Applications of Continuous ResonantAcoustic® Synthesis and Crystallization

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New Product and Process Development Manager
Resodyn Corporation

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Objectives

- Vision of Platform Technology for Continuous Synthesis and Crystallization built on RAM Technology
- Seven Development Projects Transitioning Continuous Flow Synthesis and Crystallization from Bench-Scale Development to Commercial Technology
- Applications and Benefits of Continuous ResonantAcoustic® Flow Processes

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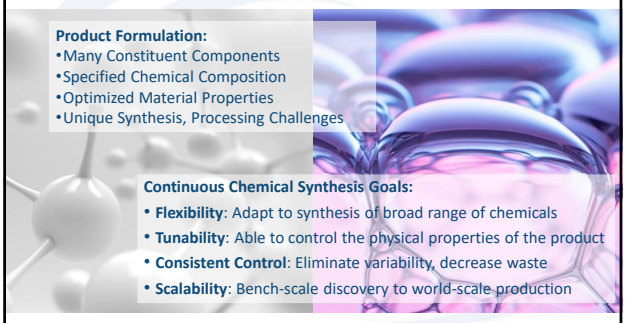
Problem Statement



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

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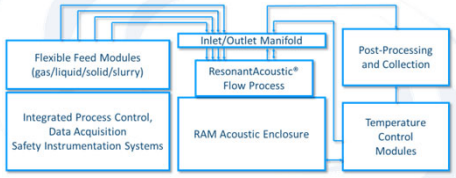
Platform Technology For Flexible Delivery of Critical Chemicals



- Turn-key System with Broad Capabilities, Flexible, Customizable
- True Continuous Operation for Improved Product Quality and Decreased Waste
- Integrated Process Analytical Technologies (PATs)

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Project 1: Continuous Microreactor for Energetic Material Synthesis

Sponsor: Naval Surface Warfare Center – Indian Head Division
DoD Ordinance Technology Consortium

Project Goals:

- Design, Build and Demonstrate a ResonantAcoustic® Continuous Microreactor (RACMR)
- Pilot continuous synthesis of a key energetic precursor molecule

Project Challenges:

- Complex reaction scheme with competitive side reactions
- Combination of a fast, heat-releasing, initial reaction followed by slow, heat-absorbing, second reaction
- Mixed slurry product stream

Competitive Reaction Scheme Example:

Desired Reactions

$$A + B \rightarrow I$$
$$I + C \xrightarrow{\Delta} P$$

Undesired Reactions

$$A + C \xrightarrow{\Delta} X$$
$$I + B \rightarrow Y$$

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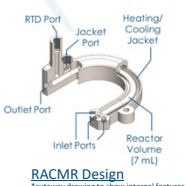
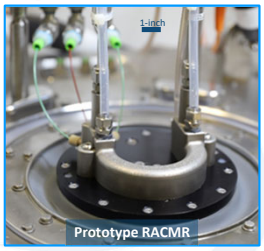
ResonantAcoustic® Continuous Microreactor

Novel RACMR Prototype Design:

- 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
*cutaway drawing to show internal features



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Continuous Synthesis Process Design

- **Key Design Features:**
  - First Acoustic Reactor: 7mL RACMR, 2°C, fast heat-generation initial reaction
  - Second Acoustic Reactor: 70 mL Coil Reactor, 40°C, Slow heat-absorbing reaction with slurry product
  - Precise process control (feed flowrates, feed and reactor temperatures)

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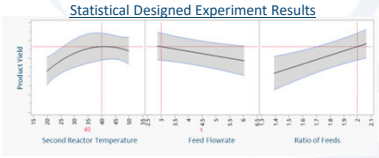
Continuous Process Development

- Statistical designed experimentation to optimized process (see example results below):
  - Maximize product purity by balancing feed chemistry
  - Maximize product yield based on residence time, reactor temperature and feed ratios

Statistical Designed Experiment Results



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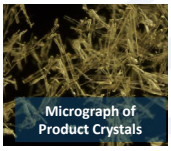
Project 1: Results and Impact

- Product purity of 99.5% (via HPLC)
- Maximum yield of 90% (compared to 68% best reported in literature)
- 11 runs with continuous run time of up to 700 mins without any upset
- >1 kg of energetic precursor product delivered to NSWC IHD
- Successful bench-scale demonstration of continuous acoustic reaction scheme
- Positive sponsor feedback for future scale-up potential



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Project 2: Continuous Microreactor for Energetic Material Synthesis

Sponsor: Lawrence Livermore National Laboratory

Lawrence Livermore National Laboratory

Project Goals:

- Design, Build, Demonstrate updated RACMR design
- Pilot continuous synthesis of a key energetic precursor molecule

Project Challenges:

- Target molecule synthesized in water at elevated temperature and pressure
- Slower heat-absorbing chemistry
- Competitive side reactions form intractable polymer biproduct
- Limited literature on synthesis process

Resodyn Acoustic Mixers

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The Chemical InterChange 2025

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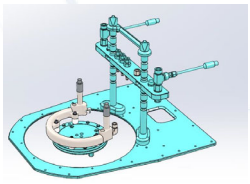
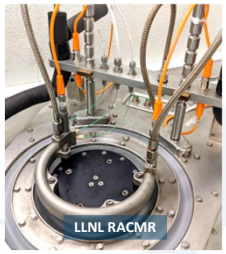
High Temperature RACMR Design

Updated RACMR Design:

- Increased volume, 18 mL, for slower reactions
- Ruggedized design for 200°C design temperature, 40 bar design pressure
- Improved temperature probe design for onboard inlet and outlet temperature measurement



RACMR Design



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The Chemical InterChange 2025

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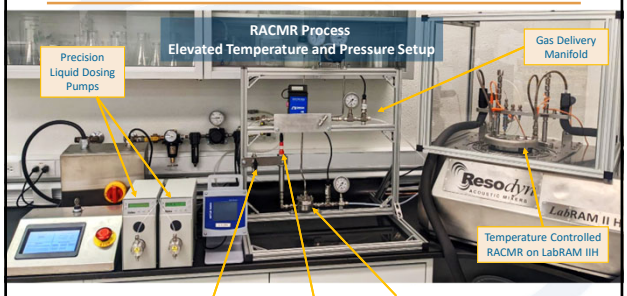
Continuous Microreactor Process



Resodyn Acoustic Mixers

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The Chemical InterChange 2025

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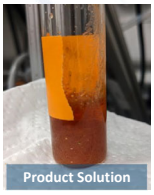
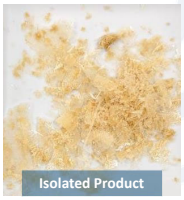
Project 2: Results and Impact

- Optimized product yield of 90%
- Demonstrated ability to control pH to optimize reaction
- Avoid producing intractable polymer biproduct
- 24-h continuous production of 140g of high purity energetic precursor compound delivered to LLNL
- Demonstrate continuous aqueous synthesis at 150-200°C and elevated pressure



Product Solution



Isolated Product

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Project 3: Continuous Synthesis of Defense Critical Materials

Sponsor: Defense Logistics Agency SBIR Phase I

Project Goals:

- Develop flexible continuous ResonantAcoustic® Synthesis Platform
- Design and Demonstrate system for Strontium Nitrate synthesis

Project Challenges:

- Single capital equipment to deliver multiple defense critical materials
- Slurry feed to reactor
- Manage carbon dioxide gas formed as byproduct of synthesis process

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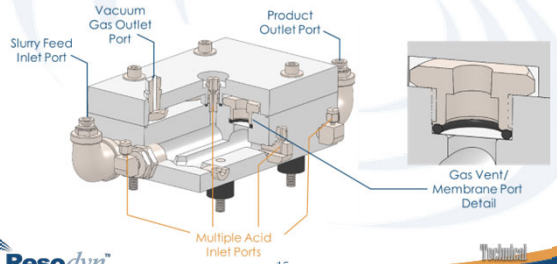
ResonantAcoustic® Membrane Reactor

Acoustic Membrane Reactor Design:

- Integrated gas vents with PTFE membrane to remove carbon dioxide gas formed
- Strontium carbonate fed as slurry up to 40% solid loading
- Multiple acid injection for controlled reaction (see next slide)



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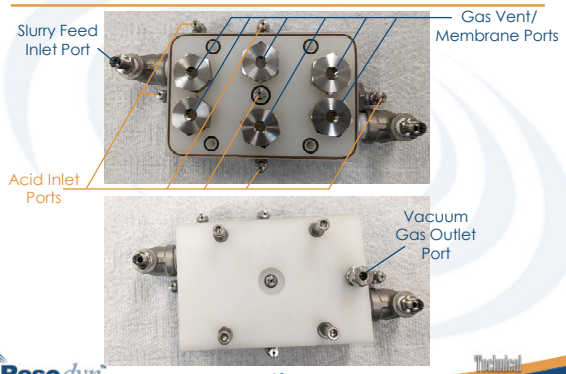
ResonantAcoustic® Membrane Reactor



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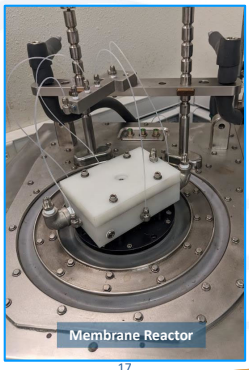
ResonantAcoustic® Membrane Reactor



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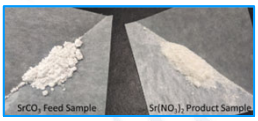
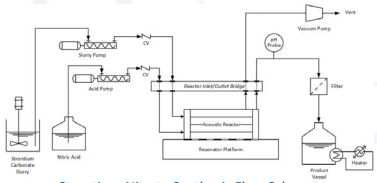
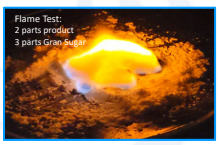
Continuous Acoustic Chemical Process

- Demonstrate bench-scale production of strontium nitrate
- Online pH measurement
- Product purity confirmed by metals analysis (>99%)







Strontium Nitrate Synthesis Flow Scheme

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Project 3: Results and Impact

- Achieved 100% conversion
- 99.6% Strontium Nitrate purity
- Demonstrated continuous production capacity of up to 7 g/min in bench-scale process
- Design pilot-scale system capable of delivery of multiple critical compounds from a single process:
  - Lead nitrate
  - Potassium nitrate
  - Strontium nitrate
  - Hexamine
  - TNT
- Potential domestic suppliers of feedstock materials identified

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Project 4: Continuous Crystallization of Energetic Materials

Sponsor: Army SBIR Phase I
AF SBIR Phase II

Project Goals:

- Design, Build, Demonstrate Completely Novel Continuous Acoustic Crystallizer
- Deliver Narrow Crystal Size Distribution
- Tune Mean Crystal Size to Enable Custom Product Formulations
- Demonstrate crystallization of HMX and CL-20
- Deliver representative energetic nitramine crystals

U.S. ARMY SBIR

AFVENTURES

SBIR·STTR America's Seed Fund

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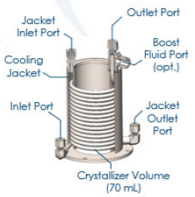
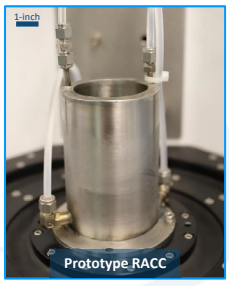
ResonantAcoustic® Continuous Crystallizer

Continuous Crystallizer Prototype Design:

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





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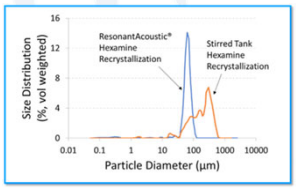
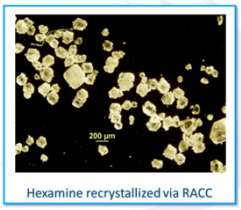
Recrystallization of Surrogate Energetics

- Demonstrated continuous recrystallization of hexamine as a surrogate energetic material
- Proven 90% narrower Crystal Size Distribution compared to traditional crystallization methods





Hexamine recrystallized via RACC

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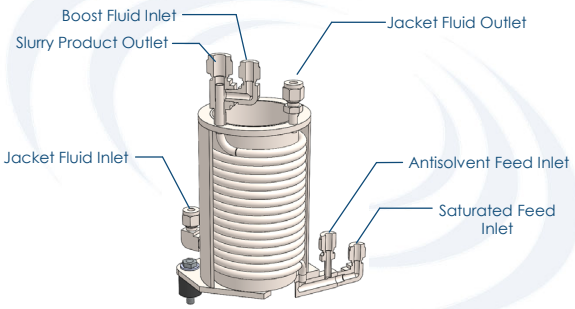
Pilot-Scale Continuous Crystallizer Design



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Energetic Recrystallization Project Update

- Project Update:
  - Surrogate recrystallization demonstrated (Feb 2024)
  - Pilot-scale RACC process delivered to Rocky Mountain Scientific Laboratory (July 2024)
  - Continuous HMX recrystallization demonstrated (Sep 2024)
- Next Steps:
  - Continuous CL-20 recrystallization demonstration
  - DOE: Process Characterization
  - Demonstrate Particle Size control

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Project 5: Continuous Synthesis and Crystallization of Critical Chemicals

Sponsor: American Center for Manufacturing & Innovation

Goals:

- Design a pilot-scale ResonantAcoustic® Continuous Crystallizer unit with associated process/equipment
- Demonstrate Continuous and Scalable Crystallization and Crystallization of Critical Chemicals
- Demonstrate that multiple materials can be synthesized from a single platform system
- Demonstrate tunability/control of product morphology

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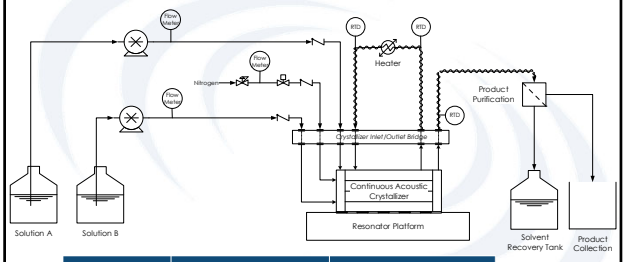
Pilot-Scale RACC OmniRAM H Platform



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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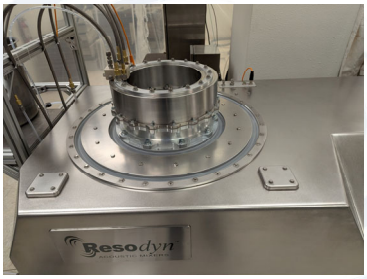
3L ResonantAcoustic® Continuous Crystallizer



3L RACC Installed on the OmniRAM H Resonator

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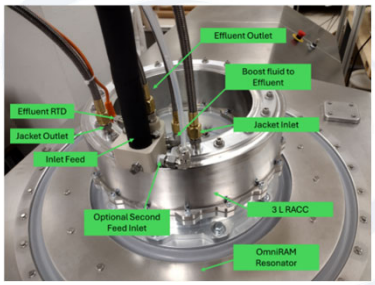
3L ResonantAcoustic® Continuous Crystallizer



3L RACC Installed on the OmniRAM H Resonator

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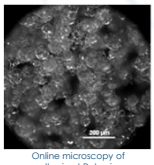
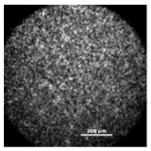
Project 5 Results and Impact

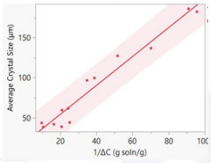
- Single Platform Process Used to Synthesize/Crystallize Multiple Critical Materials
- Integrated Online Spectroscopy, Particle Size Measurement and Microscopy
- Demonstrated Crystal Size Control and Tunability



Online microscopy of synthesized Potassium Perchlorate slurry



Online microscopy of synthesized Strontium Oxalate slurry



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Project 5 Results and Impact

- Single Platform Process Used to Synthesize/Crystallize Multiple Critical Materials
- Integrated Online Spectroscopy, Particle Size Measurement and Microscopy
- Demonstrated Crystal Size Control and Tunability

	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_2O_4	1.63		

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Project 6: First-Article Agile Continuous Chemical Reactor and Crystallizer Systems

Sponsor:

Naval Surface Warfare Center – Indian Head Division

Naval Energetic Systems and Technologies Program

Deliverables:

- First-Article Pilot-Scale Continuous Acoustic Chemical Reactor for Live Energetic Materials
- First-Article Pilot-Scale Continuous Acoustic Crystallizer for Live Energetic Materials

Period of Performance:

2023 – 2027

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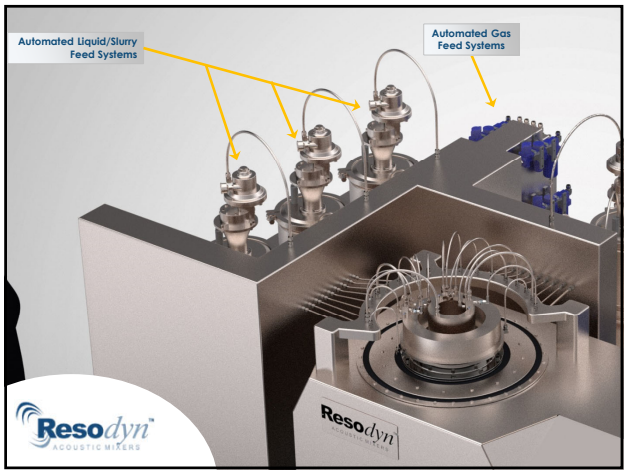

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Resodyn™ OmniRAM H
Acoustic Mixer
Continuous Chemical
Reactors/Crystallizers
OmniRAM H
Resodyn
ACOUSTIC MIXERS

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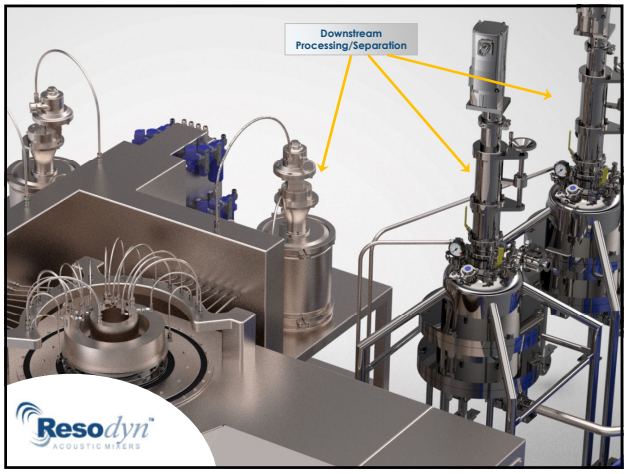
Downstream
Processing/Separation
Resodyn
ACOUSTIC MIXERS

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Additional System Features:
Built In Process Analytical Technology:
Continuous Chemical Reactor Includes Raman Spectroscopy for online Chemical ID, QA/QC, Process Insights and Chemical Kinetics
Continuous Crystallizer Includes Focused Beam Reflectance Measurement (FBRM) for Real-Time Crystal Growth Kinetics, and Crystal Size Distribution Data
Fully Automated Distributed Control System Allows Safe and Efficient Control of System from Remote Control Room
Integrated Process Cooling/Heating and Machine Cooling
Designed for System Pressures up to 22 bar and Temperatures up to 90°C
Specialized Materials of Construction and Enhanced Safety Measures for Energetic Material Handling
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Continuous ResonantAcoustic® Chemical Reactor and Crystallizer Technology

- Successfully demonstrated in a broad range of synthesis applications, with untapped future potential
- Easily process solid, liquid and gas streams (even rapid gas evolving reactions!)
- Eliminate concern of heat transfer or mass transfer limitations in continuous chemical processes
- Proven to deliver superior chemical selectivity and conversion
- Tune crystal size of product to meet strict standards
- Increased process control to decrease waste and eliminate variability
- Implement in-line chemical and physical analysis to unlock new scientific frontiers or achieve product quality assurance in real time
- Scalable process technology to deliver customer-tailored solutions from bench-scale chemical research and development to world-scale production

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Thank you for your time and attention.

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