Challenges in implementing separation processes – moving from lab to plant scale

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Context

• No advanced reprocessing of MA partitioning plants planned in Europe or North America
  – No government policies to deploy
  – No industry pull
  – But widespread interest in advanced fuel cycles for sustainability reasons

• We are in an R&D phase
  – Developing options
  – National Lab led
  – Technology push

• How do we transition from lab to plant?
Nuclear energy pathway – closed cycle

- Long timescale to transition from research to operating plant
- Need R&D now to deploy mid-century
- A fully closed cycle requires integration of different technologies
Contents

• Context
• R&D goals
  – Research led development stages
• Testing
  – Example from Thorp development
• Design & engineering
  – Moving to the design-led process
  – Space batteries example
R&D pathway

- Coordination of R&D
- Down-selection of processes
- R&D goal is demonstration
- ~TRL 6
- Develop credible options
- Address perceived problems
- Prove benefits
- Collaborate
- Integrate into fuel cycle
- Be ready to transition to industry
Use of TRLs

<table>
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<tr>
<th>Commercial feeds</th>
<th>TRL 7</th>
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<tr>
<td>Irradiated fuels or targets</td>
<td>TRL 6</td>
<td>TRL 8</td>
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<td>Full simulants</td>
<td>TRL 3-4</td>
<td>TRL 6</td>
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<td>Partial simulants or representative materials</td>
<td>TRL 1-2</td>
<td>TRL 5</td>
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<td>Inactive or trace active</td>
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**Function**
- **Fundamental studies**
- **Flowsheet tests**
- **Maloperation studies**
- **Full scale operations**

**Definition**
- **Research**
  - Proof of concept
  - Lab scale tests to prove concepts, fundamental data obtained
- **Development**
  - Proof of principle
  - Technology component or process step validated at bench scale under relevant conditions. Process models validated.
- **Demonstration**
  - Proof of performance
  - Full scale process demonstrated in a limited operational environment.
- **Industry**
  - Full scale operations
  - Multiple years of operational experience established at industrial scale. Processing and recycle of minor actinide fuels / targets.
Research focused on what’s important to decision makers

- Increase flexibility
- Reduce wastes generated
- Reduce proliferation risks
- Show benefits
- Reduce costs
- Safe processes
- Demonstrate technical maturity
- Integrated fuel cycle solutions

Decision to deploy!
Modelling & simulation

• Explain basic data & direct new experiments
• Useful in flowsheet design
• Essential in maloperations & sensitivity analyses
• Example malop – dynamic simulation:
  – recycle & accumulation of An/Ln in i-SANEX process
  – Addition of water to scrub acid feed
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.2 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.2 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.3 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.3 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.4 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.4 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.5 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.5 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.6 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.6 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.7 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.7 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.8 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.8 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.9 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 0.9 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.1 hours

M(II)(or) (mol/L)

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.1 hours

M(III)(aq) (mol/L)
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.2 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.2 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.3 hours

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Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.4 hours
**Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.5 hours**

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Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.8 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.8 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1.9 hours

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Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 2.2 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 2.2 hours
**Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 2.4 hours**

**Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 2.4 hours**
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 2.8 hours

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Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 3.5 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 3.5 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 4.5 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 4.5 hours
**Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 5 hours**

- Eu Org
- Sm Org
- Nd Org
- Pr Org
- Ce Org
- La Org
- Y Org
- Am Org

**Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 5 hours**

- Eu Aq
- Sm Aq
- Nd Aq
- Pr Aq
- Ce Aq
- La Aq
- Y Aq
- Am Aq
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 6 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 6 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 7 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 7 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 8 hours

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Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 10 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 10 hours
Organic An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1000 hours

Aqueous An/Ln Profiles Following Loss of Acidity in Scrub 1 Feed (Stage 12). Time = 1000 hours
Underpinning the flowsheet & safety case

Underpin Safety Case

Normal & Maloperation Studies

Process Model

Pilot Plant Data
Lab scale trials
Fundamental Chemistry Data
International Experience
Historic Experience
Modelling & simulation
Engineering & scale up
Dealing with the unexpected

• Flowsheet testing (alpha active and hot tests) is essential to:
  – Demonstrate flowsheet performance
  – Validate process models
  – Investigate impact of malop conditions
  – Identify potential issues not highlighted by fundamental studies

• Despite extensive testing and modelling of the flowsheet operational issues may still arise during commissioning

• An early issue for the development of Thorp flowsheet:
  – Tc behaviour in 1BX column (identified by alpha active / pilot plant trials)
Trouble with Technetium (Tc)

- Separation of U/Pu achieved using U(IV) stabilised with hydrazine.
- Alpha active testing and pilot plant trials identified excessive consumption of hydrazine occurring in the process.
- Tc is co-extracted with U and Pu in HA/HS contactor.
- Excessive consumption attributed to Tc catalysed oxidation of hydrazine by nitric acid.

\[
\begin{align*}
\text{U}^{4+} + 2\text{Pu}^{4+} & \rightarrow 2\text{Pu}^{3+} + \text{UO}_2^{2+} + 4\text{H}^+ \\
\text{HNO}_2 + \text{N}_2\text{H}_5^+ & \rightarrow \text{HN}_3 + \text{H}_3\text{O}^+ + \text{H}_2\text{O}
\end{align*}
\]
Consequences for U/Pu separation

- Excess hydrazine added to process to ensure stability of U(IV) and Pu(III)
- Operation of U/Pu separation column modified from aqueous continuous to solvent continuous (validated by additional trials using pulsed column test rig).
A Hazard not identified cannot be protected against

Piper Alpha

- Explosion and subsequent fire
- Fatalities – 167
Process Safety

• It is important to consider safety at each stage of development.
  – Changes and improvements to the process.
  – New designs at different scales.

• HAZOP study developed for use in industry.
HAZOP Approach

**CONCEPT DESIGN LEVEL**
- Feed materials, hazards, process / technology

**SYSTEM DESIGN LEVEL**
- Re-use of process stage design / process if possible

**DESIGN CONFIRMATION LEVEL**
- Re-use of existing system level P&ID level design if possible

**HAZOP 0**
- Main hazards
  - Hazard Management Strategy (HMS)

**HAZOP 1**
- Confirm design features deliver chosen HMS
  - Identify potential key safety measures

**HAZOP 2**
- Confirm design meets HMS approach
  - Finalise key safety measures
## HAZOP Study – Hazard and Operability Study

- Multi-disciplinary team based
- Systematic approach to identifying hazards
- Split system into manageable units (based upon mechanical or process operations)
- Apply a series of relevant key words to each unit in turn.
- Complete all key words for each unit before moving onto the next unit.
- Different key words depending upon stage in design process.

### Property Words

**Basic Property Words**
- Flow
- Temperature
- Pressure
- Level

**Additional Property Words**
- Concentration
- Radiation
- Oxygen
- Viscosity
- Density
- Acidity
- Settle
- Dissolve

### Guide Words

<table>
<thead>
<tr>
<th>Node</th>
<th>Guide words</th>
<th>Possible Cause</th>
<th>Consequence</th>
<th>Indications/ Safeguards</th>
<th>Safety Assessment Required</th>
<th>Additional Notes</th>
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### Guide words

**Basic Guide Words**
- None
- More of
- Less of
- Part of
- As well as
- Early
- Late

**Additional Guide Words**
- Reverse

### Indications/Safeguards

- Safety Assessment Required
- Additional Notes
SACSESS & GENIORS

- HAZOP style safety reviews performed for Euro GANEX under SACSESS & GENIORS Programmes
- Key words specific to EU flowsheet projects developed under SACSESS
- Think Tank adopted HAZOP style process – output will be used in GENIORS safety review deliverable
**241\text{Am Space Battery Project**

- Project Initiates from a requirement

\[
\begin{align*}
^{241}_{94} Pu & \xrightarrow{\beta \quad 14.4\, y} \quad ^{241}_{95} Am \\
\end{align*}
\]

- Chem. Sep Flowsheet produced which can achieve the required separation.

- This then requires expansion to include all the process ‘blocks’ required. i.e. the job doesn’t start with Pu nitrate and end with Am nitrate

- Assign and scale equipment to each operation
Layout the Process

• Layout process flow
• Add mechanical movement equipment
• Build containment around this
• Understand faults / hazards & iterate
Layout the Plant
Summary – steps in moving from lab to plant scales

• Stick with reference processes => flowsheet optimisation
• Greater use of modelling & simulation => process models
• Underpin process safety => maloperations
• Address interfaces between head end, SX & conversion stages
• Process monitoring & control needed
• Understand S&T gaps => basic research needed
• What are the plans for demonstration tests to reach TRL 6?
• Increase communications with designers & engineers => layout the plant
• Integration with fuels & transmutation => feeds & products