UPDATE ON LARGE-SCALE CHEMICAL PROCESSING FOR NEUTRON CAPTURE, AND ACCELERATOR DRIVEN PRODUCTION OF $^{99}\text{Mo}$

PETER TKAC

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ARGONNE’S SUPPORT TO NORTHSTAR

Irradiations, radiation dose, beam transport, shielding and target design, MCNPX

Post-irradiation handling and hot-cell processing

Chemical processes R&D
MOEX RECYCLE PROCESS - SOLVENT EXTRACTION

- Spent Mo generator solution (~0.3M Mo in ~5M HCl)
  - HCl
  - Filtration
  - KCl solid waste
  - ~0.3M Mo in ~5M HCl

- Mo metal powder
  - Reduction

- Solid AHM
  - NH₄Cl in EtOH
  - Evaporation
  - Wash with EtOH
  - Solid AHM + NH₄Cl

- NH₄Cl in EtOH
  - Evaporation
  - Recycled EtOH
  - NH₄Cl waste

- Solid AHM + NH₄Cl
  - Evaporation
  - Recycled TBP

- ~0.3M Mo in TBP
  - NH₄OH
  - K in aq. phase

- AHM + NH₄Cl in NH₄OH
  - Recycled TBP
UPDATE ON CENTRIFUGAL CONTACTOR WORK

Tests using 2-cm contactor

- Performed multiple 9-stage runs with 3D-printed plastic contactor
- Incorporated extended residence time stages for strip
- Added cooling plates to reduce heat transfer between motor and contactor body
- Re-designed coupling between motor and rotor for better stability
- Re-designed inter-stage lines
- Discovered that acrylic material is not suitable for long-term use
UPDATE ON CENTRIFUGAL CONTACTOR WORK

Mo feed in 5M HCl

Mo scrub 4M HCl

Mo strip NH₄OH

Extraction

Scrub

Strip

Raffinate

30%TBP/TCE

TBP out

Mo product in NH₄OH
### PROCESSING MURR-IRRADIATED SAMPLE

<table>
<thead>
<tr>
<th>EXTRACTION</th>
<th>FEED</th>
<th>SCRUB</th>
<th>STRIP</th>
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<tbody>
<tr>
<td>R</td>
<td>A1</td>
<td>A5</td>
<td>P</td>
</tr>
<tr>
<td>A2</td>
<td>A3</td>
<td>A7</td>
<td>A8</td>
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<tr>
<td>stage 1</td>
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<td>stage 8</td>
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<tr>
<td>stage 4</td>
<td>stage 6</td>
<td>stage 9</td>
<td>TBP out</td>
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<tr>
<td>&lt;&lt;&lt; Raf</td>
<td>TBP &gt;&gt;&gt;</td>
<td>O2 O3</td>
<td>O4 O5 O6 O7 O8 O9 TBP out</td>
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</tbody>
</table>

Mo feed: 12mL/min  
Mo scrub: 10mL/min  
TBP: 20mL/min  
Strip: 12mL/min  
O/A ex: 0.91  
O/A scrub: 2.0  
O/A strip: 1.67
**PROCESSING MURR-IRRADIATED SAMPLE**

<table>
<thead>
<tr>
<th>element</th>
<th>Mo in KOH mg-Me/kg-Mo</th>
<th>Mo feed-HCl mg-Me/kg-Mo</th>
<th>Product (NH₄OH) mg-Me/kg-Mo</th>
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SCALE UP TO 3CM CONTACTER
RECOVERY OF MO BY SOLVENT EXTRACTION

Total Mo in KOH solution processed: ~9L

Total Mo feed processed: ~45L

Total AHM recovered: ~2.7kg

AHM Size distribution
~10% fines (<45µm)
~90% 45-425µm

Amorphous material occasionally

crystalline

amorphous

Ammonium heptamolybdate
FULL-SCALE DISSOLVER FOR HOT CELL OPERATIONS

- Dissolution of up to 600g of Mo
- Vigorous and exothermic reaction
- ~1L of water condensed in 15min ~2.6kW
- Optimized processing time from 8hrs to ~2.5-4hrs
FULL-SCALE DISSOLVER FOR HOT CELL OPERATIONS
DISSOLUTION ASSEMBLY

Steam line for cleaning
Peroxide in
Plug for disks port
Saturated KOH solution
Disks insert
EVAPORATION ASSEMBLY

- Dissolved Mo in peroxide
- Saturated KOH solution
- Steam line for cleaning
- Stirring – magnetic coupler
- Mo solution for filtration
FUTURE PLANS

Mo Recycle

- Leach testing of potential alloys and thermoplastics for centrifugal contactor (PEEK, PP, PVDF, Inconel)
- Long-term testing of materials in contactor with Mo-HCl-TBP-TCE
- Manufacture full-scale 3-cm contactor

Dissolution

- Testing the dissolution apparatus for hot cell operations
- Water run to check solution transfers
- Dissolution of several hundred gram batches of sintered Mo disks
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