Continuous ResonantAcoustic® Synthesis and Crystallization

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Building Value through Discovery and Innovation®

Problem Statement

Product Formulation:

- Many Constituent Components
- Specified Chemical Composition
- Optimized Material Properties
- Unique Synthesis, Processing Challenges

Continuous Chemical Synthesis Goals:

- **Flexibility:** Adapt to synthesis of broad range of chemicals
- **Tunability:** Able to control the physical properties of the product
- **Consistent Control:** Eliminate variability, decrease waste
- **Scalability:** Bench-scale discovery to world-scale production

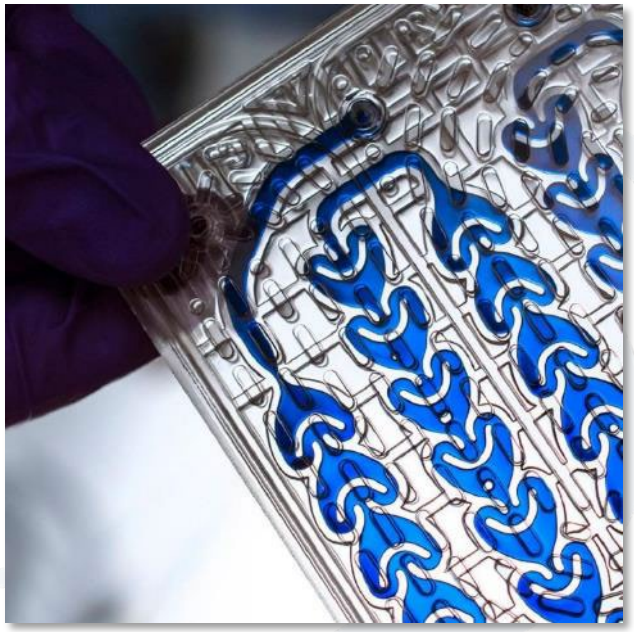
Legacy Batch vs. Advanced Continuous Flow Reactors

Chemglass 10L Reactor

Conventional Batch Reactors:

+ Inexpensive and Flexible
+ Multiphase Processes
- Variability on product quality
- Mixing and Heat-Exchange Limitations

Corning AdvancedFlow™ Reactor



Microfluidic Reactors:

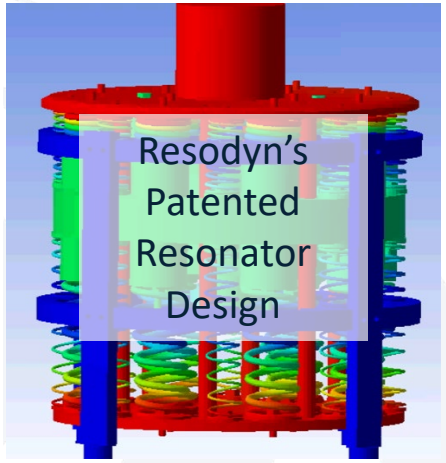
+ Improved Mixing and Temp Control
+ Control and Tunability of Product
+ Decreased Waste
- Complexity and Cost
- Difficulty Processing Solids

ResonantAcoustic® Mixing Phenomenon

RAM Features:

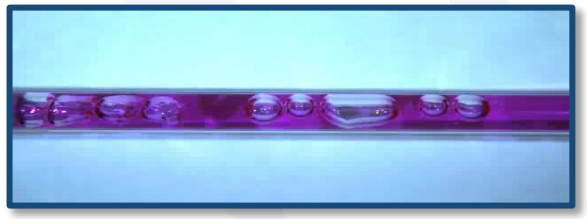
- Oscillates Contents at ~60 Hz, 100g
- Intense material interaction driven by uniform acoustic energy
- Broad mixing application base:
  - Liquids, Pastes, Solids, Multi-phase
  - Energetics, Chemical, Pharmaceutical, Energy Storage
- Seamlessly scalable from bench to industrial-scale processes

Bench-Scale Energetic Mixer
Hazardous Materials Rated

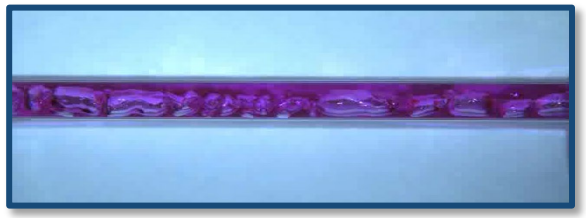


Multiphase Visualization in RAM Continuous Flow Cell

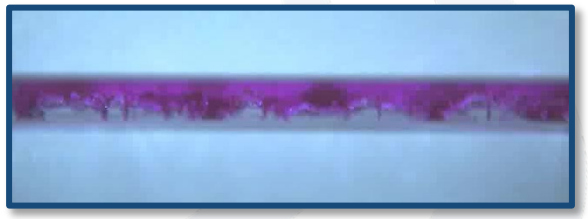
Compressive Regime vs. Splitting and Combining Regime



0 *g* Acceleration, 150 mL/min
4 mm Inner Diameter
Re = 780 → laminar flow



40 *g* Acceleration,
150 mL/min

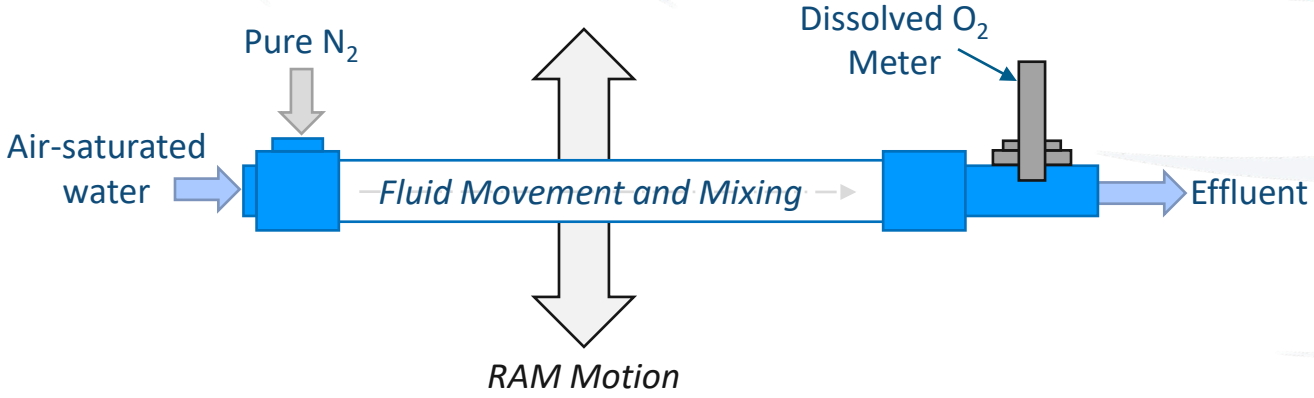


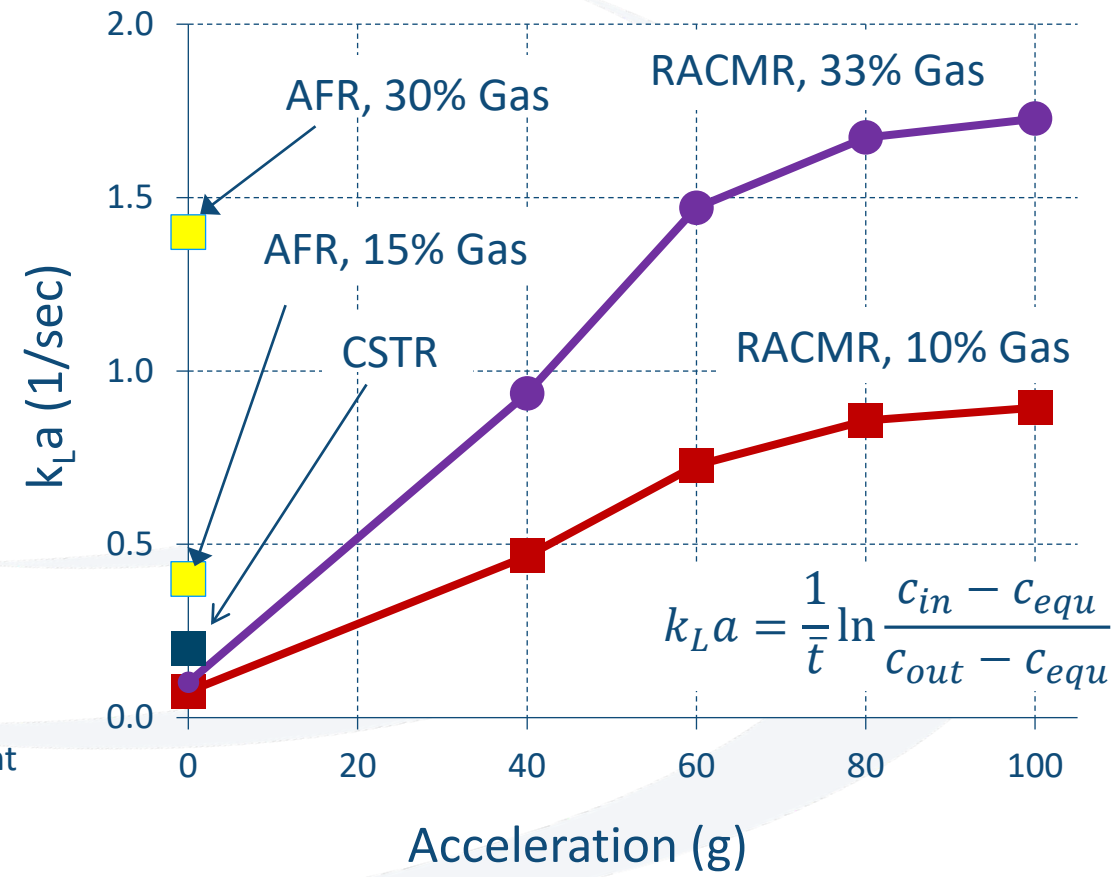
100 *g* Acceleration,
150 mL/min

Gas Headspace is Required for RAM Enhanced Mixing

Mass Transfer Coefficient Measurement

- **Dissolved Oxygen Experiments:**
  - Air-saturated water fed with pure N_2
  - Dissolved-oxygen meter measures transfer of O_2 to gas phase
  - Corning AFR is 7-fold improvement in Mass Transfer compared to legacy CSTR
  - Continuous ResonantAcoustic® Mixing is a further 35% improvement in Mass Transfer with an 88% lower residence time





Model Reaction Study: Mixing Time Measurement

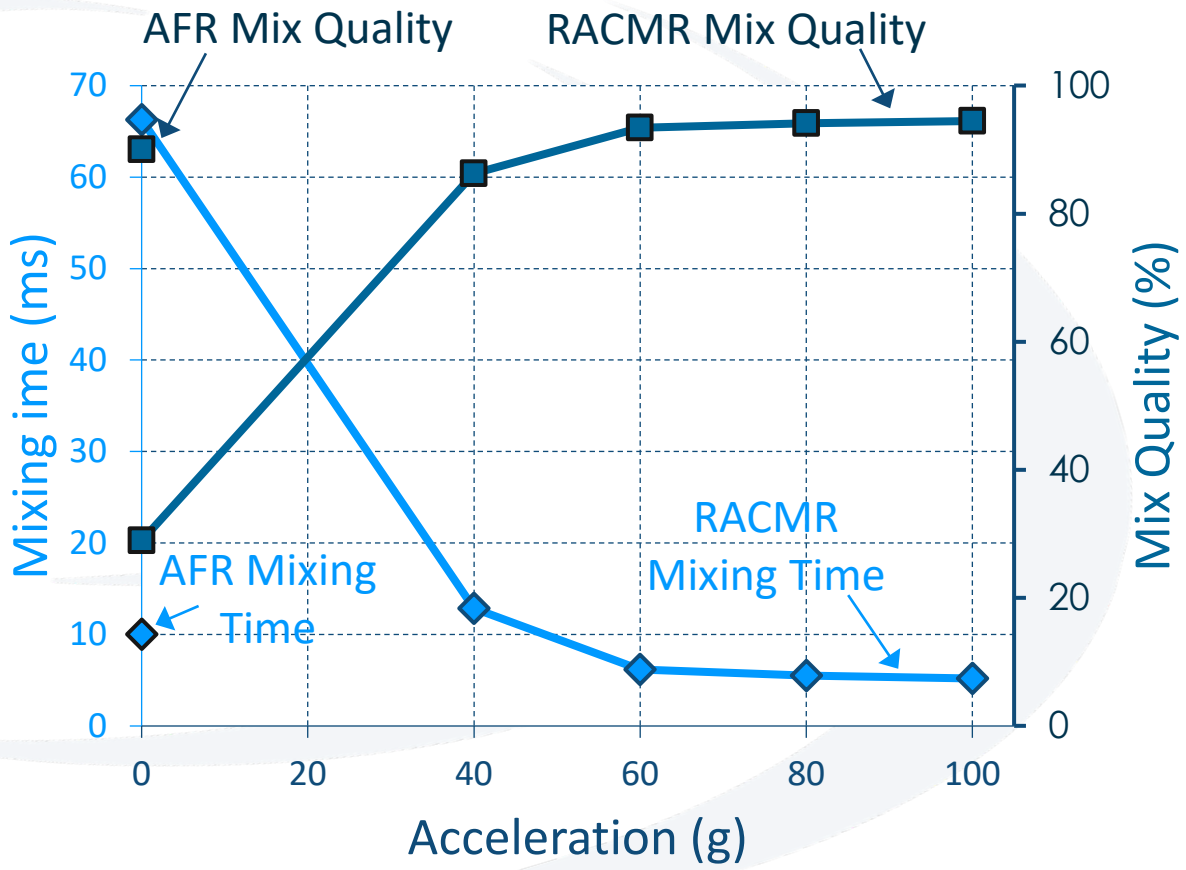
- Parallel Competitive Reactions to quantify mixing time

$$H_2BO_3^- + H^+ \overset{fast}{\longleftrightarrow} H_3BO_3$$

$$IO_3^- + 5I^- + 6H^+ \overset{slower}{\longleftrightarrow} 3I_2 + 3H_2O$$

$$I_2 + I^- \leftrightarrow I_3^-$$

- Fast reaction consumes acid when perfectly mixed
- Any degree of poor mixing, side reaction will form I_2 formation
- UV/VIS measurement of [I_3^-] in product, used to calculate mix quality and mixing time



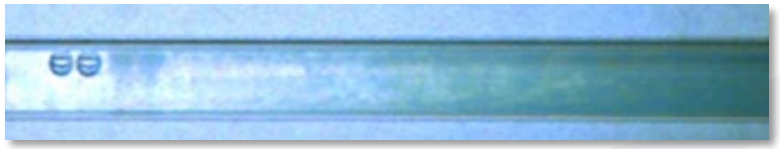
Precipitation Reaction Demonstration

- Advanced microreactors easily clog in the presence of solids
- Continuous ResonantAcoustic® process can handle solids

- Example: Formation of Basic Copper Carbonate solids

$$2\,Cu^{2+}(aq) + 2CO_3^{2-}(aq) + H_2O \rightarrow Cu_2(OH)_2CO_3(s) + CO_2(g)$$

0 g Acceleration

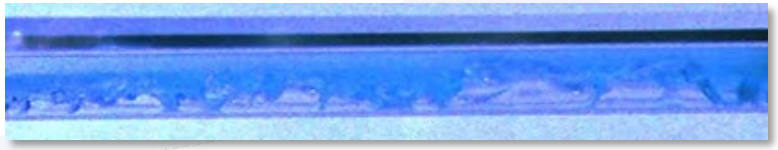


40 g Acceleration

60 g Acceleration

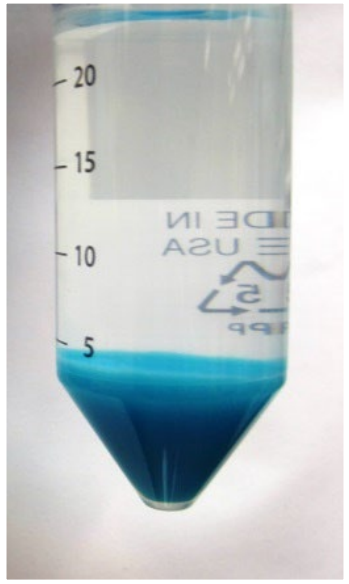
80 g Acceleration

100 g Acceleration

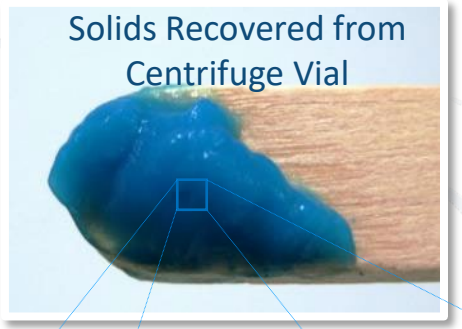


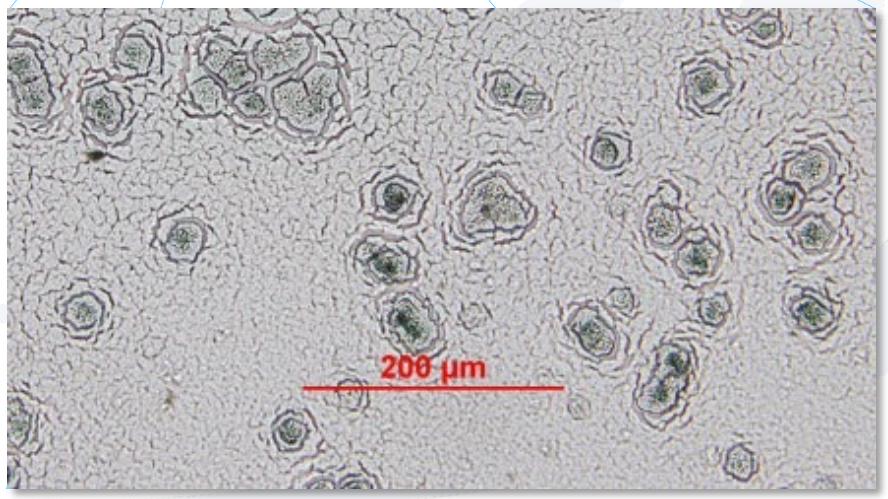
Precipitation Reaction Demonstration

Sample collected from microreactor
80-100 *g* acceleration



After Centrifuging

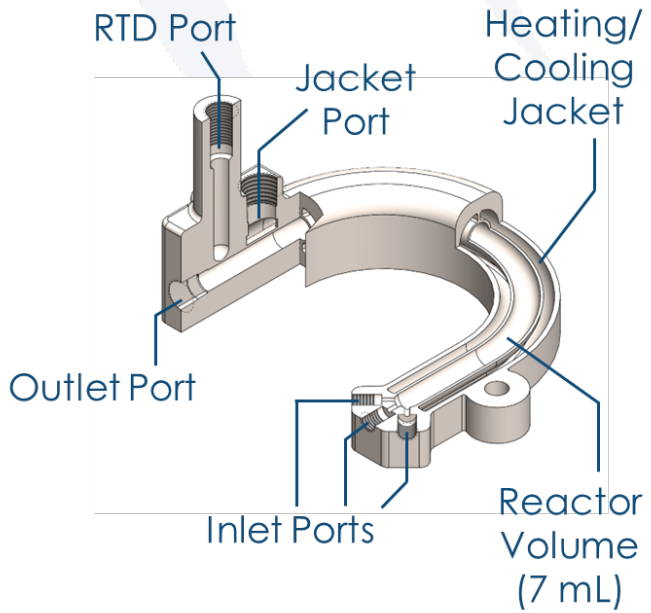




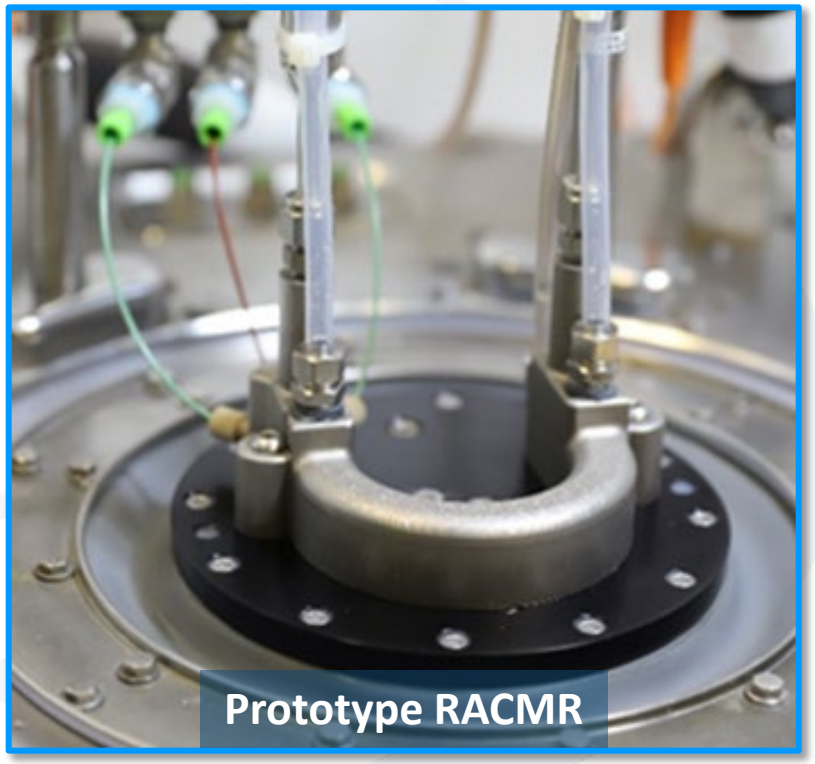
Microscope Images Reveal Basic Copper Carbonate Particles

ResonantAcoustic® Continuous Microreactor (RACMR)

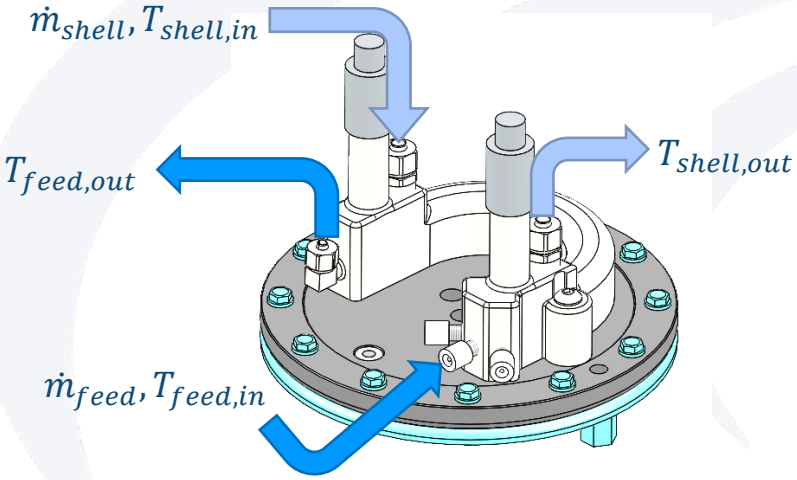
- RACMR Prototype:
  - Incorporation of up to three reactants
  - Immediate and complete mixing of reactants
  - RAM results in plug flow fluid profile
  - Jacketed for cooling or heating of reaction mixture



RACMR Design

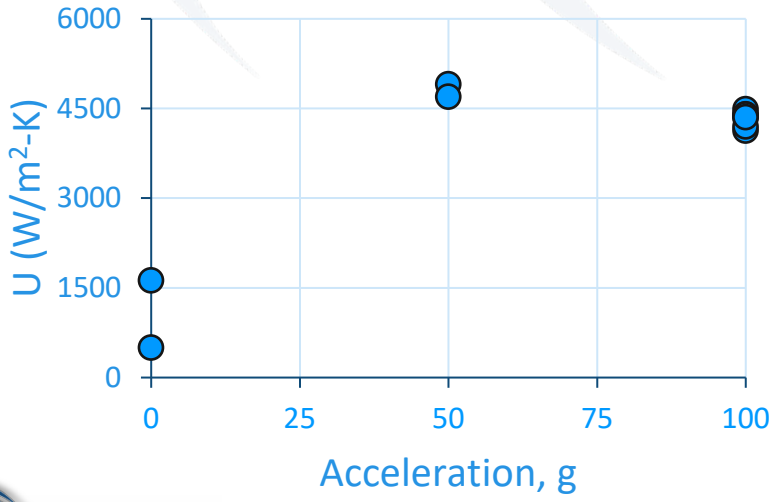


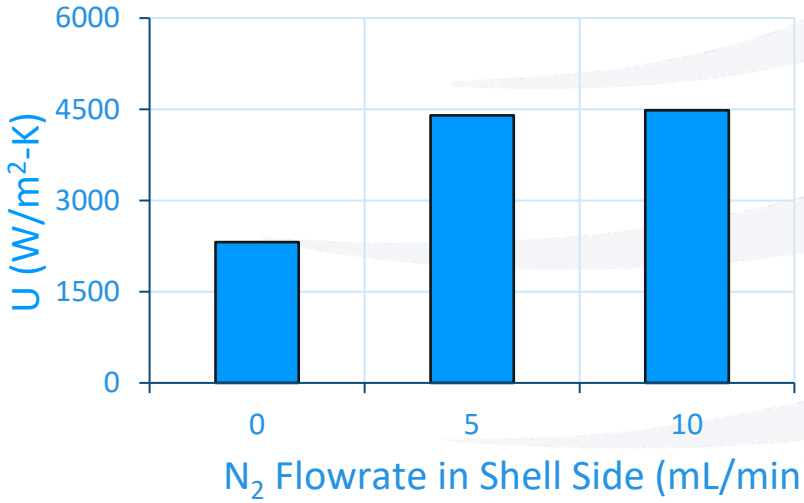
Heat Transfer Coefficient Measurement

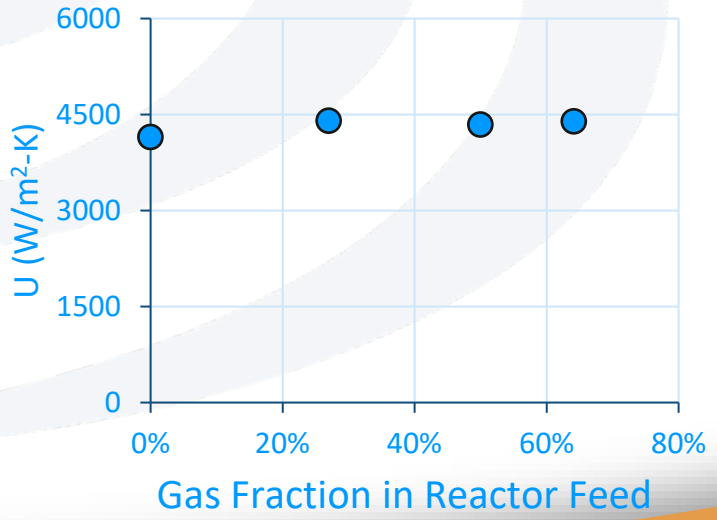


$$\dot{q} = \dot{m}_{feed} C_{p,feed} \left(T_{feed,in} - T_{feed,out}\right)$$
$$= \dot{m}_{shell} C_{p,shell} \left(T_{shell,in} - T_{shell,out}\right)$$

$$\dot{q} = UA\Delta T_{LM} = UA \frac{\left(T_{shell,out} - T_{feed,in}\right) - \left(T_{shell,in} - T_{feed,out}\right)}{\ln\left(\left(T_{shell,out} - T_{feed,in}\right) \Big/ \left(T_{shell,in} - T_{feed,out}\right)\right)}$$



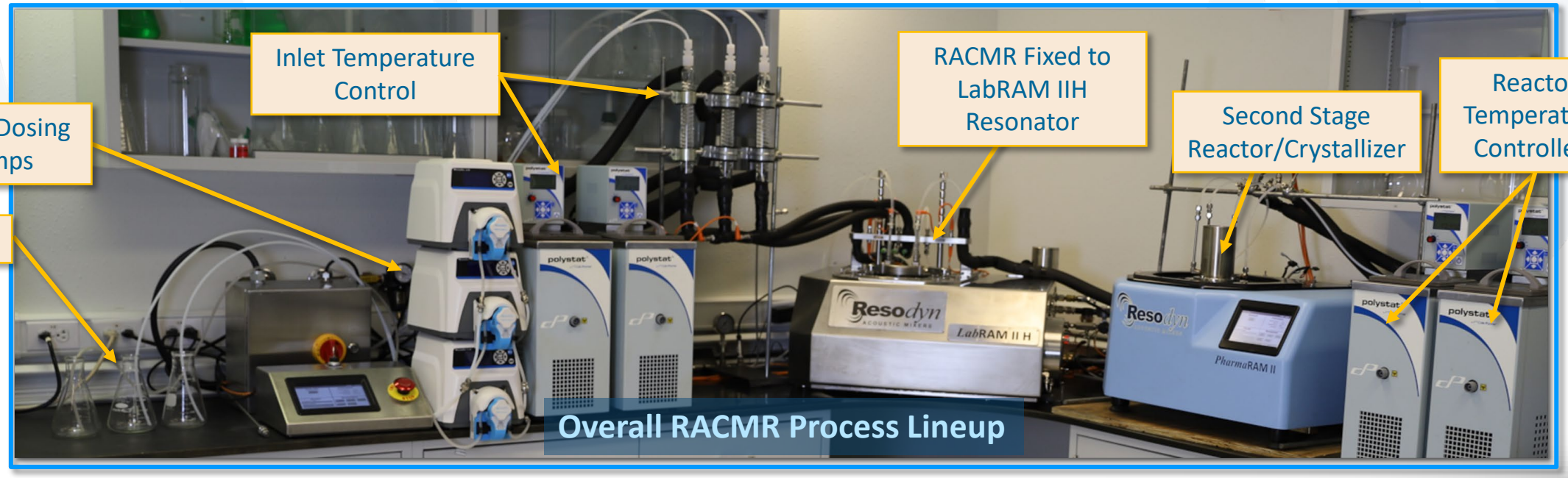




Demonstration of RACMR Flow Process

Highlights:

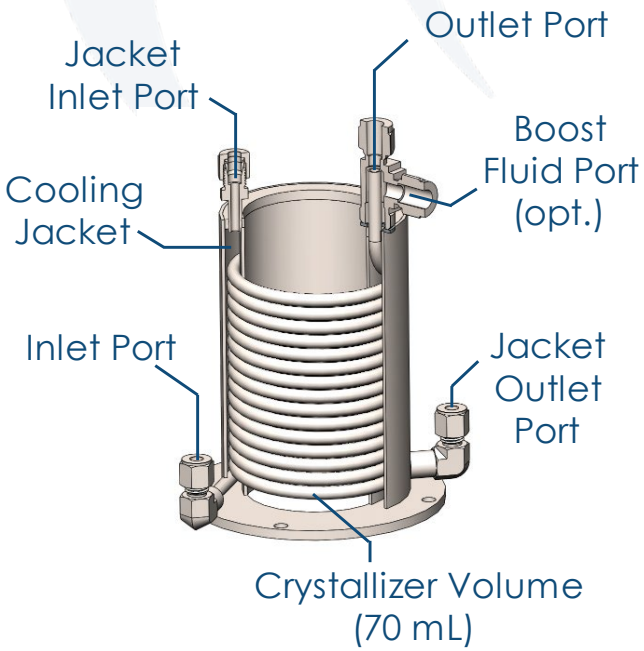
- Demonstrated synthesis of energetic precursors
- Continuous synthesis with high purity and yield
- Delivery of >1kg of >99% pure product



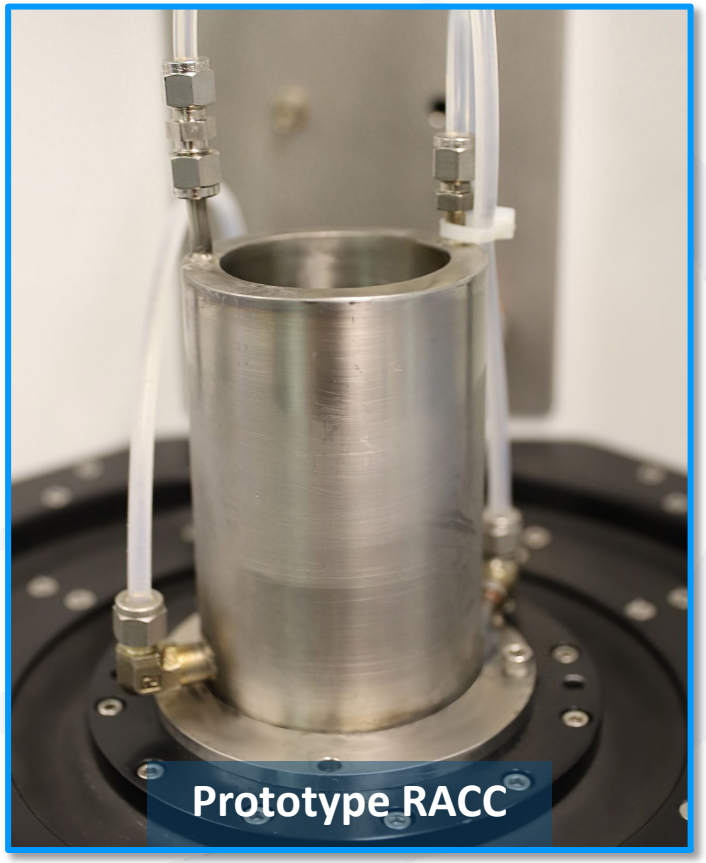
Overall RACMR Process Lineup

ResonantAcoustic® Continuous Crystallizer (RACC)

- RACC Prototype:
  - Optimized for Cooling Crystallization
  - Saturated solution fed through coil
  - Jacket supplied with coolant
  - Slurry product filtered

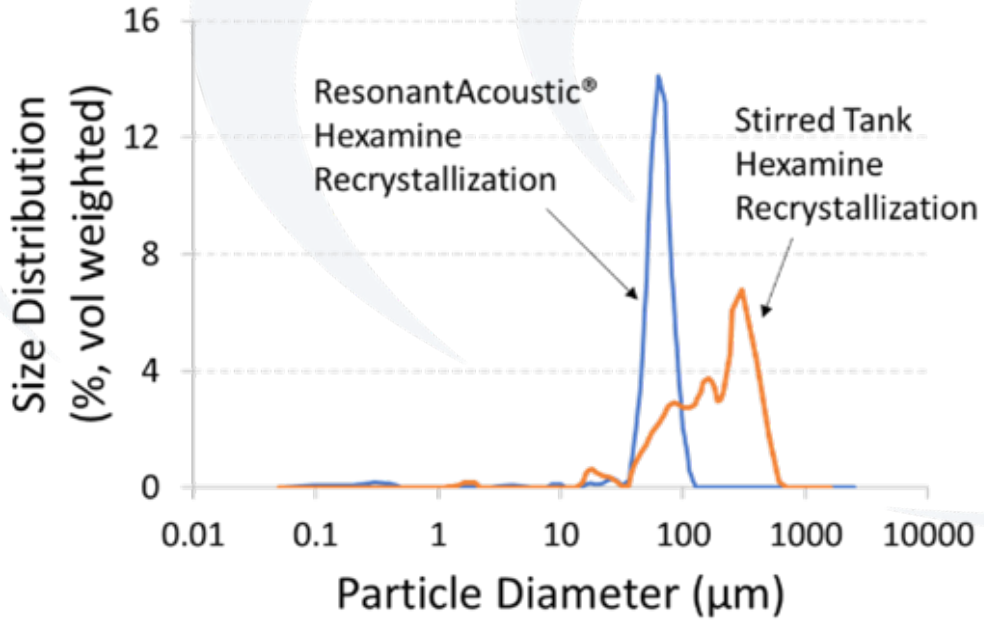


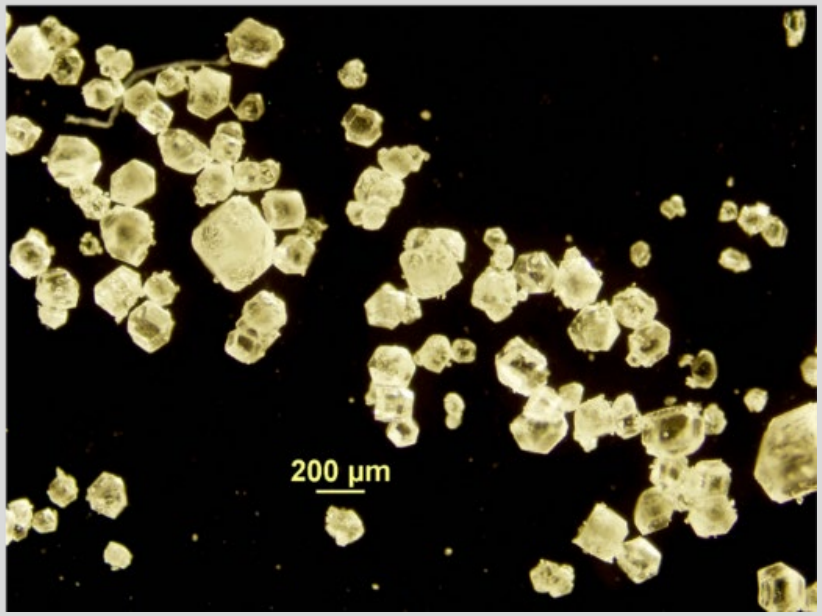
RACC Design



Recrystallization of Hexamine

- **RACC provides 90% narrower Crystal Size Distribution**

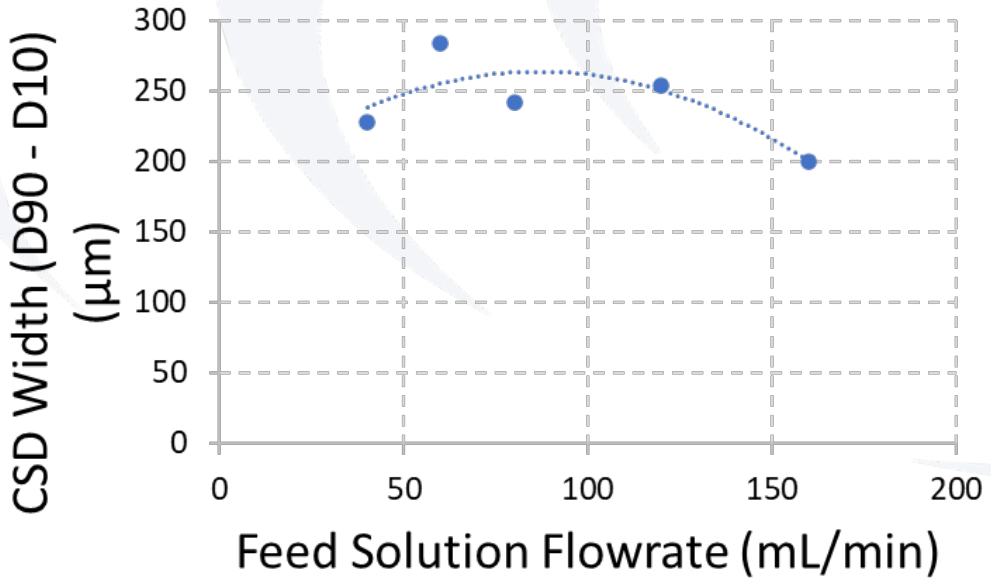




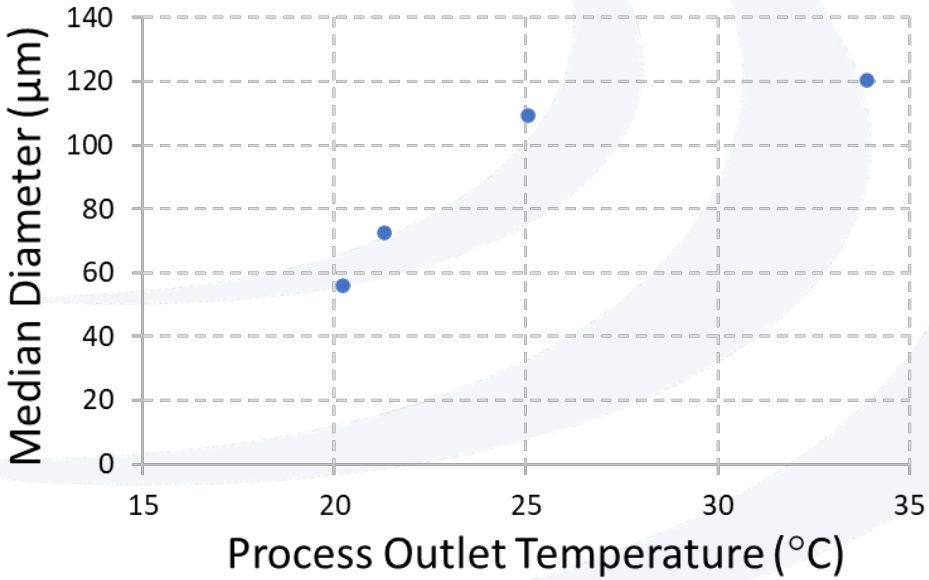
Hexamine recrystallized via RACC

Recrystallization of Hexamine

- Narrow CSD across broad inlet RACC flow range

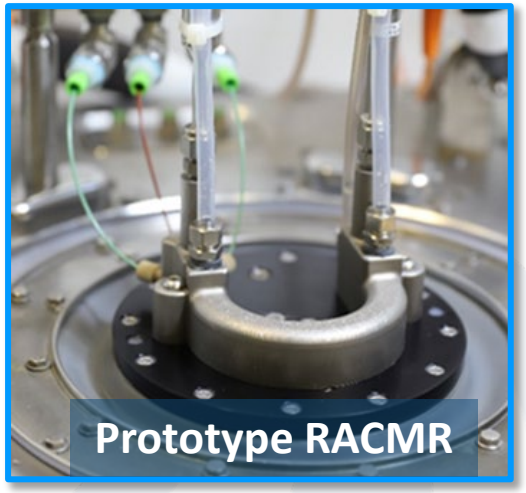


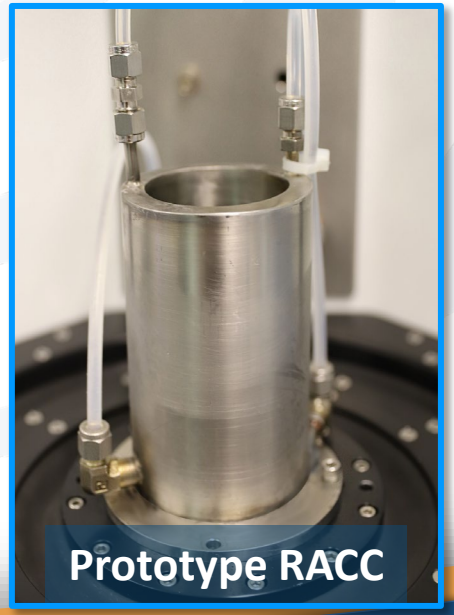
- Mean Particle Size is tunable based on Jacket Temperature



Summary

- Continuous Chemical Processing Benefits:
  - Rapid and complete incorporation of feeds (mixing time: 6 ms)
  - Highly effective heat transfer (U = 4,400 W/m^2-K)
  - Mass/Heat Transfer independent of flow velocity
  - Ability to handle slurries, viscous flow, multi-phase processes etc.
  - Scalable process from bench-scale to industrial-scale reactors
- ResonantAcoustic® Continuous Microreactor:
  - Demonstrated synthesis of energetic precursors
  - Continuous demonstration with consistent high yield
- ResonantAcoustic® Continuous Crystallizer:
  - 90% narrower crystal size distribution than legacy crystallizer
  - Ability to tune crystal morphology to desired range based on shell temperature


Prototype RACMR


Prototype RACC

Acknowledgements

Thank you for your time and attention.