Caustic Leaching of SRS Sludges

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Objective

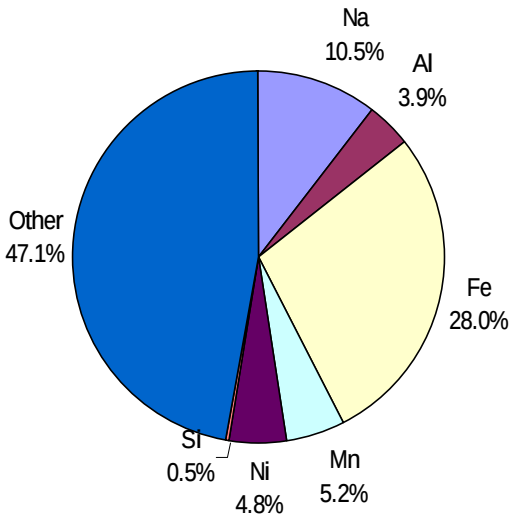
- Determine the fate of aluminum upon contact of Purex and HM sludges with
  - water (ambient temperature)
  - excess NaOH (elevated temperature)
  - excess NaOH + NaF + Na_3PO_4 (elevated temperature)

Testing Methodology

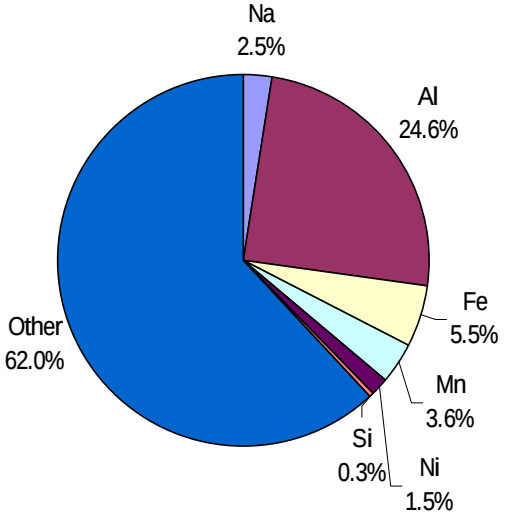
- Sludge washing
  - Mix 3 g of dried sludge with 10 mL of DDI water for 5 minutes
  - Centrifuge and decant off clear liquid
  - Repeat 2 times
  - Analyze solids
- Sludge Leaching
  - Add 50 wt% NaOH to 3 g of dried sludge to provide mole ratio for OH:Al of 3:1
  - Add inhibited water (0.01 M NaOH and 0.001 M $NaNO_2$)
  - [Add 0.03 M NaF and 0.01 M Na_3PO_4]
  - Heat to 85 – 87 °C for up to 168 hours
  - Analyze solutions and residual solids

Elemental Composition – As Found

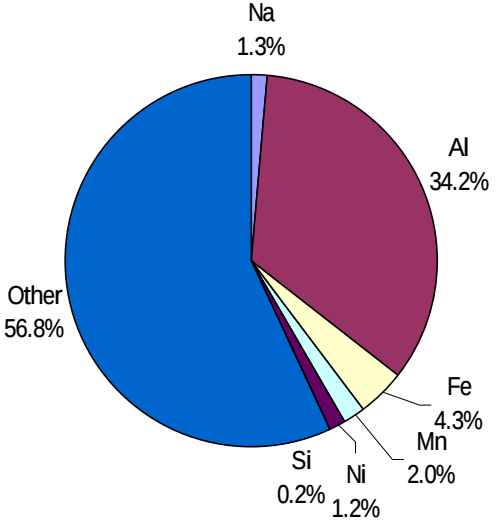
As-Found Tank 8F



As-Found Tank 11H



As-Found Tank 12H

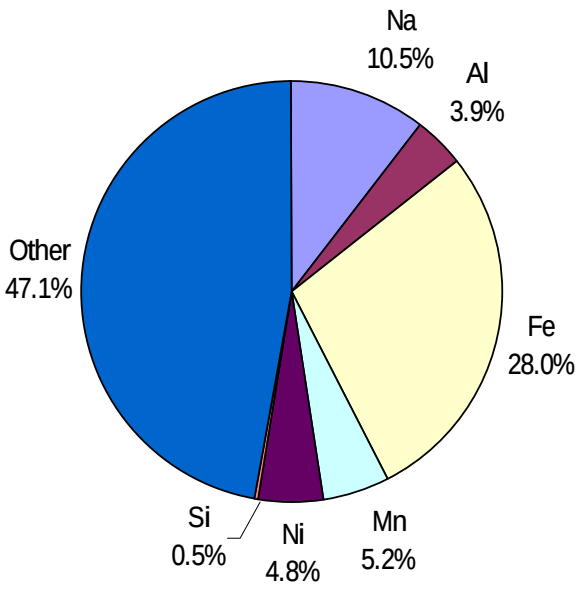


- Al and Fe content in archived sludge samples is consistent with sludges produced in Purex and HM processes

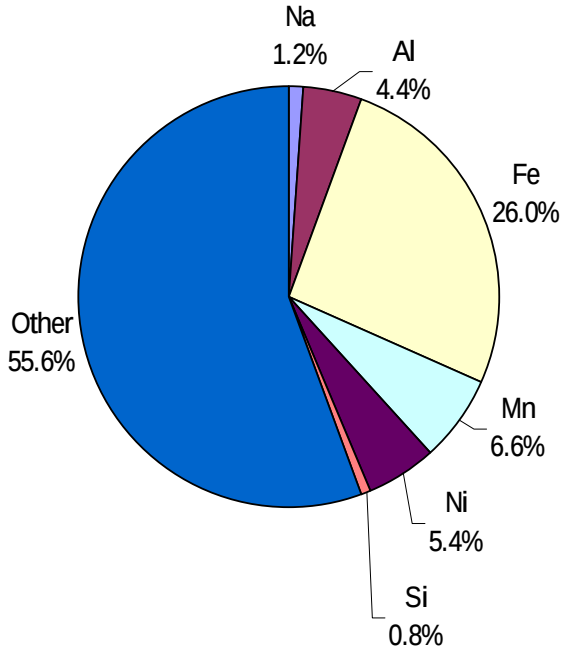
Effect of Washing – Tank 8F Sludge

- Washing significantly reduced Na content

As-Found Tank 8F



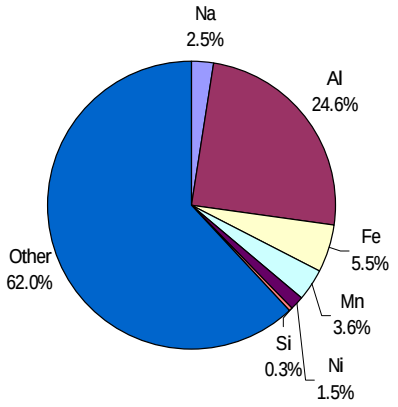
Tank 8F After Washing



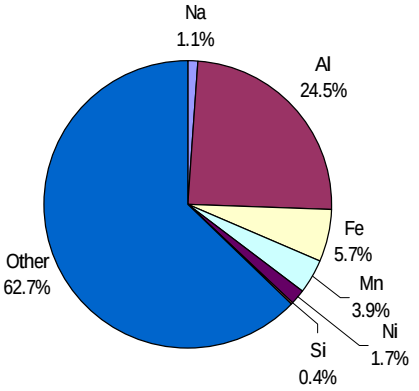
Effect of Washing – Tanks 11H and 12H Sludges

- Little or no change in elemental composition of HM sludges

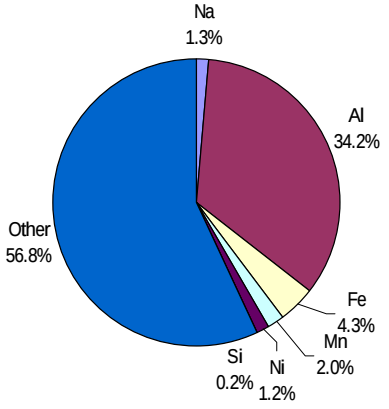
As-Found Tank 11H



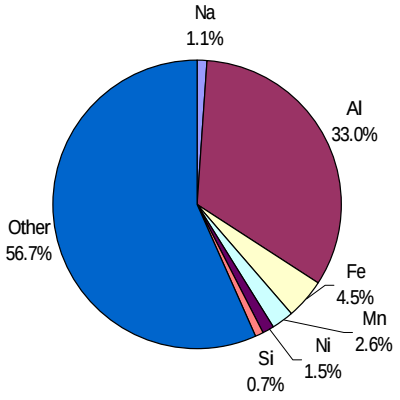
Tank 11H After Washing



As-Found Tank 12H

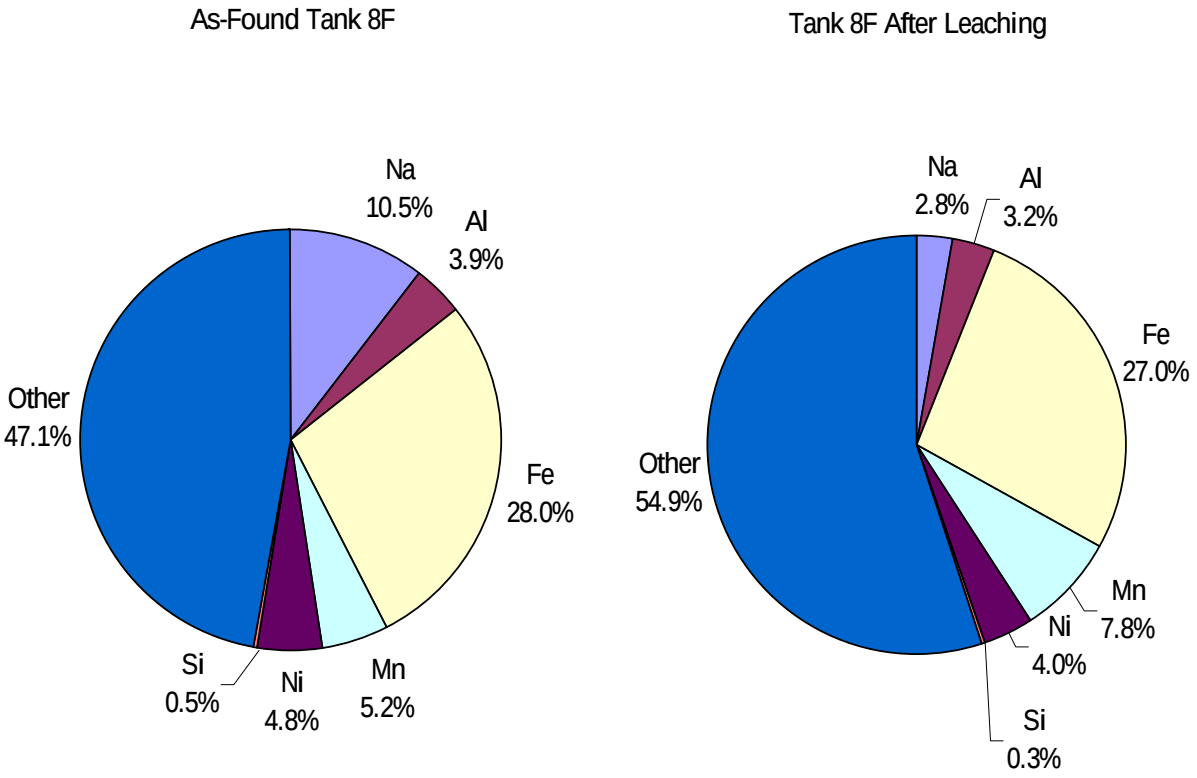


Tank 12H After Washing



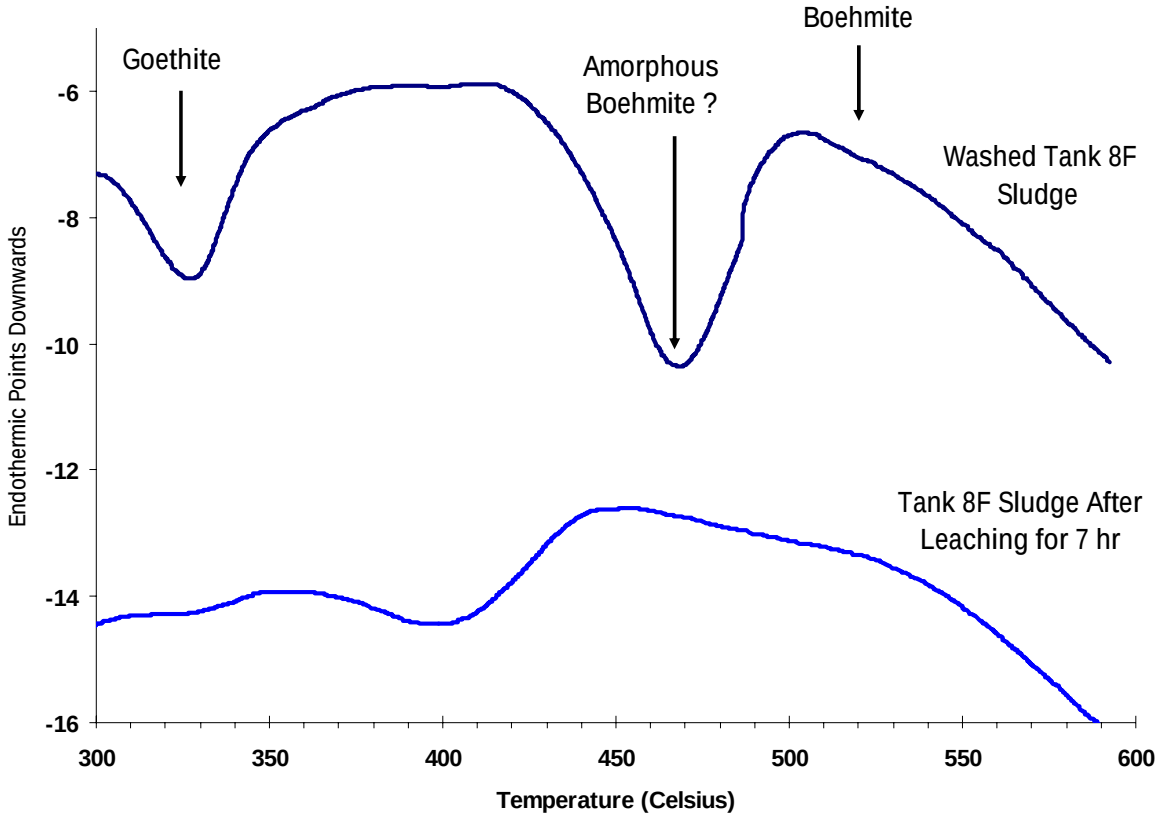
Effect of Caustic Leaching – Tank 8F Sludge

Caustic leaching removed Na and small amount of Al



Tank 8F Sludge Calorimetry

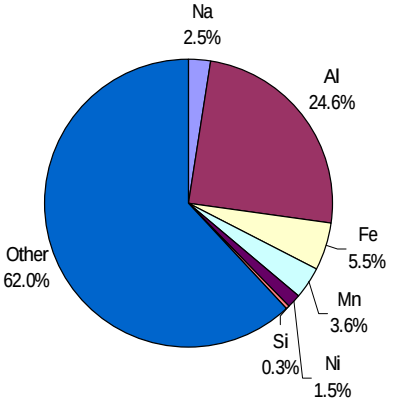
- No evidence of boehmite by DSC before or after leaching
- Goethite converted to another iron oxide phase



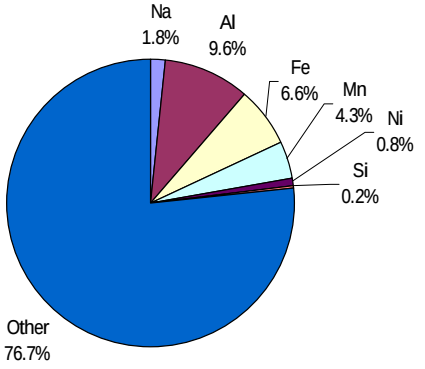
Effect of Caustic Leaching – Tanks 11H &12H Sludges

- Significant removal of Al from HM sludges
- Little change in other elements

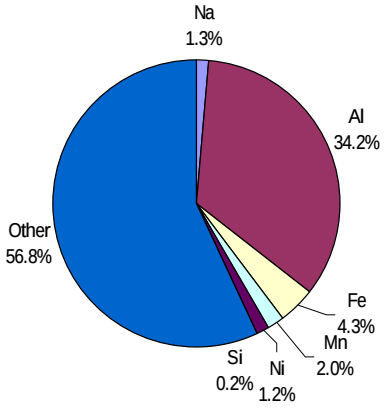
As-Found Tank 11H



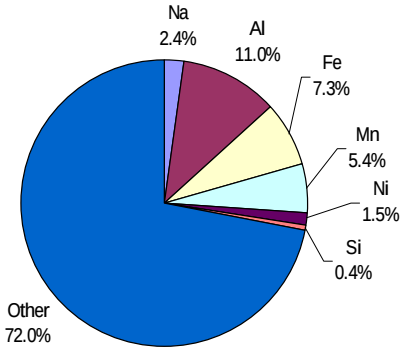
Tank 11H After Leaching



As-Found Tank 12H

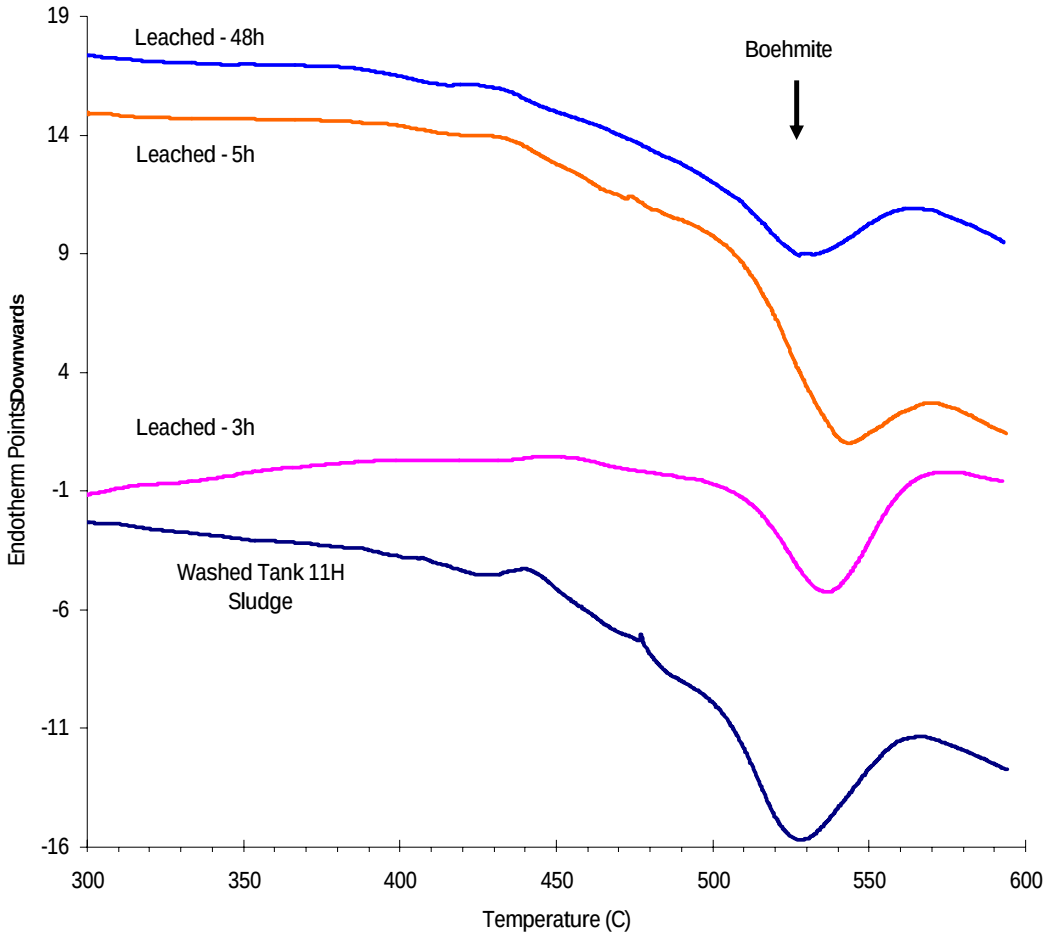


Tank 12H After Leaching



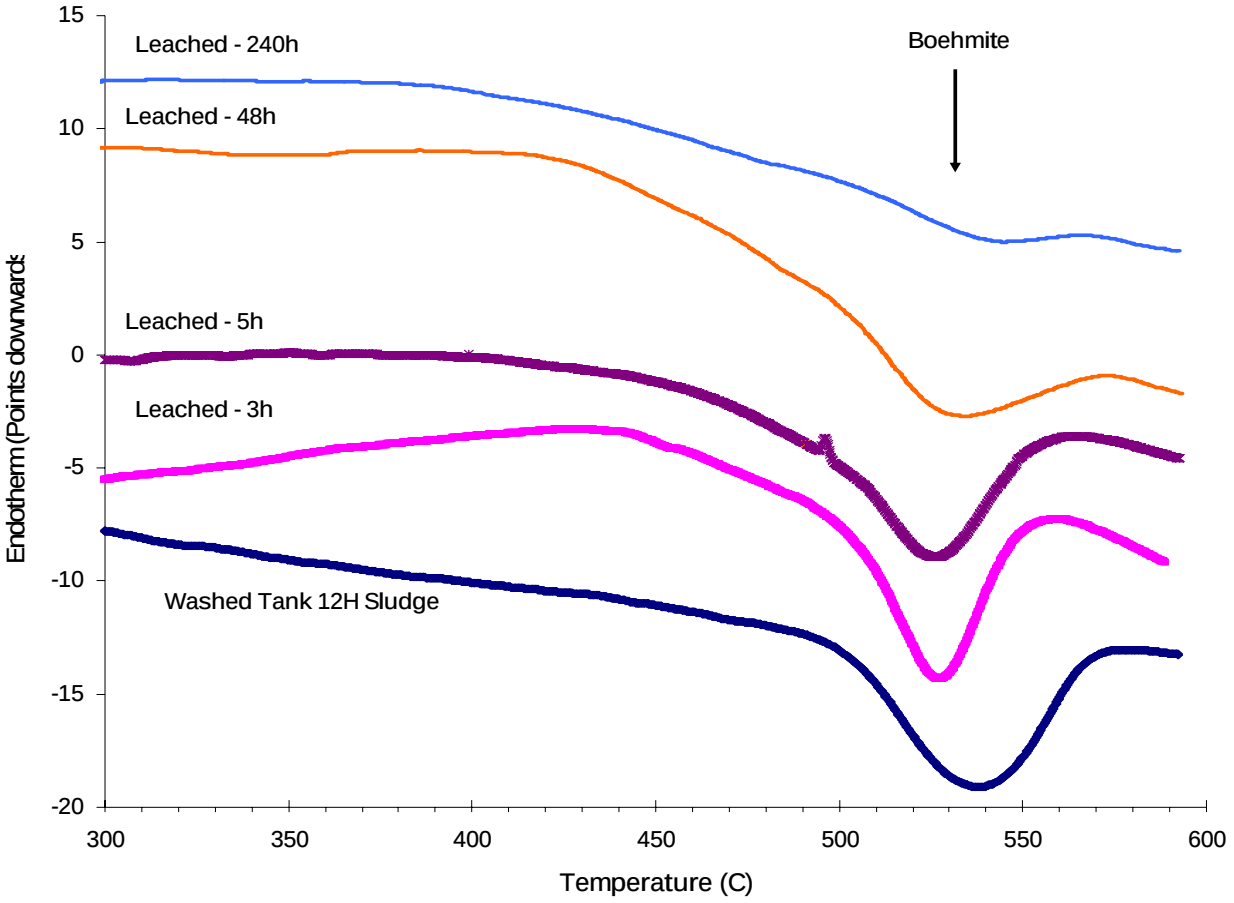
Tank 11H Sludge Calorimetry

- Boehmite present in washed sludge sample
- Boehmite content reduced from 17 to 8.6 wt% after 48h of caustic leaching



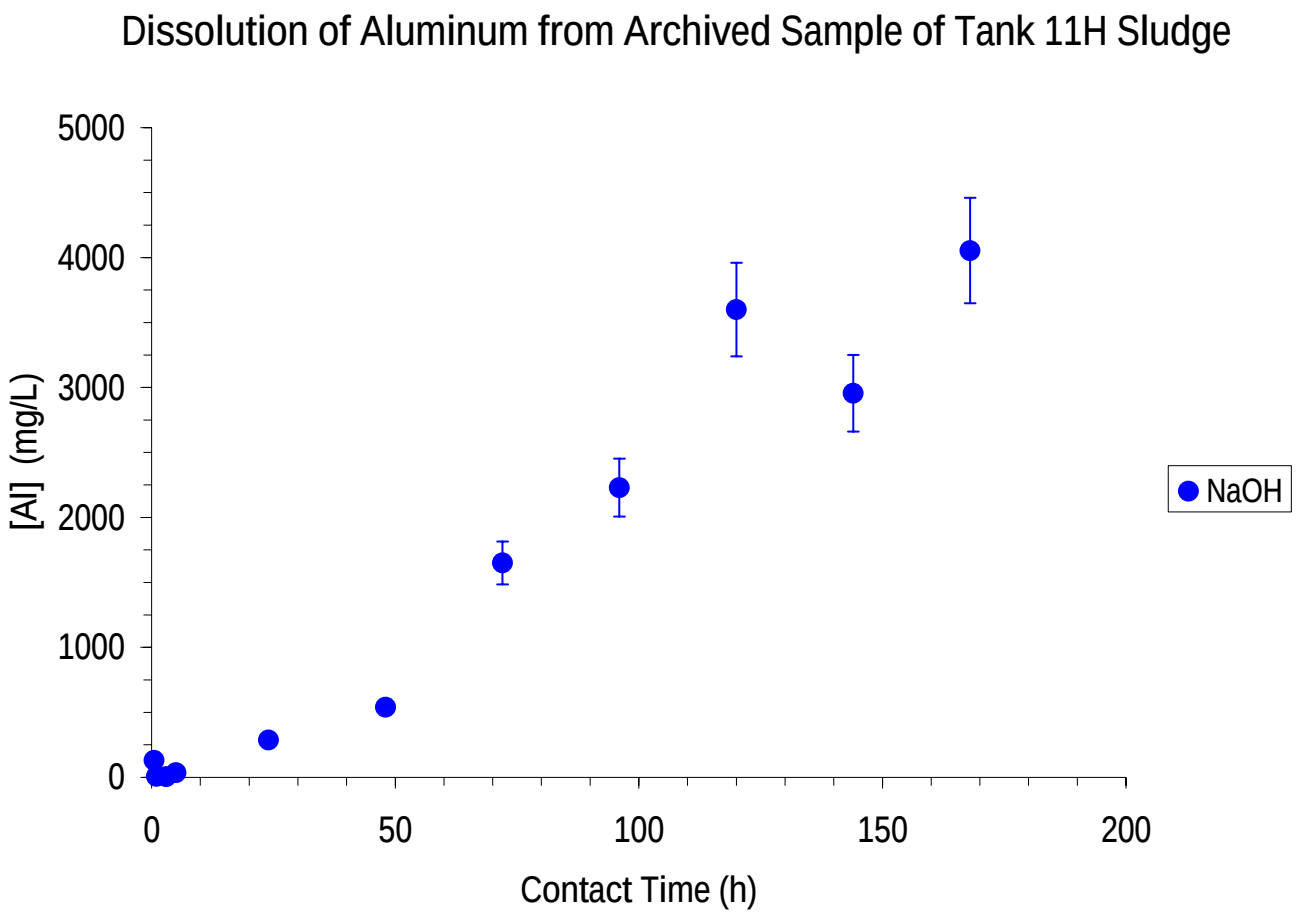
Tank 12H Sludge Calorimetry

- Boehmite present in washed sludge sample
- Boehmite content reduced from 20.8 to 9.2 wt% after 48h of caustic leaching
- Continued leaching reduces boehmite content even further



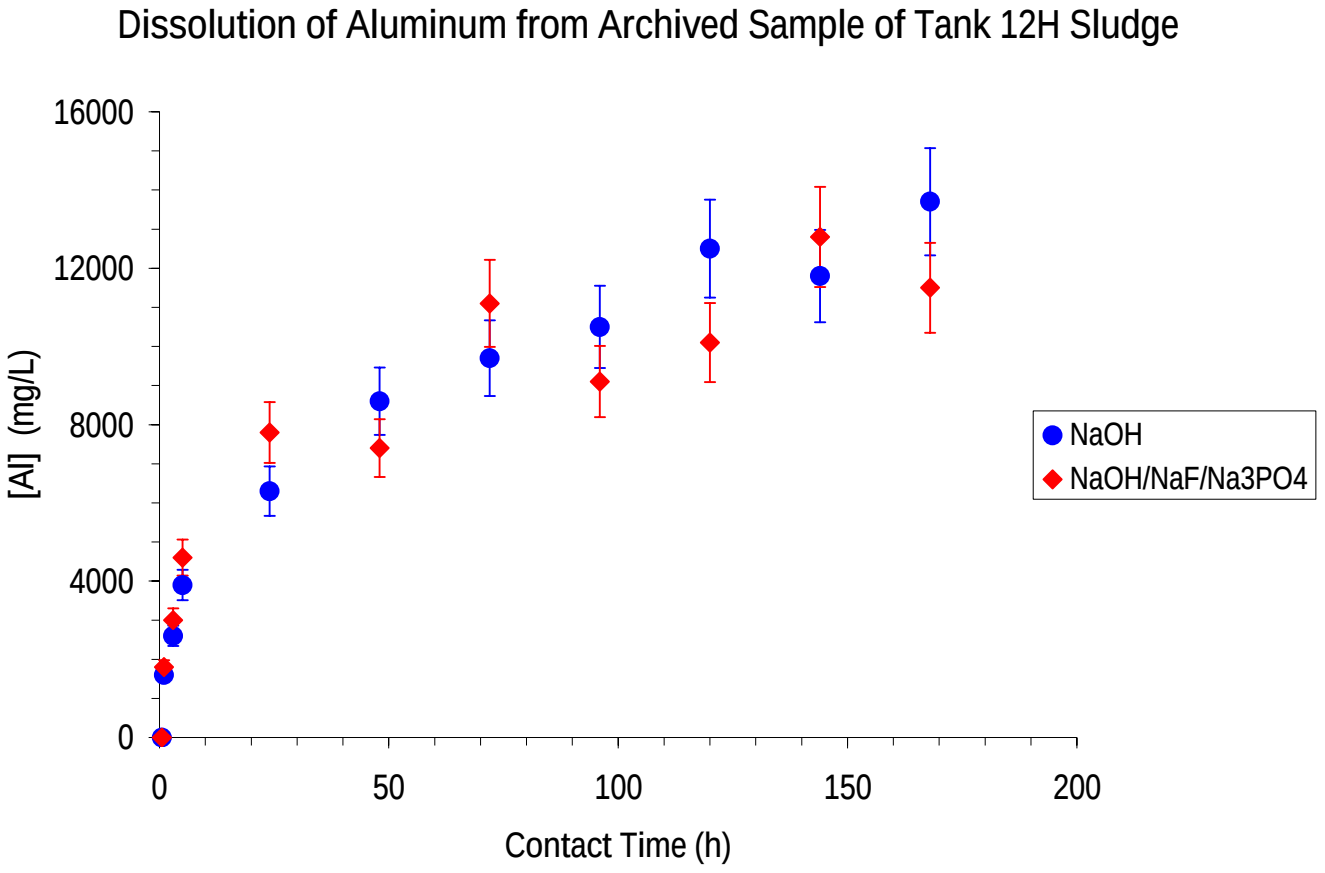
Aluminum Dissolution Rate in Tank 11H Sludge

Aluminum dissolution is mass-transfer limited



Aluminum Dissolution Rate in Tank 12H Sludge

- Aluminum dissolution is mass-transfer limited
- Addition of NaF and Na_3PO_4 did not enhance Al dissolution



Aluminum Speciation Before & After Leaching

- Sludges contain significant quantities of aluminum associated with other sludge components (e.g., solid solution with Fe/Mn oxides)

- Both boehmite and solid solution aluminum dissolves upon contact with caustic solution at elevated temperature

	Boehmite (wt%)		Solid Solution Al (wt%)	
Tank	Before	After	Before	After
8F	0	0	4.4	2.3
11H	17	8.6	7.2	1.1
12H	20.8	9.2	15	2.0