

Leach Testing of FGD Materials

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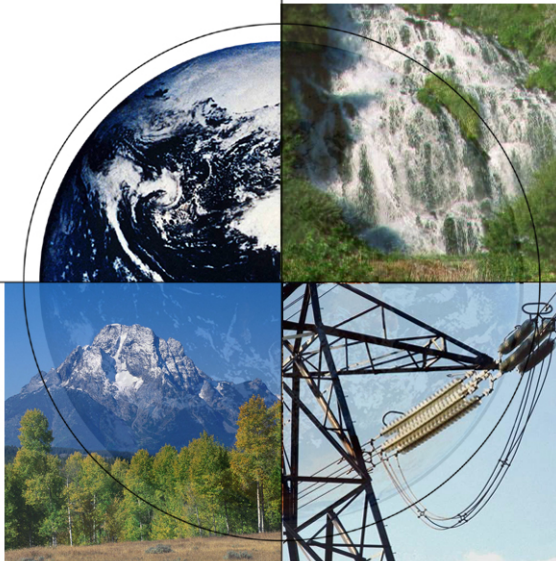
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DOE / NETL

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Objective

Project:

To determine element mobility

- Which elements are released
- How much is released
- Under what conditions
 - Liquid / solid ratio
 - Effect of pH (mildly alkaline to acidic)
- Why



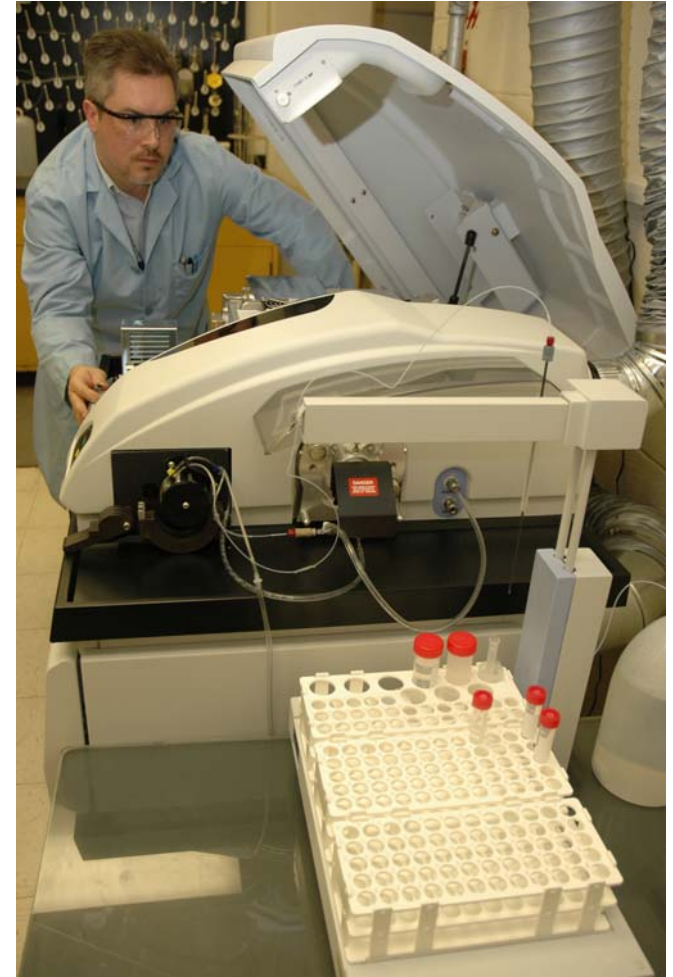
Today's Presentation:

Mercury in FGD gypsum is present in 3 forms:

- Bound to a phase containing Al, Fe or both
- A H_2O_2 oxidizable phase such as a sulfide or carbonaceous material
- Precursor to elemental mercury

Experimental

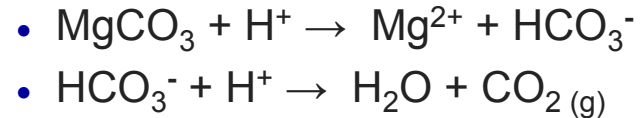
- **Sample suspended in MQW under N₂ purge**
- **Leachants**
 - MQW leach for 6 samples
 - Followed by 0.01-0.02N HCl leach
- **pH, ORP continuously monitored**
- **300 mL samples collected**
- **Leachate analysis**
 - ICP-OES
 - ICP-MS
 - CVAA or CVAF
 - QA/QC
- **Remaining material removed, filtered**
 - Solids analysis of separate splits
 - Digestion EPA 3051, EPA 3052, ASTM D6349
 - ICP-OES, ICP-MS
 - DMA-80



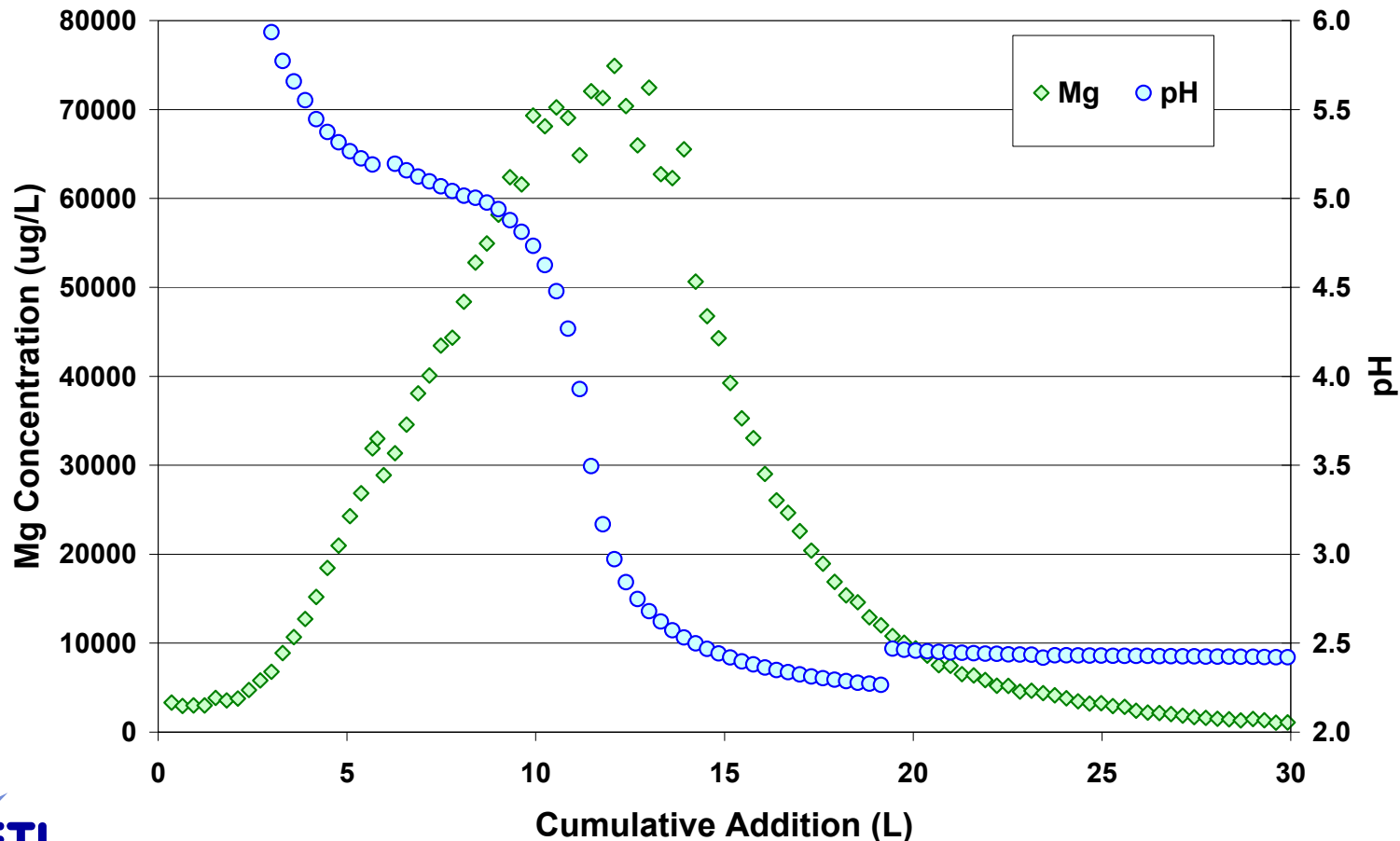
Neutralization Reaction Driven Release: Mg

- Elution indicative of acid neutralizing capacity

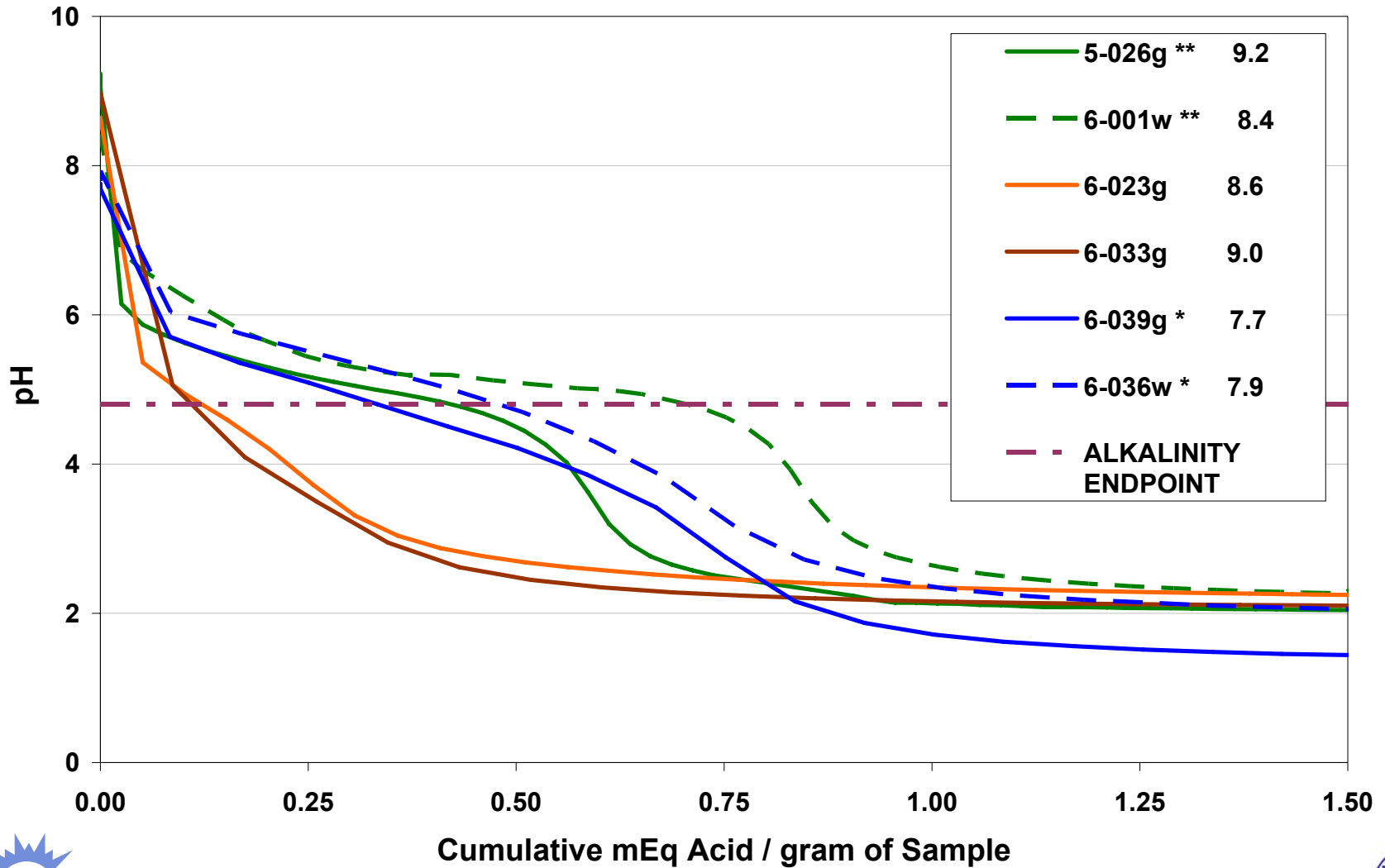
- Mg present as carbonate?



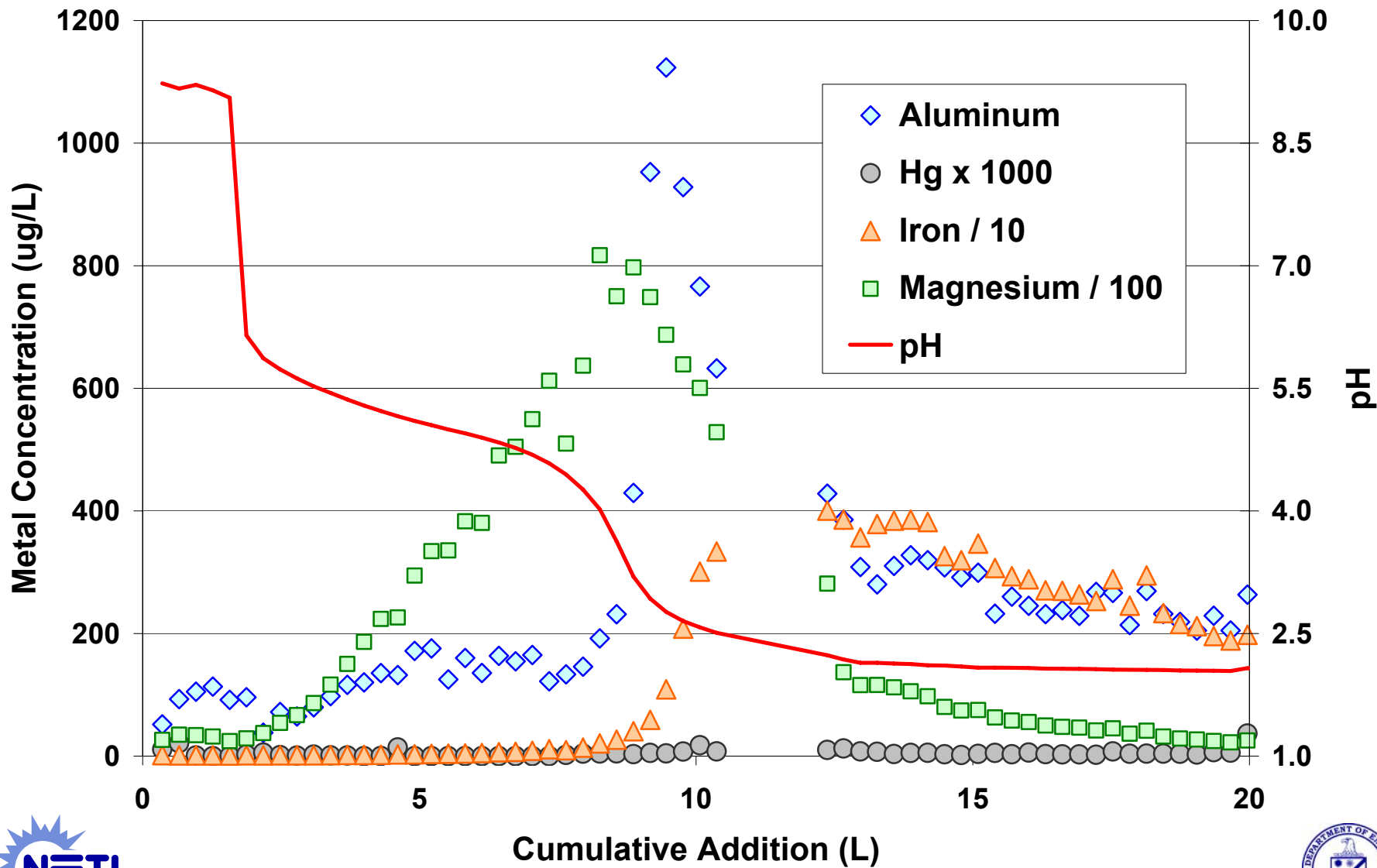
Run 6-001



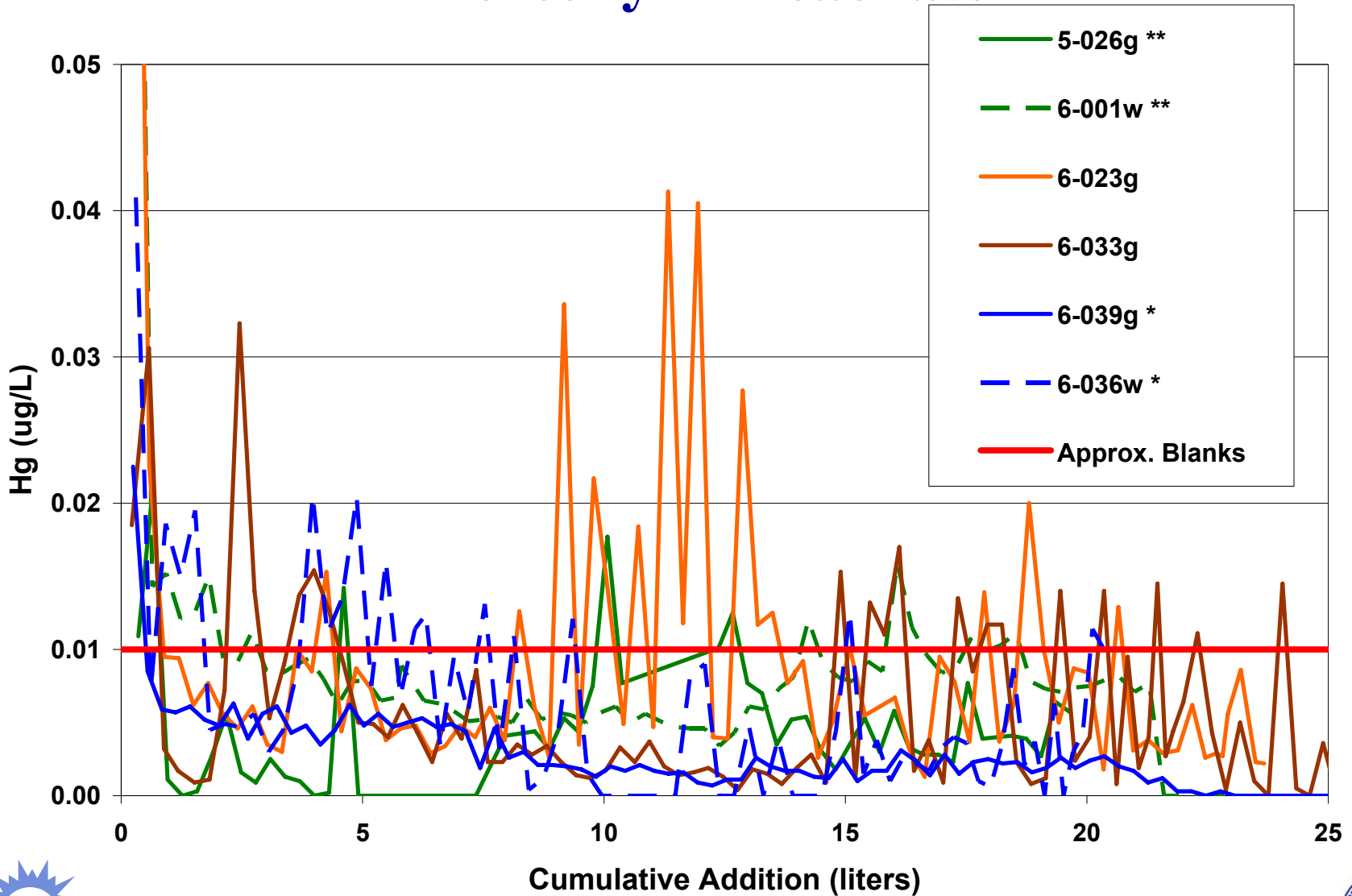
Alkalinity in FGD Products



Acid Leaching of 5-026



Mercury in Leachate



Post-leaching Residue

Table 3. Amount in residue (ug/g)

	Plant A	Plant B	Plant D	Plant E
Al	30900	40100	75400	88900
As	16.5	19.0	173	18.5
Ca	3080	1820	1900	6760
Cd	5.40	< 2	< 2	<2
Cr	222	257	287	145
Fe	28500	23800	102000	89200
Hg	33.1	6.53	57.0	54.2
Mg	7080	14100	7990	7410
Mn	118	80.0	394	171
Ni	33.2	171	134	115
Pb	29.6	25.0	39.6	40.6
S	10400	350	8710	7010
Se	54.6	< 9	< 9	276
Zn	592	285	340	139



- Accounts for < 2% of the original material
 - Phase responsible for retention is not gypsum
 - Fe or mixed Fe-Al phase

Mineral Extractions

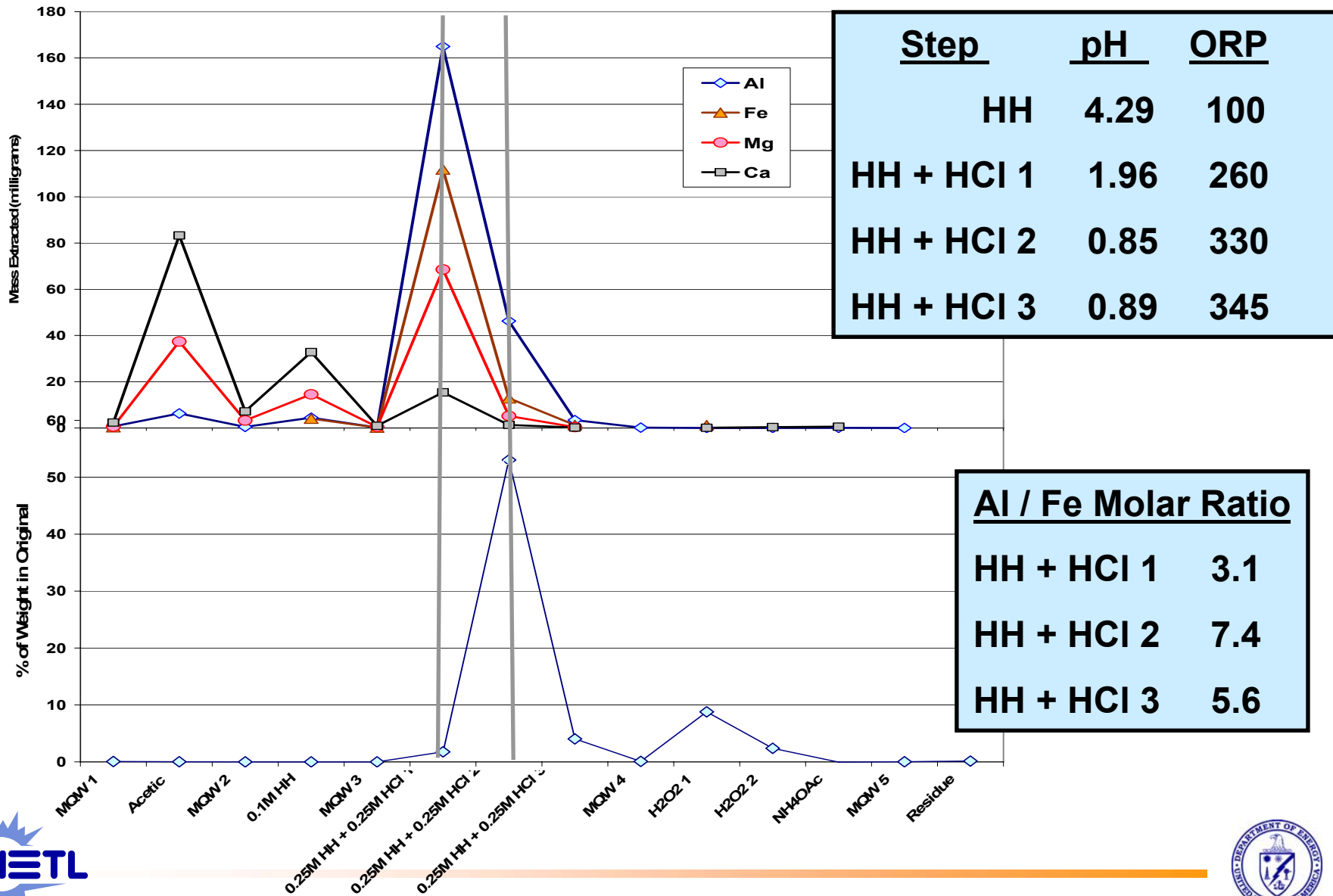
Sequential Extraction Procedure

Step	Target
• MQW Rinse	Water soluble/loosely sorbed fraction
• 0.11 M Acetic Acid	Carbonate and exchangeable fraction
• 0.1 M Hydroxylamine Hydrochloride Extraction	Manganese oxides and hydroxides
• 0.25 M Hydroxylamine Hydrochloride in 0.25 M HCl	Iron oxides and hydroxides
• Hydrogen Peroxide, Ammonium Acetate	Organic matter and sulfide mineral fraction

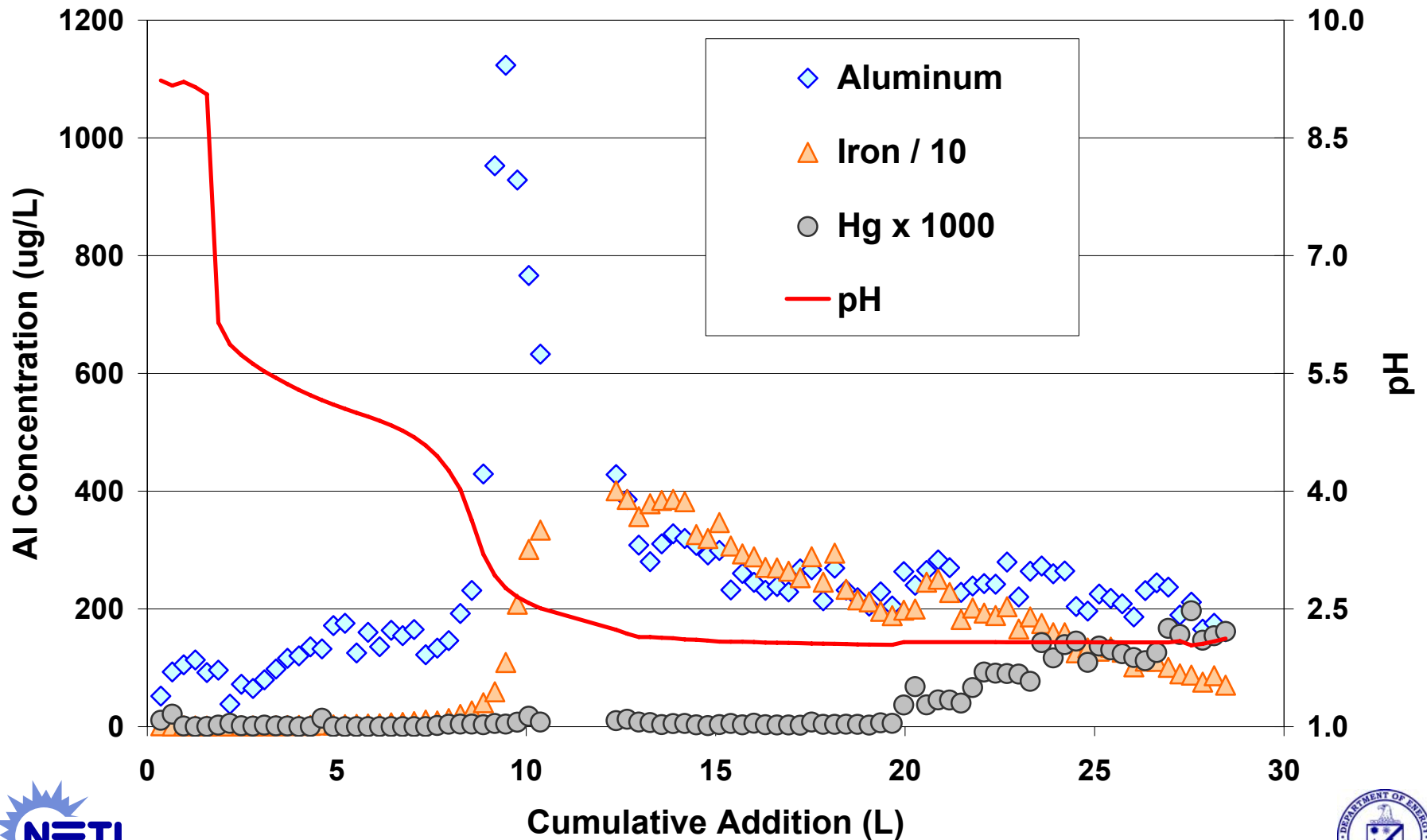
EERC has found HgCl_2 , HgO , HgSO_4 , HgS , HgSe , Hg-Carbon, and HgFeOxide as particulate-bound mercury. (Workshop class notes)



Mineral Extractions of FGD Material



Acid Leaching of 5-026 Followed by Hydroxylamine Hydrochloride Beginning at 20 L



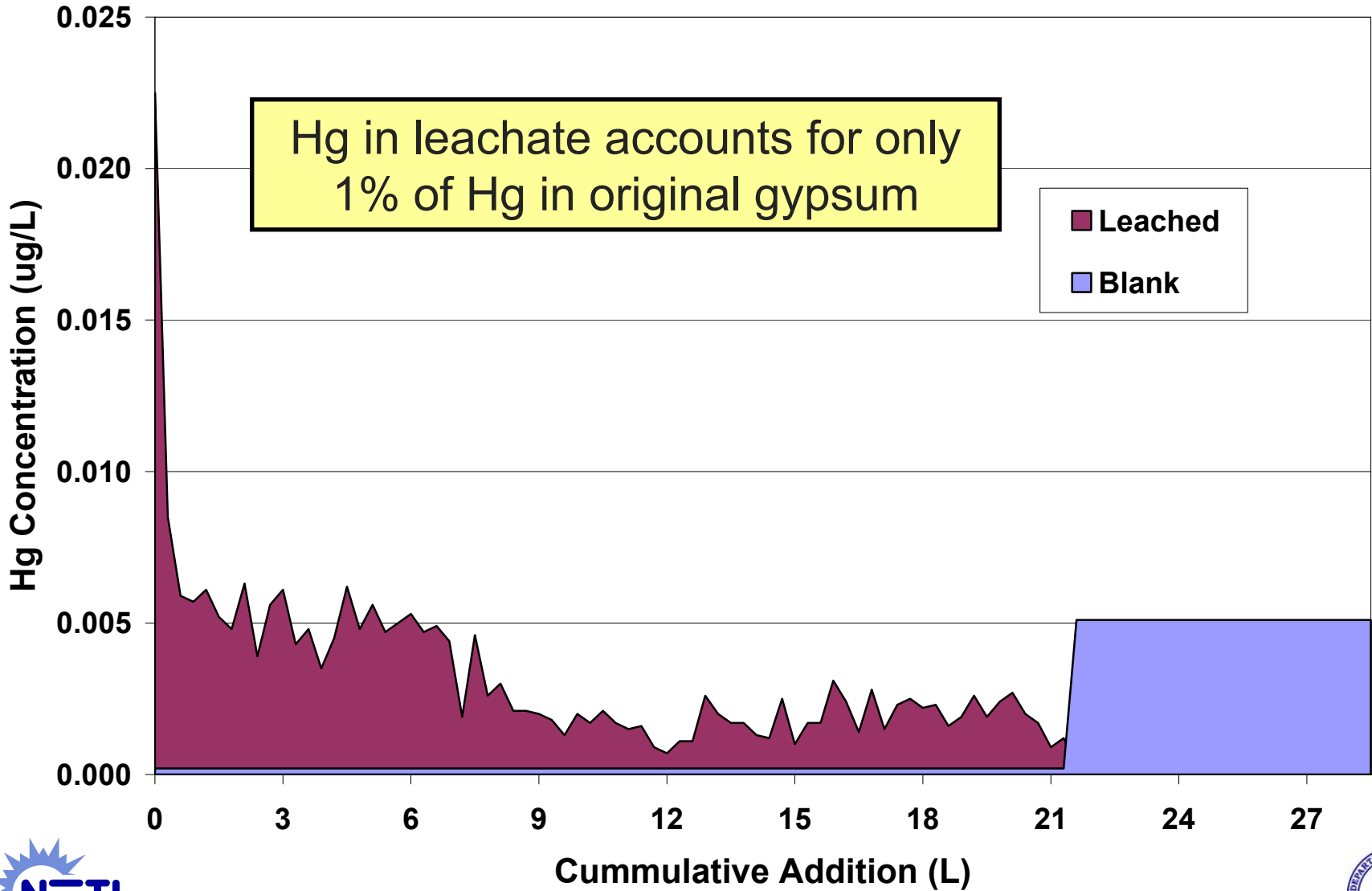
Mercury Recoveries

Site	Material	Hg material balance
Plant A	FGD gypsum	103 %
Plant A	FGD wallboard	109 %
Plant B	FGD gypsum	55 %
Plant D	FGD gypsum	56 %

FGD gypsum from plant B selected for re-examination using Hg(0) traps on the exit gas stream

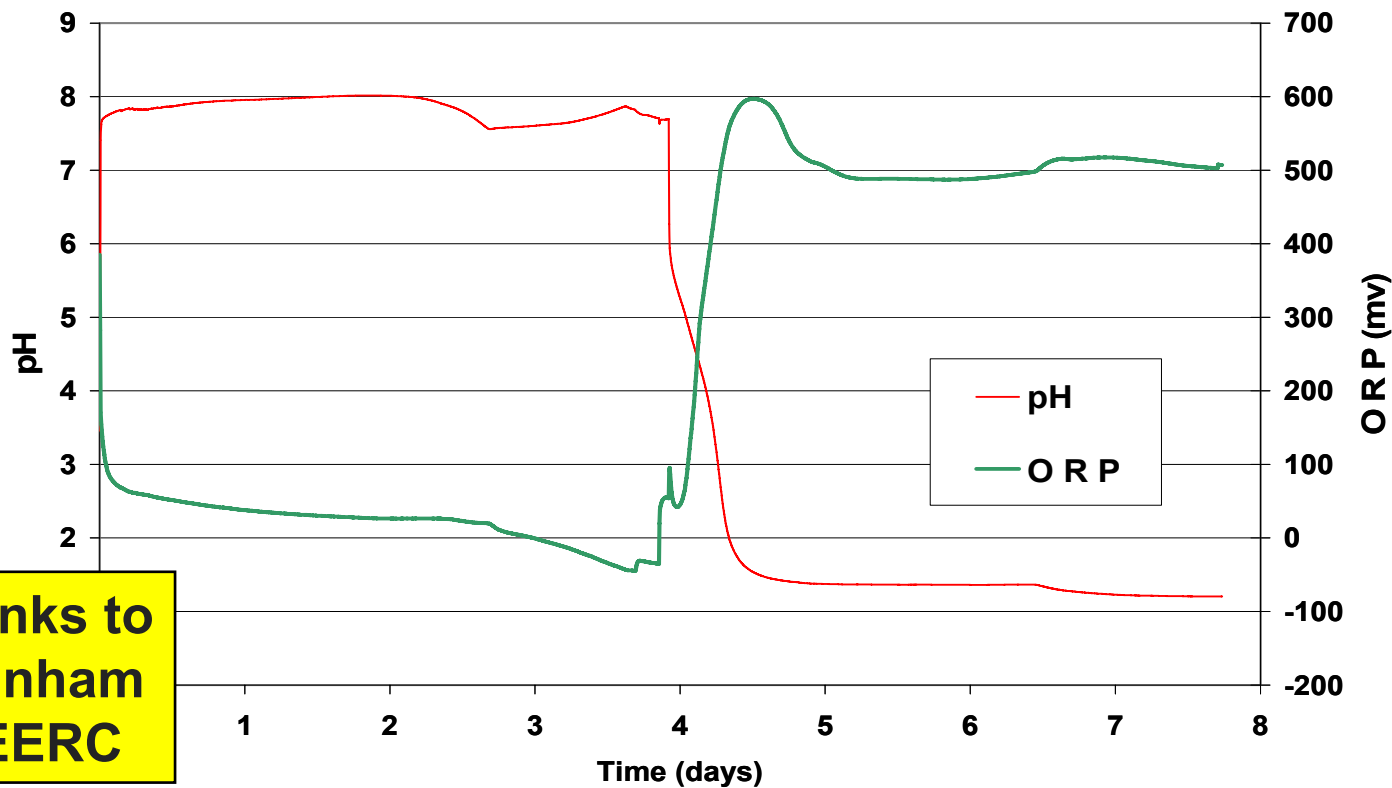


Leachate 6-039

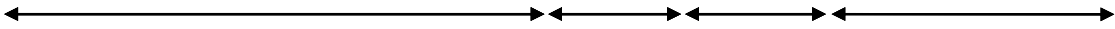


Loss of Elemental Mercury to the Gas Phase During Continuous Stirred Tank Leaching

In Tank Values
6-039



Many thanks to
Grant Dunham
at UNDEERC



Mercury Vapor Trap 1
4.49 ug

Trap 2
LOST
(1.82)

Trap 3
0.49 ug

Trap 4
< DL



Summary

- Leaching of 6 FGD materials to pH >2 under oxic conditions does not release appreciable amounts of mercury to the leachate
- Evolution of Hg(0) to the gas phase did occur for the 1 FGD gypsum studied so far
- 80% of retained Hg is in a residue high in Fe and Al
- 20% of retained Hg requires H₂O₂ oxidation for release
- If pH > 4 and ORP > 100 mv no release occurs
- If pH <1 and ORP ≈ 350 mv dissolution of the major immobilized form is complete
- The Hg-retaining phase will immobilize Hg in many applications; exception = placement in mine-land reclamation areas that will be low pH and anoxic

Conclusions and Application

- **Conclusion: Mercury in FGD gypsum is present in 3 forms**
 - Precursor to elemental mercury
 - Bound to a phase containing Al, Fe or both
 - A H_2O_2 oxidizable phase such as a sulfide or carboneous material
- **Application: Hg capture and sequestration in FGD materials**
 - Capture and eliminate from FGD gypsum
 - Al / Fe phase easily separates with the fines
 - Sequester Hg in the gypsum / wallboard to prevent subsequent release
 - Al / Fe phase is good sorbent
 - H_2O_2 oxidizable phase may be better, especially for reducing environments
 - May be possible to add sorptive material to the wallboard formula

