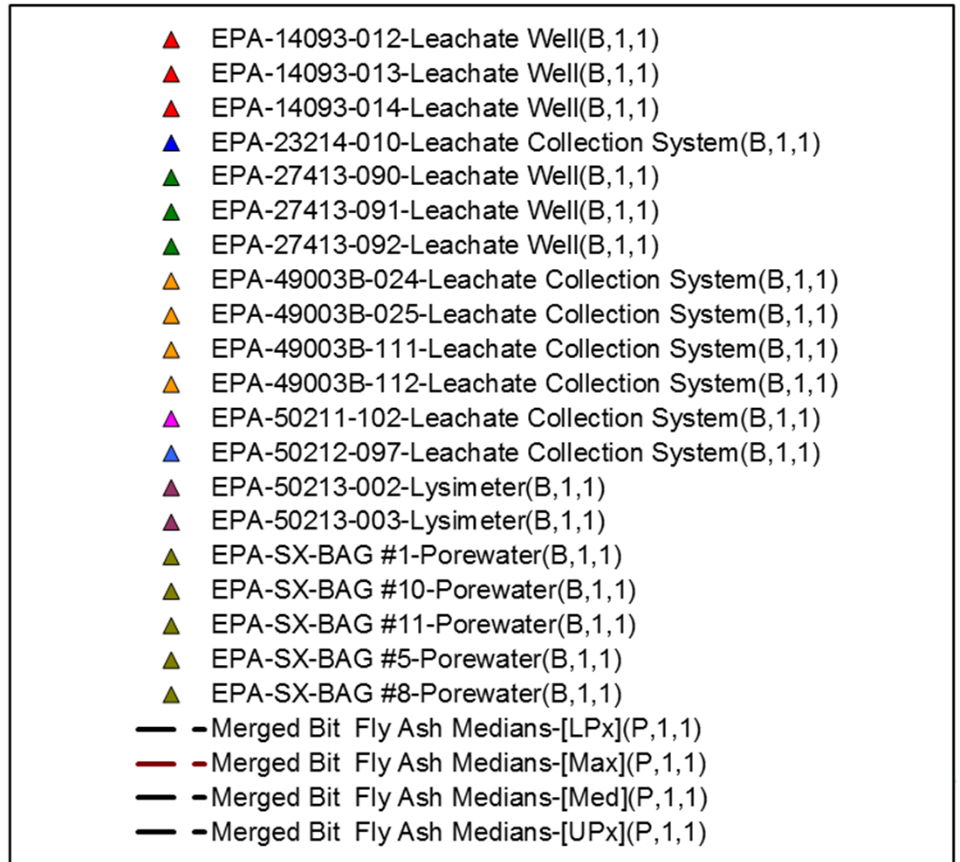


Method Precision

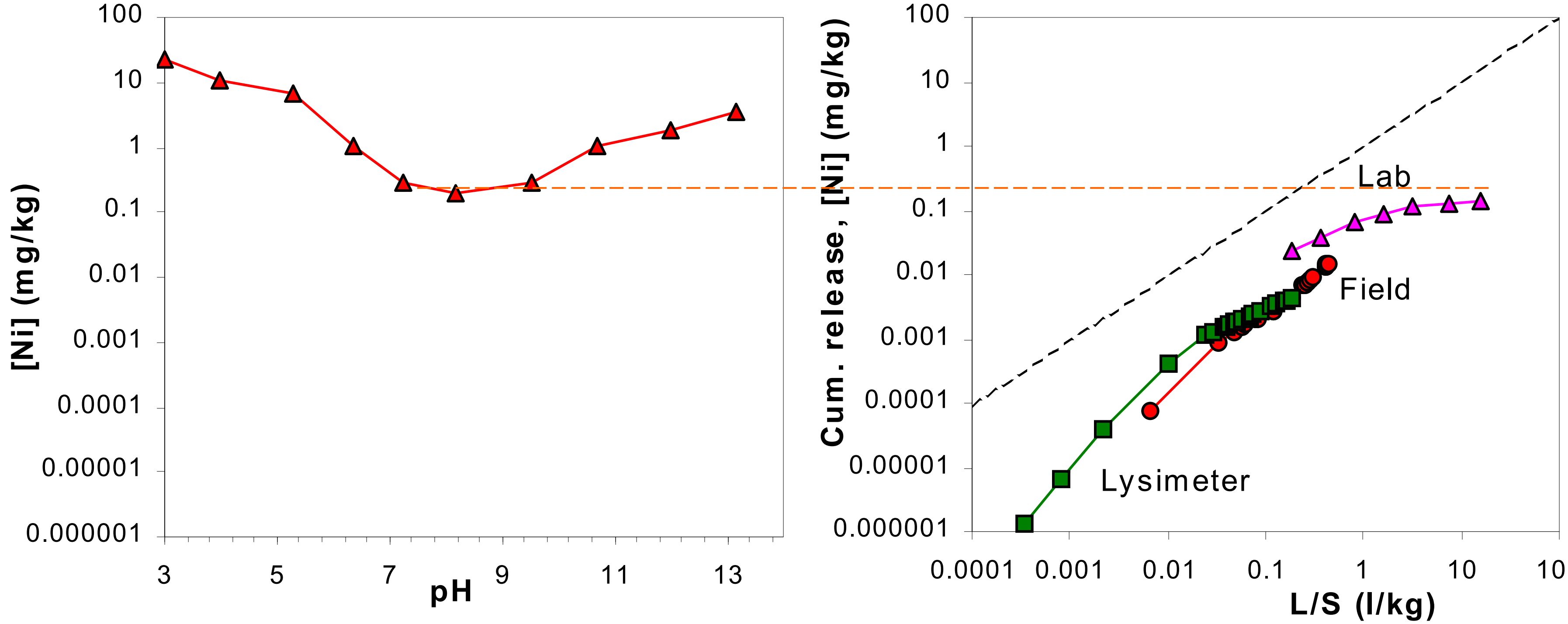
- Method 1314 Cumulative Release at L/S = 10 L/kg
- DIN 19528 Cumulative Release at L/S = 4.0 L/kg
- TCLP Eluate Concentration (L/S = 20 L/kg)

EPA Database Field leachate samples for fly ash only comparison with LEAF (EPA-600/R-09/151) All Fly Ash 5th, 50th, 95th percentiles and maximums



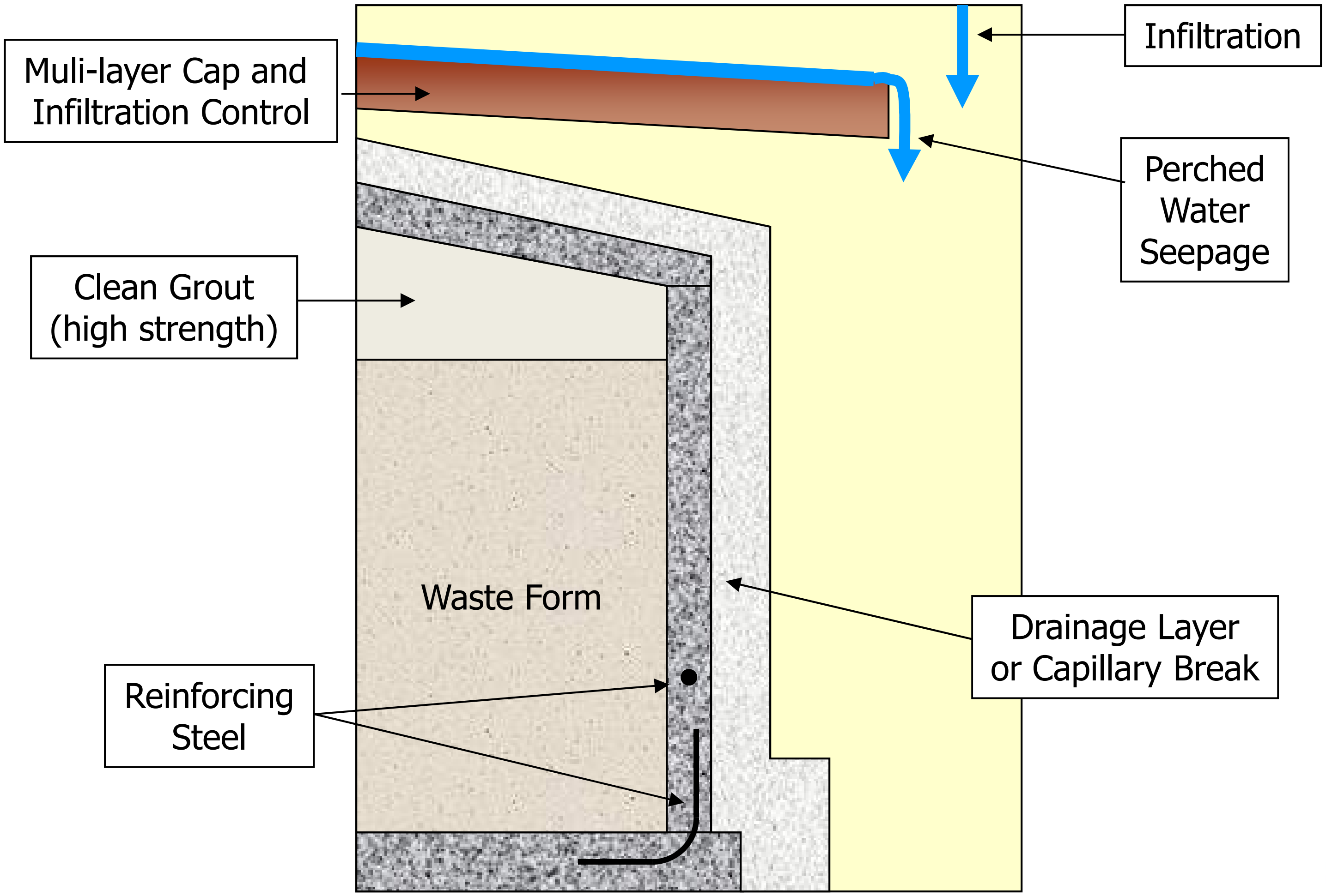


Comparison of Laboratory and Field Results



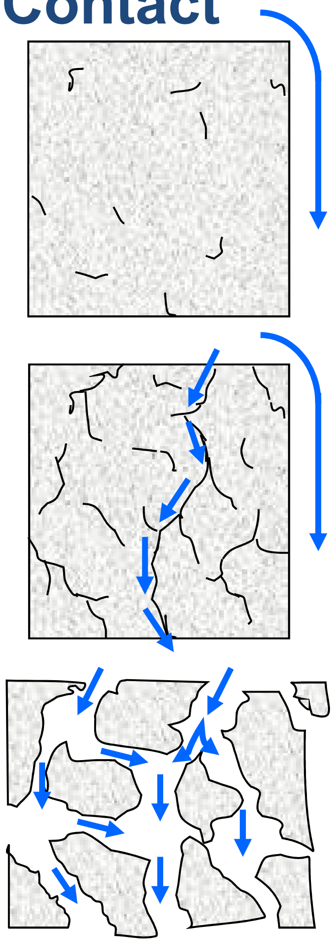
Consistent behaviour in different scales of testing

Generic Vault Disposal System



Processes and Impacts

Physical Integrity & Water Contact



Monolithic Matrix
- Flow-around
- Low interfacial area
- Diffusive release

Stressed Matrix
- Flow-around/through
- Higher interfacial area
- Diffusion-convection

Spalled Matrix
- High permeability
- Very high interfacial area
- Equilibrium-based release

Conceptual Model

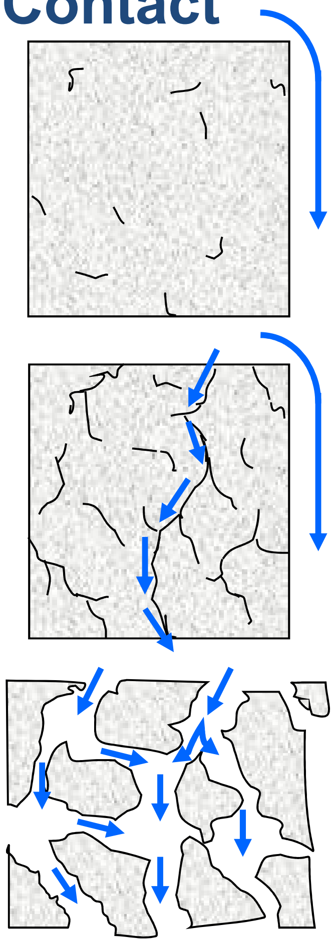
- Micro-cracks develop, increasing solid-liquid surface area
- Bridging of micro-cracks create macro-cracks
- Through-cracks develop over time, leading to convective flow
- Ultimate end state may be permeable matrix – release based on local equilibrium

Impact

- Need to account for the sequence of physical states and rate of changes
- Influences chemical reactions and constituent release
- Both "intact" & "degraded" cases are simplistic and may not be realistic

Processes and Impacts

Physical Integrity & Water Contact



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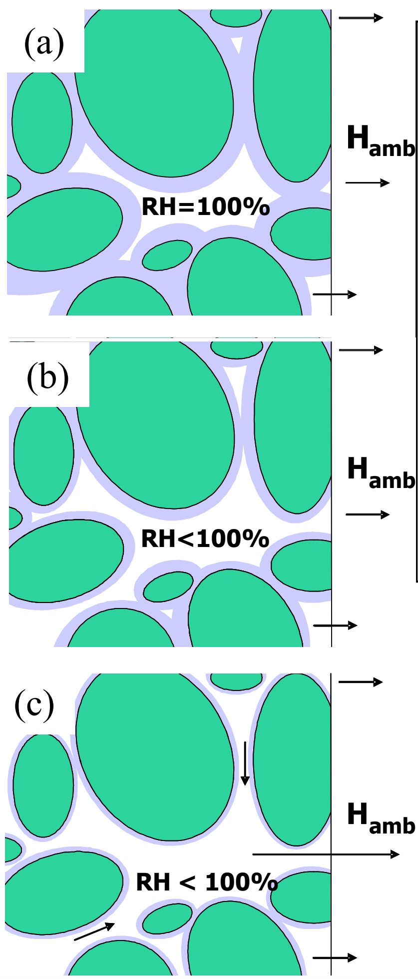
Needed Information

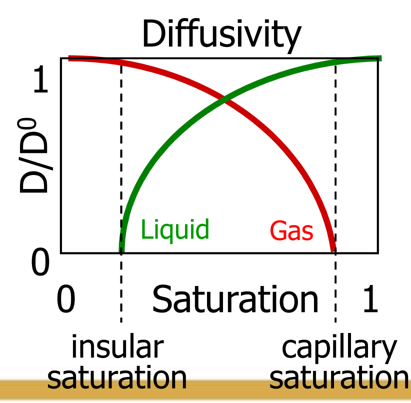
- External stresses
  - External loads
  - Differential settlement
- Material strength (e.g., Young's Modulus)
- Material pore structure
- Internal stresses
  - Shrinkage/dehydration
  - Expansive reactions within pores

Processes and Impacts

Moisture Transport



- **Full Saturation**
- **Capillary Saturation (a)**
  - Continuous Liquid
  - Discontinuous Gas
- **Transition Zone (b)**
  - Continuous Liquid
  - Continuous Gas
- **Insular Saturation (c)**
  - Discontinuous Liquid
  - Continuous Gas
- **Completely Dry**



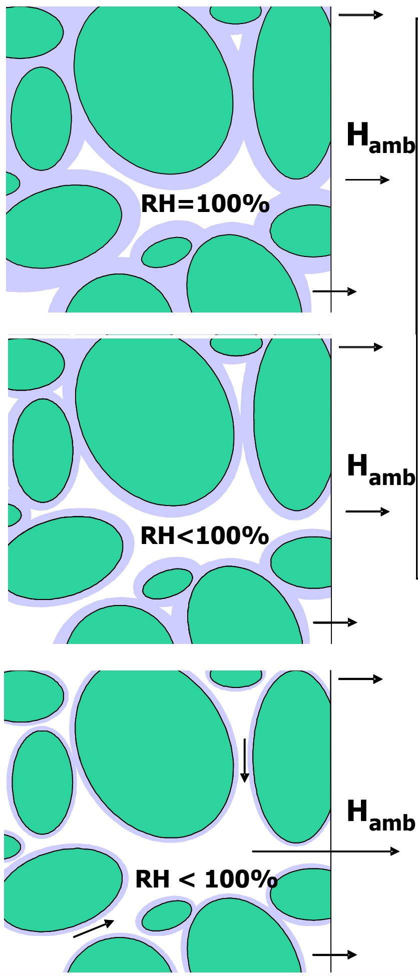
Conceptual Model

- Waste form consumes water via hydration reactions
- Moisture exchange w/environment
  - Evaporation/condensation
  - Capillary suction
  - Intermittent wetting (precipitation)
  - Percolation (degraded matrix)
- Water content determines
  - Gaseous degradation processes (oxidation, carbonation)
  - Constituent diffusion pathways

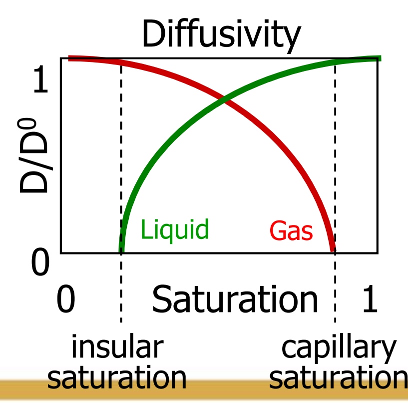
⇨ Impact

- Spatial & temporal moisture gradients
- Diffusivities are not constant over moisture regime
- Fractional saturation
  - Increases the importance of gas phase transport & reactions

Moisture Transport



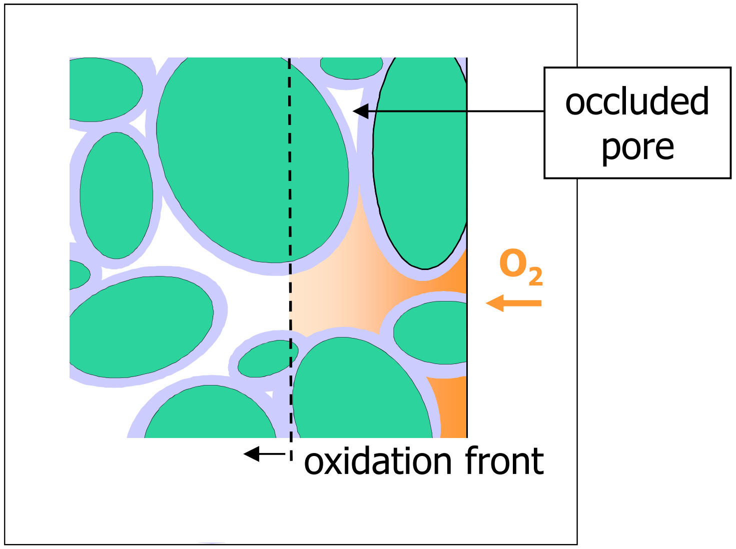
- **Full Saturation**
- **Capillary Saturation**
  - Continuous Liquid
  - Discontinuous Gas
- **Transition Zone**
  - Continuous Liquid
  - Continuous Gas
- **Insular Saturation**
  - Discontinuous Liquid
  - Continuous Gas
- **Completely Dry**



Needed Information

- Water producing & water consuming reactions
- Water retention curves (capillarity)
- Relative humidity-material saturation equilibrium
- Drying rates
- Permeability (water)
- Boundary conditions
  - Episodic infiltration
  - Relative humidity

Oxidation
Rates and Extent



	Air	Water	Ratio (A/W)
D_{O2} [cm²/s]	0.21	0.000019	1.1E+04
Conc of O_2 [mole/L]	8.9E-03	2.6E-04	1.4E+01

(1) Wilke and Chang, 1955
(2) www.swbic.org/education/ env-engr/gastransfer/gastransf.html

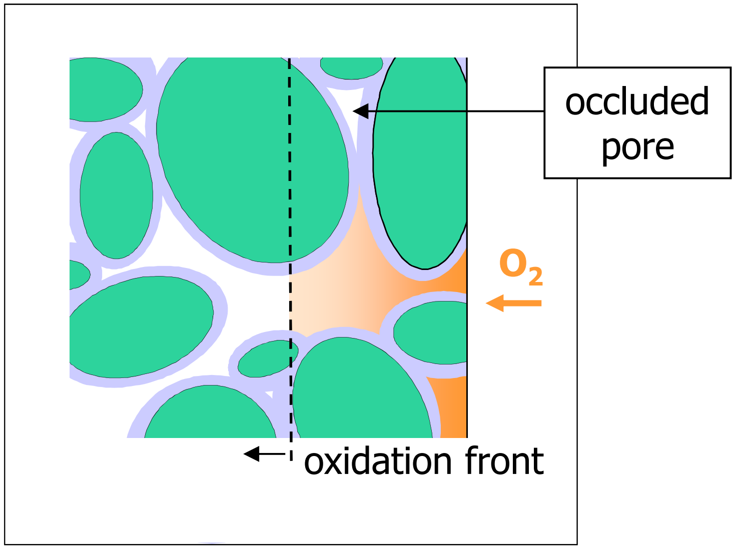
Conceptual Model

- Waste form pores – two phase system of gas and liquid; depends on moisture content (saturation)
- O_2 transport via gaseous diffusion may be important depending on saturation.
- Oxidation may lead to change in leaching behavior
  - Increased Tc-99 release; other redox sensitive constituents

Impact

- Gas phase transport must be considered
  - Flux of O_2 (gas) ~10^5 > liquid phase flux

Oxidation
Rates and Extent

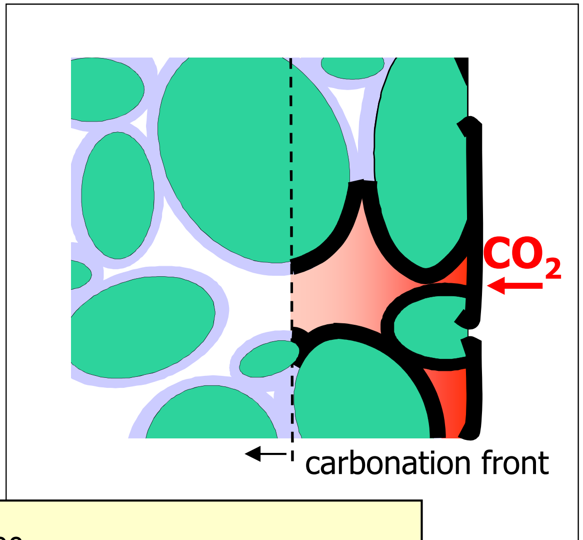


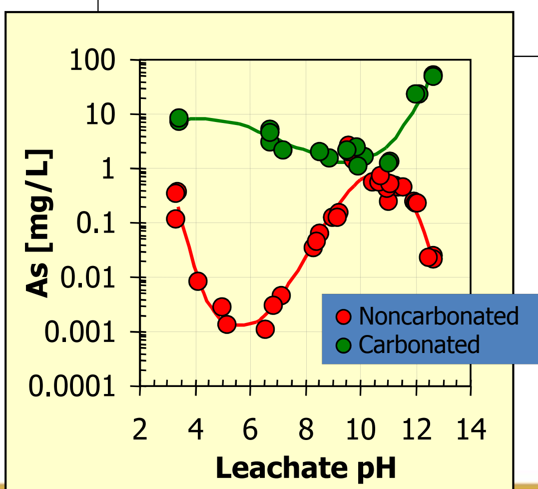
Needed Information

- Reducing capacity & redox titration
- Moisture status
- Gas phase diffusivity f(saturation)
- Liquid phase diffusivity f(saturation)
- Boundary conditions

Processes and Impacts

Carbonation





Conceptual Model

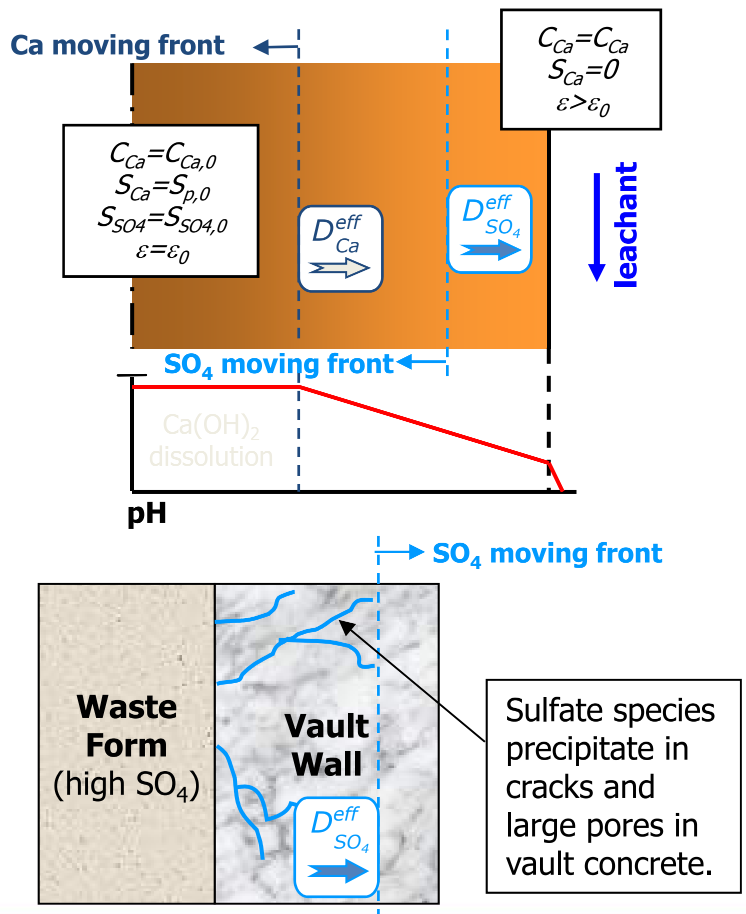
- $CO_3^{-2} + Ca^{+2} \rightarrow CaCO_3$ (s)
  - Gas phase diffusion of CO_2
  - Liquid phase diffusion of HCO_3^-
- Pore water pH decreased
  - Alters solubility of constituents (increase or decrease depending on species).
- Carbonation
  - Expansive precipitate – internal stress (cracking)
  - Pore blocking – increases diffusional resistance (decreases oxidation, release rates).
  - Extent and pore effects depend on waste form alkalinity and saturation

Impact

- Potential for speciation changes (e.g., As)
- Impact on sorption sites
- Pore structure changes
- May have either positive (e.g., pore capping) or detrimental (i.e., increased solubility) impacts

Processes and Impacts

Leaching of Major Constituents



Conceptual Model

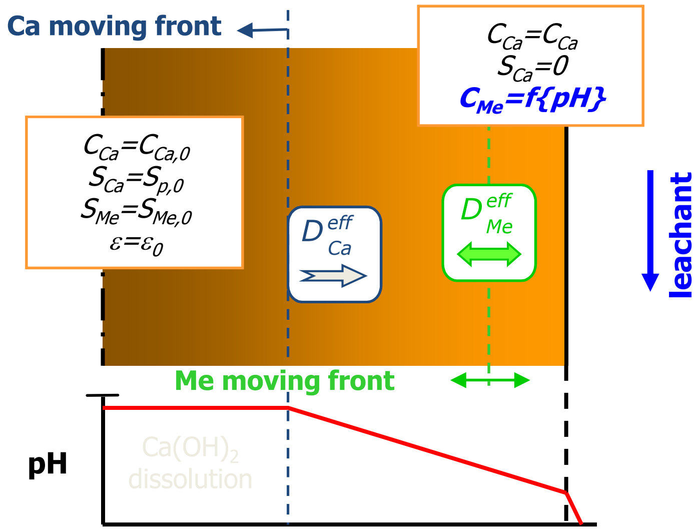
- Transport described by moving dissolution fronts
- Precipitation/reaction processes near external boundaries may significantly impact release (+ or -)
- Dissolution/diffusion of $Ca(OH)_2$ and CSH control pore water pH
  - pH gradients alter trace species release
- SO_4 leaching from waste into vault attacking concrete physical structure.
- Source of SO_4 may be waste or external environment

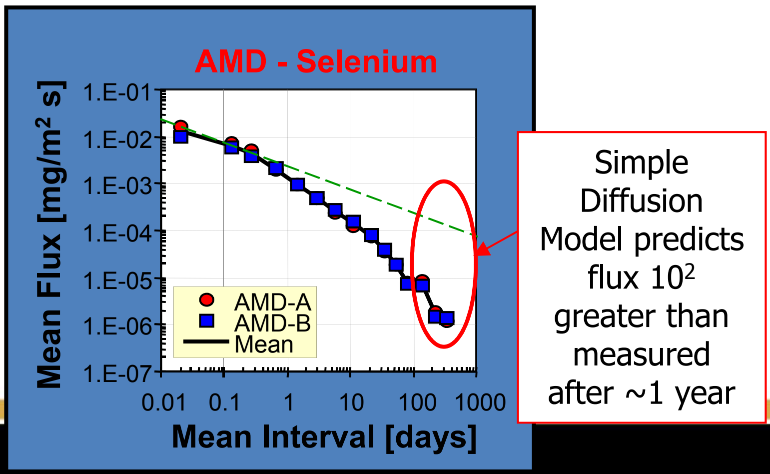
Impact

- Mass transport estimates may not reflect the dynamic chemistry and mineralogy of the waste form.
- Release rates and extents mechanistically different from simplified assumptions, effecting predictability.

Processes and Impacts

Leaching of Trace Constituents





Conceptual Model

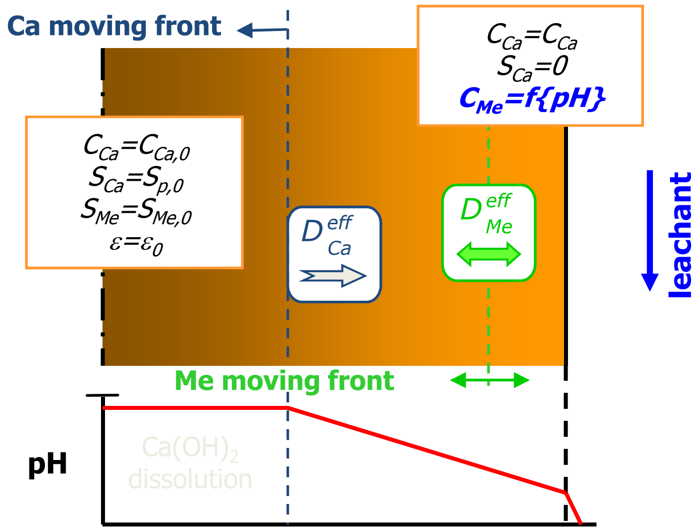
- Release based on coupled chemistry and mass transport.
- Release dependent on:
  - ❑ Moisture conditions
  - ❑ pH gradients
  - ❑ Redox chemistry
  - ❑ Boundary layer formation

Impact

- Performance assessments may over- or under-predict release

Processes and Impacts

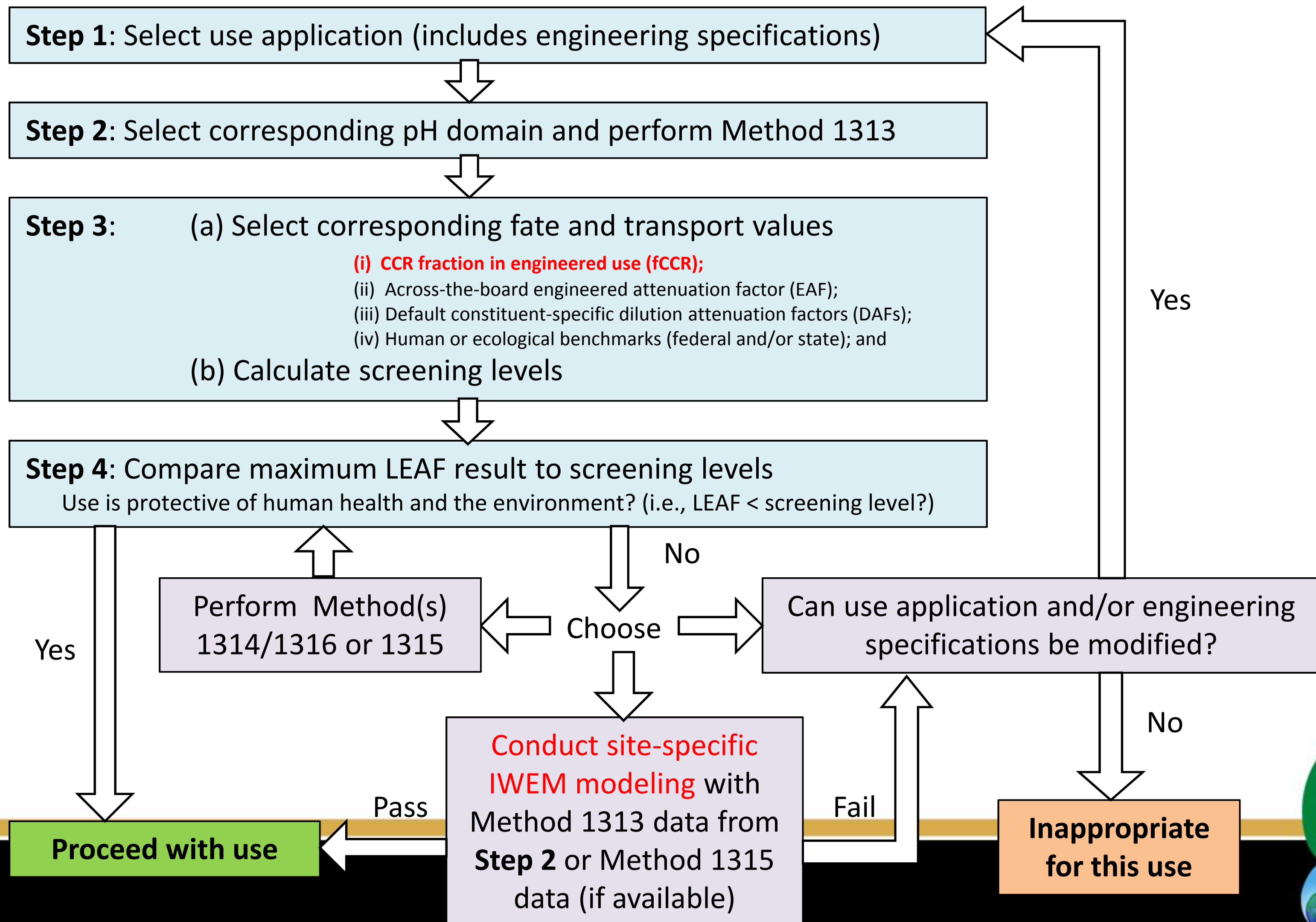
Leaching of Major & Trace Constituents



Needed Information

- pH dependent equilibrium
- Column test
  - ❑ Pore water
  - ❑ LS evolution
- Analysis of
  - ❑ pH, EC, Eh
  - ❑ Full suite of cations and anions
  - ❑ TOC, TIC, DOC, DIC
- Boundary Conditions!

A Possible Approach to Beneficial Use Screening Levels



Supporting Documentation

H.A. van der Sloot, D.S. Kosson, A.C. Garrabrants and J. Arnold *The Impact of Coal Combustion Fly Ash Used as a Supplemental Cementitious Material on the Leaching of Constituents from Cements and Concretes*, draft report in administrative review (submitted Nov 2011).

A.C. Garrabrants, D.S. Kosson, L. Stefanski, R. DeLapp, P.F.A.B. Seignette, H.A. van der Sloot, P. Kariher and M. Baldwin *Interlaboratory Validation of the Leaching Environmental Assessment Framework (LEAF) Leaching Tests for Inclusion into SW-846: Method 1313 and Method 1316*, draft report in administrative review (submitted Nov 2011)

A.C. Garrabrants, D.S. Kosson, H.A. van der Sloot, F. Sanchez, and O. Hjelmar (2010) *Background Information for the Leaching Environmental Assessment Framework Test Methods*, EPA/600/R-10/170, December 2010; http://www.epa.gov/nrmrl/pubs/600r10170/600r10170.pdf.

S.A. Thorneloe, 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 & Technology*, 44(19), 7351-7356, http://pubs.acs.org/doi/pdfplus/10.1021/es1016558.

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F. Sanchez, D. Kosson, R. Keeney, R. DeLapp, L. Turner, P. Kariher, and S. Thorneloe (2008) *Characterization of Coal Combustion Residues from Electric Utilities Using Wet Scrubbers for Multi-Pollutant Control,* EPA-600/R-08/077, July 2008; www.epa.gov/nrmrl/pubs/600r08077/600r08077.pdf.

F. Sanchez, R. Keeney, D. Kosson, R. Delapp and S. Thorneloe (2006) *Characterization of Mercury-Enriched Coal Combustion Residues from Electric Utilities Using Enhanced Sorbents for Mercury Control*, EPA-600/R-06/008, February 2006; http://www.epa.gov/ORD/NRMRL/pubs/600r06008/600r06008.pdf.

D.S. Kosson, H.A. van der Sloot, F. Sanchez, and A.C. Garrabrants (2002) "An integrated framework for evaluating leaching in waste management and utilization of secondary materials," *Environmental Engineering Science*, 19(3), 159-204.

Benefits to Use of LEAF

- Provides standardization in leach testing and comparability in resulting data for use across different materials and management scenarios
- Analytical work complete for all four methods as part of the interlaboratory validation for inclusion into SW-846
- Comparable methods being used abroad enable more robust data sets that provide better characterization across material types and management scenarios
- Provides data needed for binning materials into categories that enable more efficient beneficial use decisions (no or less stringent testing required depending upon the leaching behavior of the material)

Benefits to Use of LEAF

- Industry that generates CCRs and other industrial by-products has clarity in what is required and access to labs across the U.S.
- Provides objective and independent analysis of claims made by producers or technologies
- LEAF allows one to understand the mechanistic behavior of materials across a range of management scenarios across long terms (can not use snap shot approach that doesn't consider future environmental conditions)
- Provides robust source term for risk assessment by considering physical and chemical factors that control leaching behavior over time

Conclusions

The LEAF test methods

- Can be used to evaluate leaching behavior of a wide range of materials using a tiered approach that considers the effect of leaching on pH, liquid-to-solid ratio, and physical form
- Prepared for inclusion into SW846 – EPA's compendium of test methods for waste and material characterization
- Supporting software (LeachXS-Lite) available for data entry, analysis, visualization, and reporting
- Demonstrated relevance for assessing release behavior under field conditions for use and disposal scenarios

Current efforts

- Complete interlaboratory validation for Method 1314 and Method 1315
- Provide information on
  - ❑ Relationship between the LEAF testing results and field leaching
  - ❑ Application of LEAF test methods for evaluating CCR use and disposal

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