Chemical Analysis of Molten Salts Before and During Use in a Test Loop by Flame Atomic Absorption Spectroscopy

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NEXT Collaboration
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• 10 full-time faculty/staff + 20 undergraduate students
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• $4.5M funding
  • Local and federal
  • Private and public
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• Goal: Build non-nuclear test and research MS system
  • Use to evaluate and demonstrate emerging technologies
Gen IV Technology Roadmap

- MSR R&D should address
  - Corrosion
  - Molten salt chemistry control
  - REDOX control
  - Liquid-liquid extraction
  - Salt purification

US DOE Nuclear Energy Research Advisory Committee, Generation IV International Forum,
A technology roadmap for generation IV nuclear energy systems; December 2002; pp 34-35.
GIF Technology Roadmap

• MSR R&D should address
  • Corrosion
  • Molten salt chemistry control
  • REDOX control
  • Liquid-liquid extraction
  • Salt purification

• Needed: Direct chemical analysis of flowing molten salt
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  • Direct chemical analysis of flowing molten salt
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• Salt: Dynalene MS-2
  • LiNO$_3$ – NaNO$_3$ – KNO$_3$
  • 130°C – 500°C
  • Good corrosion performance for SS316
Current System: Molten Salt Test Loop

- **Purpose:** Gain experience with molten salt in a pumped flowing loop
- **Salt:** Dynalene MS-2
  - $\text{LiNO}_3$ – $\text{NaNO}_3$ – $\text{KNO}_3$
  - $130^\circ\text{C} – 500^\circ\text{C}$
  - Good corrosion performance for SS316
- **System Capacity:** 19 L
- **Mass of salt loaded:** 35 kg
- **Operating salt temp:** $<200^\circ\text{C}$
Current System: Molten Salt Test Loop

• Purpose: Gain experience with molten salt in a pumped flowing loop

• Salt: Dynalene MS-2
  • LiNO$_3$ – NaNO$_3$ – KNO$_3$
  • 130°C – 500°C
  • Good corrosion performance for SS316

• System Capacity: 25 L

• Mass of salt loaded: 35 kg

• Operating salt temp: <200°C
Sampling the Flowing Salt

• Sampling methods
  • SS316 dip cup on rod
Sampling the Flowing Salt

- Sampling methods
  - SS316 dip cup on rod
  - SS316 threaded rod
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  • Dispensing valve and tube mounted on test loop
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• Disadvantages
  • Exposure to atmosphere
  • Contamination from container
  • No instantaneous results
Flame Atomic Absorption Spectrometer

- Equipped to measure Fe, Cr, Mn, Ni
- Sub-ppm detection limits
- Must have lamp for each element
Calibration Curves for AAS

• Graph that shows calibration curves for four metals in solutions
Calibration Curves in Nitrate Salt Mixture

- Graph with four metals calibration
Determine metals in Dynalene MS-2

Metal concentrations in Dynalene MS-2 after introduction to MSTL

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration before pumping (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron</td>
<td>71.9</td>
</tr>
<tr>
<td>Nickel</td>
<td>2.1</td>
</tr>
</tbody>
</table>
Determine metals in Dynalene MS-2

Metal concentrations in flowing Dynalene MS-2 in MSTL

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration before pumping (ppm)</th>
<th>Concentration while pumping (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron</td>
<td>71.9</td>
<td>97.6</td>
</tr>
<tr>
<td>Nickel</td>
<td>2.1</td>
<td>10</td>
</tr>
</tbody>
</table>
Future Chemical Analysis Work

• Sampling from loop without exposure to atmosphere
• Direct measurement on flowing loop
  • Cyclic voltammetry
• Explore analytical methods applicable to more elements
  • Laser Induced Breakdown Spectroscopy
  • Mass Spectrometry
Physical Chemistry Group

• Dr. Aaron Robison, Dakota Martinez, Ron Laehn, Ashley Archambeau
• Characterization and purification of salts
• Differential Scanning Calorimetry (DSC)
DSC of Dynalene MS-2
(LiNO$_3$ – NaNO$_3$ – KNO$_3$)

Melting Point: 129°C (115-145°C)
Use of DSC to Study Salt Mixture Properties
Conclusions

• Achieved crude sampling and analysis of pumped flowing salt
• Need vast improvements in sampling
• Need better analytical methods for more elements
• Continue search for direct analysis of flowing molten salt
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