

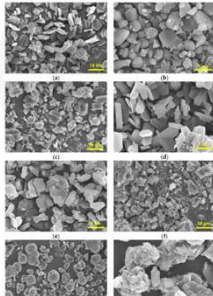
Continuous Crystallization using ResonantAcoustic® Technology

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Product Quality Starts with Crystallization



Crystallization Influences:

- Purity/Polymorphism
- Particle Size Distribution (PSD)
- Crystal Morphology

↓

Which Drives Material Properties:

- Flowability & Handling
- Downstream Processing
- Final Product Performance

Effect of Crystallization Environment on PYX Crystal Morphology



Source: Wang et al. (Molecules, 2023, 28, 4735). DOI: 10.3390/molecules28124735



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Challenges in Crystallization Process Development

Critical Questions:

- Crystal Size Distribution
 - Can crystal size be controlled consistently?
- Crystal Morphology
 - Can crystal shape and habit be controlled?
- Scale-Up
 - Will crystal properties remain consistent across all scales?

Nucleation

Many Small Crystals

Crystal Growth

Few Large Crystals

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Shifting the Balance:

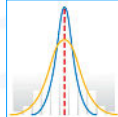
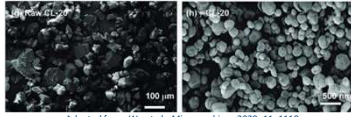
- Temperature
- Supersaturation
- Mixing
- Pressure
- Residence Time
- Seeding




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Advantages of Continuous Crystallization

Batch Crystallization	Continuous Crystallization
Broad PSD/CSD	Consistent PSD/CSD
Limited Process Visibility	Enhanced Process Visibility
Scale-Up Challenges	Easier Scale-Up/Scale-Out
Transient Crystallization Environment	Controlled Crystallization Environment
Time-Varying Conditions	Steady-State Operation



Source: HORIBA Scientific

Adapted from: Wu et al., *Micromachines* 2020, 11, 1110.

Continuous crystallization seeks improved control, consistency, and product quality.



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A ResonantAcoustic® Solution for Crystallization

The ResonantAcoustic®

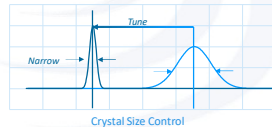
Environment:

- ✓ Uniform Energy Distribution
- ✓ Intense Mixing
- ✓ Low-Shear Processing
- ✓ Efficient Heat Transfer

Crystallization Benefits:

- Controlled Supersaturation
- Consistent Nucleation Environment
- Crystal Size Control
- Narrower PSDs/CSDs

Can ResonantAcoustic® processing improve nucleation and crystal growth control?



Crystal Size Control



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Experiment: Batch Stirred-Tank Crystallization

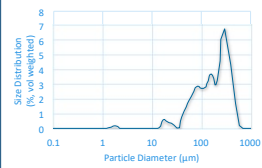


Baseline Crystallization System:

- Conventional stirred-tank crystallizer
- Cooling crystallization
- Hexamine selected as feed material
- Batch operation



Hexamine CSD via Stirred Tank Crystallization



Baseline Crystal Population

- Broad, left-skewed crystal size distribution



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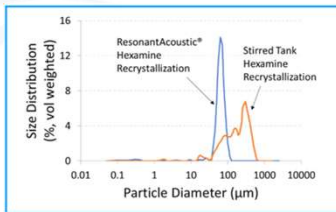
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Experiment: Batch RAM Crystallization



Jacketed LabRAM II Crystallizer Vessel
• 90 g Acceleration



Improved Crystal Size Uniformity

- 90% narrower Crystal Size Distribution vs. traditional crystallization methods

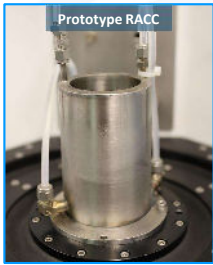


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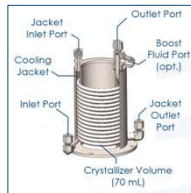
Development of the ResonantAcoustic® Continuous Crystallizer



Prototype RACC

Design Objectives:

- Continuous cooling crystallization
- Controlled residence time
- Continuous slurry product
- Future integration of PATs
- Simplified geometry

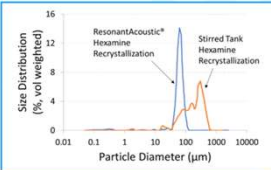
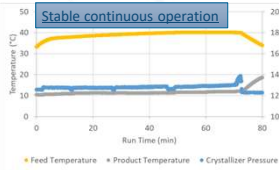
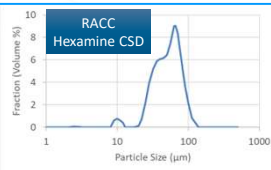
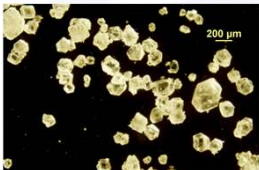


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Experiment: Continuous Cooling Crystallization



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Continuous Crystallization Design of Experiment (DOE)

DOE Factors

Flow Rate

Gas Fraction

Acceleration

Jacket Temperature

Measured Response Variables

Median Crystal Size

• D_{50}

Crystal Size Distribution Width

• $D_{90} - D_{10}$

Which operating parameters influence crystal size distribution?

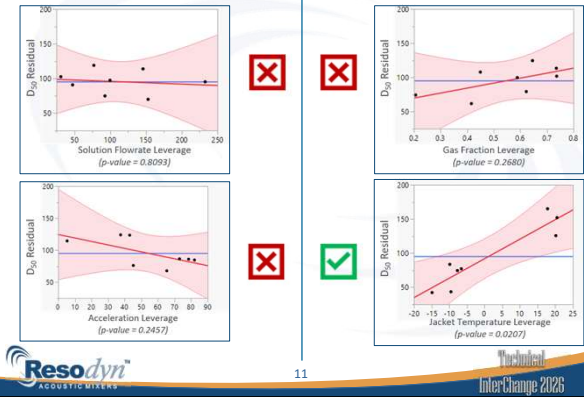


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Median Crystal Size (D_{50}) Response

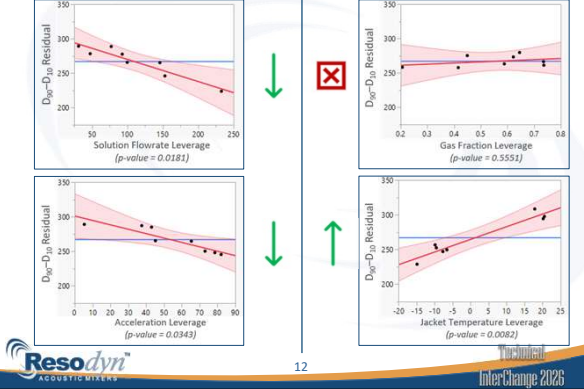


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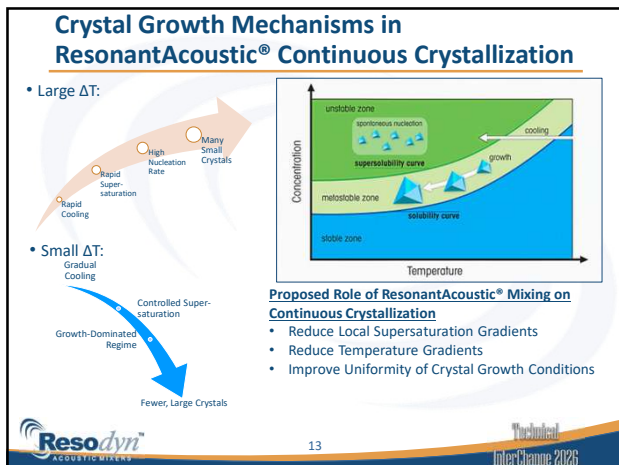
Crystal Size Distribution Width ($D_{90}-D_{10}$) Response



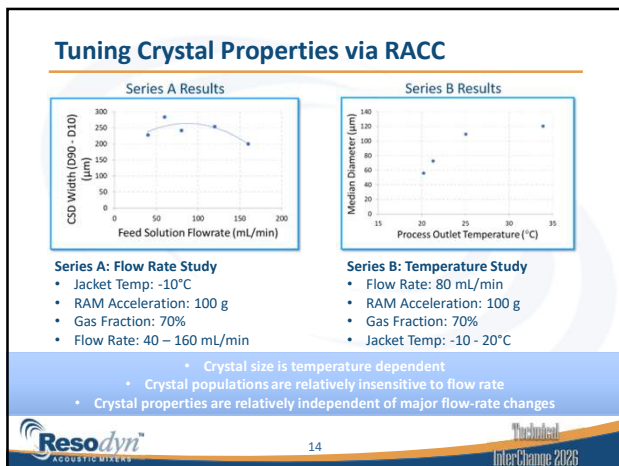
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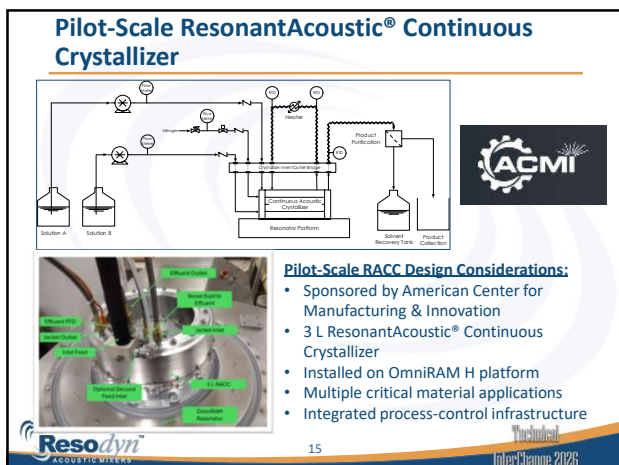
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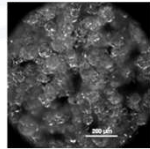


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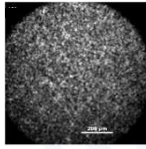
Crystallization of Critical Raw Materials

Key Points:

- Multiple chemistries demonstrated
- Diverse crystallization behaviors
- Continuous Operation



Online microscopy of synthesized Potassium Perchlorate slurry



Online microscopy of synthesized Strontium Oxalate slurry

Critical Chemical	Reagent A	Reagent B
Potassium Nitrate	Sodium Nitrate	Potassium Chloride
Potassium Chlorate	Sodium Chlorate	
Potassium Perchlorate	Sodium Perchlorate monohydrate	
Strontium Oxalate	Strontium Chloride hexahydrate	Ammonium Oxalate monohydrate

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Crystallization of Critical Raw Materials

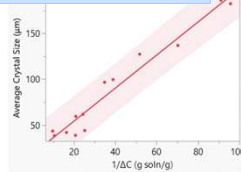
Key Observations:

- Crystal size decreased with increasing supersaturation
- Trend observed for all potassium salts
- Single correlation described all datasets

Conclusions

- Common design principles may apply across materials
- Pilot-scale results set the path toward relevant manufacturing throughput

Avg. Crystal Size vs. Inverse of Supersaturation for All K-Salts



	3L RACC Production Rate (Steady-State)	3L RACC Production Rate (Increased Utility)	20L RACC Production Rate (Theoretical RAM 5 Scale-up)
KNO ₃	4.54 g/min	23.78 g/min	1.23 kg/min (163 lb/hr)
KClO ₃	6.55	10.51	0.54 (72 lb/hr)
KClO ₄	2.51	5.81	0.30 (40 lb/hr)
SrC ₂ O ₄	1.63		

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Summary

1. Continuous crystallization was demonstrated using ResonantAcoustic® technology
 - Batch and continuous cooling crystallization achieved
 - Crystal properties characterized and correlated
2. Crystal properties can be controlled through process conditions
 - Supersaturation is primary driver of crystal growth
 - Acoustic energy influences crystal-size distribution
3. RACC platform demonstrated at both lab- and pilot-scale

ResonantAcoustic® technology provides a flexible, tunable platform for continuous crystallization

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Thank you for your time
and attention.





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