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The webinar will begin promptly at
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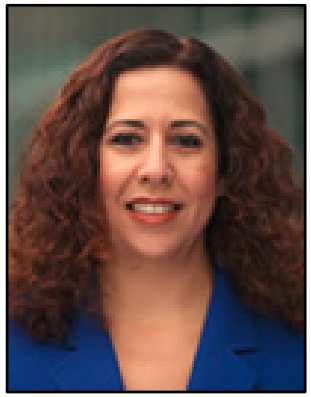
Environmentally Sustainable Manufacturing for Energetic Formulations

May 31, 2018

SERDP DOD • EPA • DOE | ESTCP

Welcome and Introductions

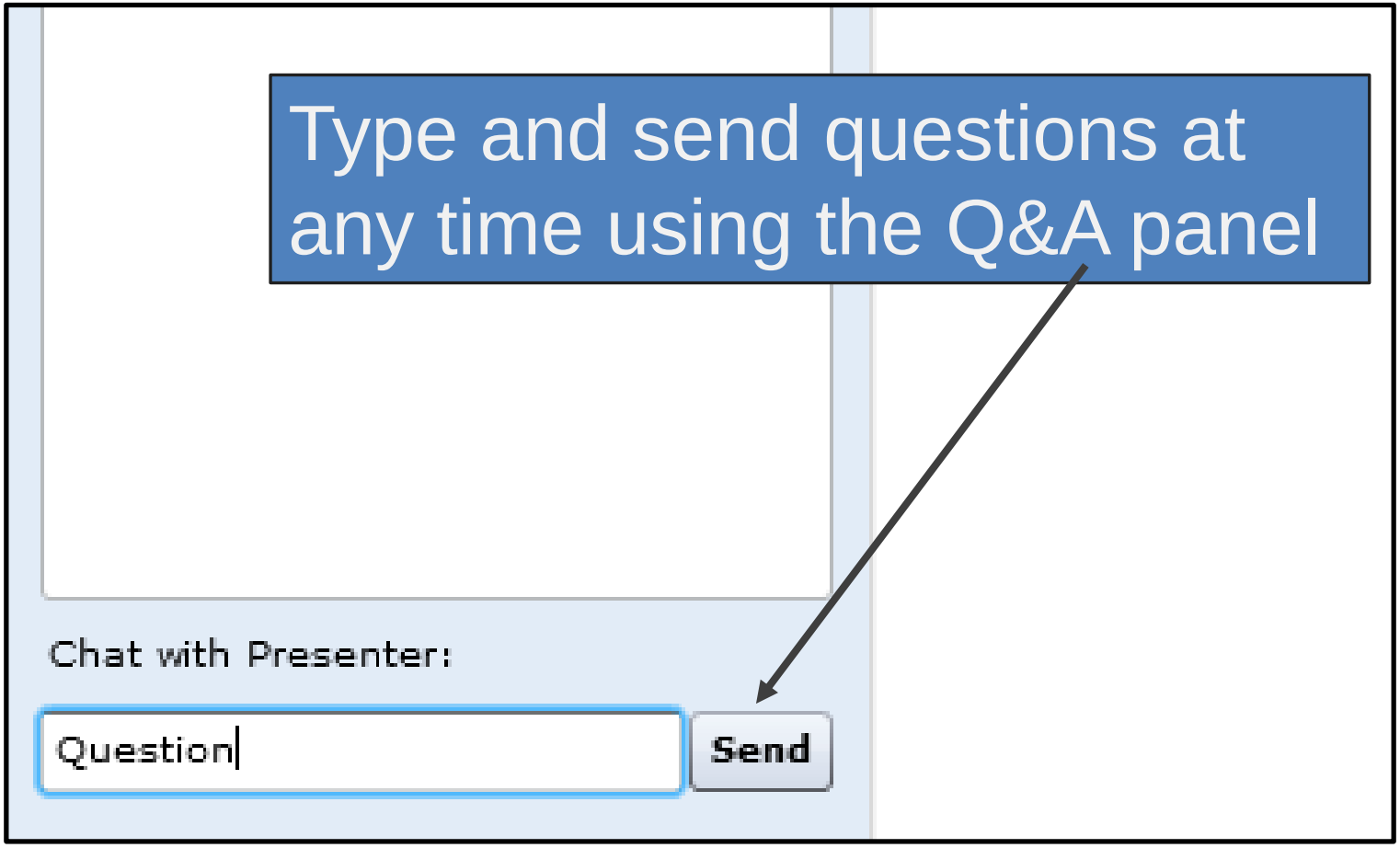


Rula A. Deeb, Ph.D.
Webinar Coordinator

Webinar Agenda

- **Webinar Logistics** (5 minutes)
  Dr. Rula Deeb, Geosyntec Consultants
- **Overview of SERDP and ESTCP** (5 minutes)
  Dr. Robin Nissan, SERDP and ESTCP
- **Environmentally Friendly Energetic Processing Via Resonant Acoustic® Mixing (RAM)** (25 minutes + Q&A)
  Dr. Eric Beckel, U.S. Army Armament Research, Development and Engineering Center
- **Mix-In-Case Green Processing with RAM** (25 minutes + Q&A)
  Dr. Andrew Nelson, Naval Air Warfare Center Weapons Division
- **Final Q&A session**

How to Ask Questions



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  - If delays continue, call into the conference line
    - Call into the conference line: 303-248-0285
    - Required conference ID: 6102000
- Submit a question using the chat box

SERDP and ESTCP Overview

Robin Nissan, Ph.D.
SERDP and ESTCP

SERDP

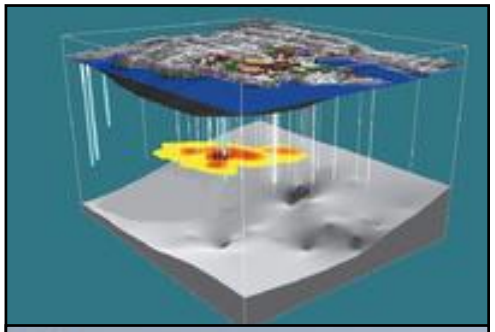
- Strategic Environmental Research and Development Program
- Established by Congress in FY 1991
  - DoD, DOE and EPA partnership
- SERDP is a requirements driven program which identifies high-priority environmental science and technology investment opportunities that address DoD requirements
  - Advanced technology development to address near term needs
  - Fundamental research to impact real world environmental management

ESTCP

- Environmental Security Technology Certification Program
- Demonstrate innovative cost-effective environmental and energy technologies
  - Capitalize on past investments
  - Transition technology out of the lab
- Promote implementation
  - Facilitate regulatory acceptance

Program Areas

1. Environmental Restoration
2. Installation Energy and Water
3. Munitions Response
4. Resource Conservation and Resiliency
5. Weapons Systems and Platforms



Weapons Systems and Platforms

- Major focus areas
  - Surface engineering and structural materials
  - Energetic materials and munitions
  - Noise and emissions
  - Waste reduction and treatment in DoD operations
  - Lead free electronics

SERDP and ESTCP Webinar Series

Date	Topic
June 14, 2018	Approaches to Managing Threatened, Endangered and At-Risk Bird Species
June 28, 2018	Energy Sustainable Wastewater Treatment Systems for Forward Operating DoD Installations
July 12, 2018	Innovative Low-cost Building Automation Sensors and Controls
August 16, 2018	Informing Restoration Programs for Threatened and Endangered Plant Species
September 6, 2018	Managing Sites Impacted by 1,4-Dioxane: New Developments in Molecular Tools and Conceptual Site Models
September 20, 2018	Plant Diversity and Biological Nitrogen Fixation in Longleaf Pine Ecosystems at Military Installations

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Save the Date

SERDP • ESTCP
SYMPOSIUM
2018 | Enhancing DoD's Mission Effectiveness

A three-day symposium showcasing the latest technologies that enhance DoD's mission through improved environmental and energy performance

November 27 - 29, 2018

Washington Hilton Hotel

Registration is now open!

Environmentally Friendly Energetic Processing Via Resonant Acoustic® Mixing

Eric Beckel, Ph.D.

U.S. Army Armament Research, Development and Engineering Center

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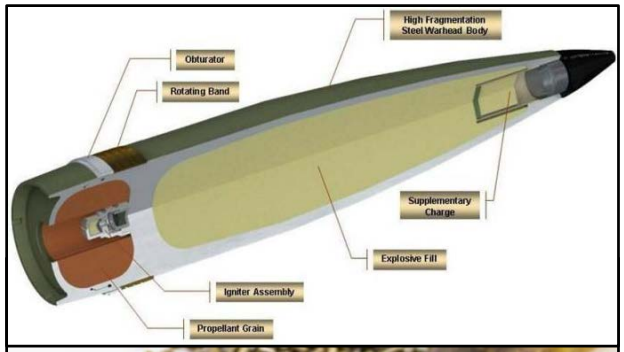
Agenda

- Project motivation
- Background
  - Resonant Acoustic® Mixing (RAM)
  - Pressing powder generation
- Results for waterless slurry pressing powder generation
- Conclusions

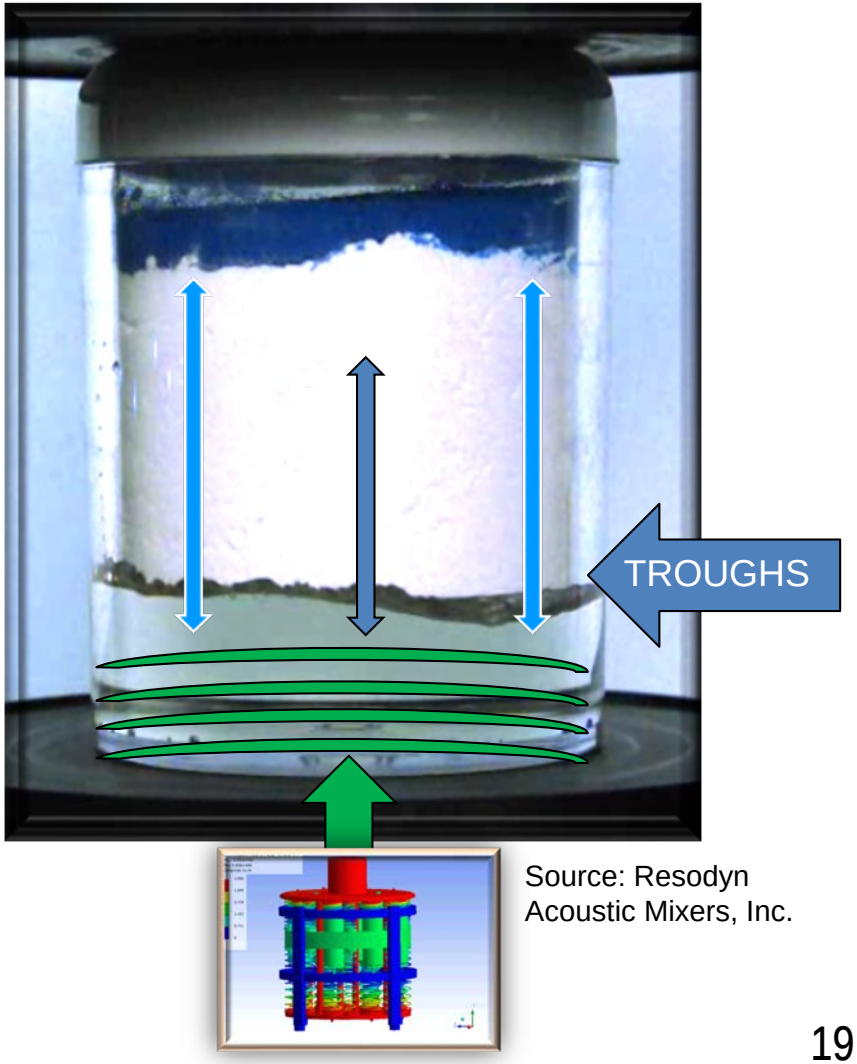
Project Motivation

- Energetic materials are used extensively throughout the DoD
  - Explosives (main fills, boosters, fuzes)
  - Propellants (small, medium and large caliber, rocket)
  - Pyrotechnics (flares, illuminants, smoke, igniters)
- Current processing limitations
  - Cannot mix extremely high viscosity materials
  - Wastes from processing and cleaning equipment
- Need environmentally friendly processing techniques for high performance materials



RAM Background

- New technology uses resonant acoustic energy coupled with the formulation medium to perform mixing process
- Bladeless process produces Faraday Instabilities within the medium for rapid and efficient blending



Source: Resodyn Acoustic Mixers, Inc.

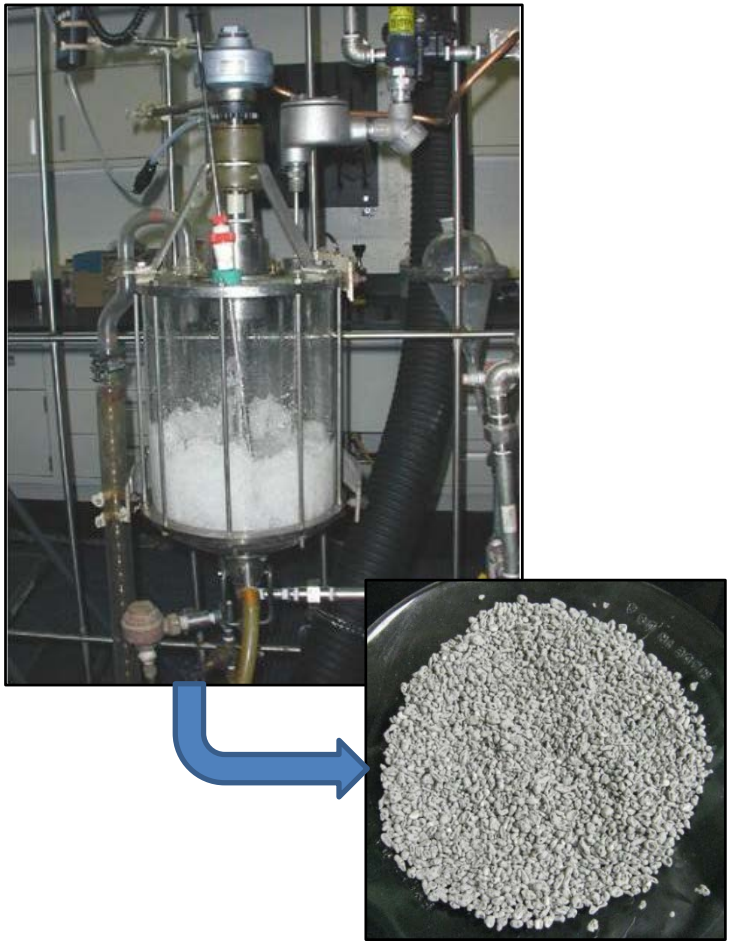
RAM for Energetics

- RAM has been used for almost a decade for processing energetic materials
  - Predominantly used for highly solids loaded cast-cure formulations
    - Develop “extreme viscosity” materials that cannot be processed with traditional high shear mixing techniques
- RAM technology application to different areas of energetics processing
  - Five ongoing SERDP/ESTCP projects related to RAM processing of energetic materials

Pressing Powder Background

Formulations processed via slurry coating

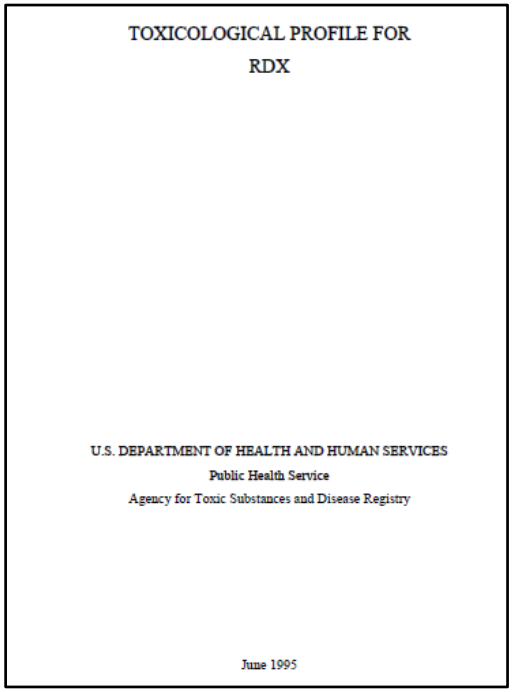
- Energetic crystals slurried in aqueous emulsion
- Binder dissolved in solvent and lacquer added to emulsion
- Solvent stripped from system allowing binder to precipitate and coat energetic crystals



Pressing Powder Background

Drying and compaction

- Granules dried to ensure no moisture present
- Consolidated using high force, elevated temperature and vacuum
- Contaminated water decanted from still and remediated prior to release into environment
  - RDX and HMX can dissolve into water at approximately 12 mg/L
  - RDX Lethal Dose 50% (LD50) values for rats and mice is 71 to 118 mg/kg (HHS, 1995)



TOXICOLOGICAL PROFILE FOR

RDX

U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES

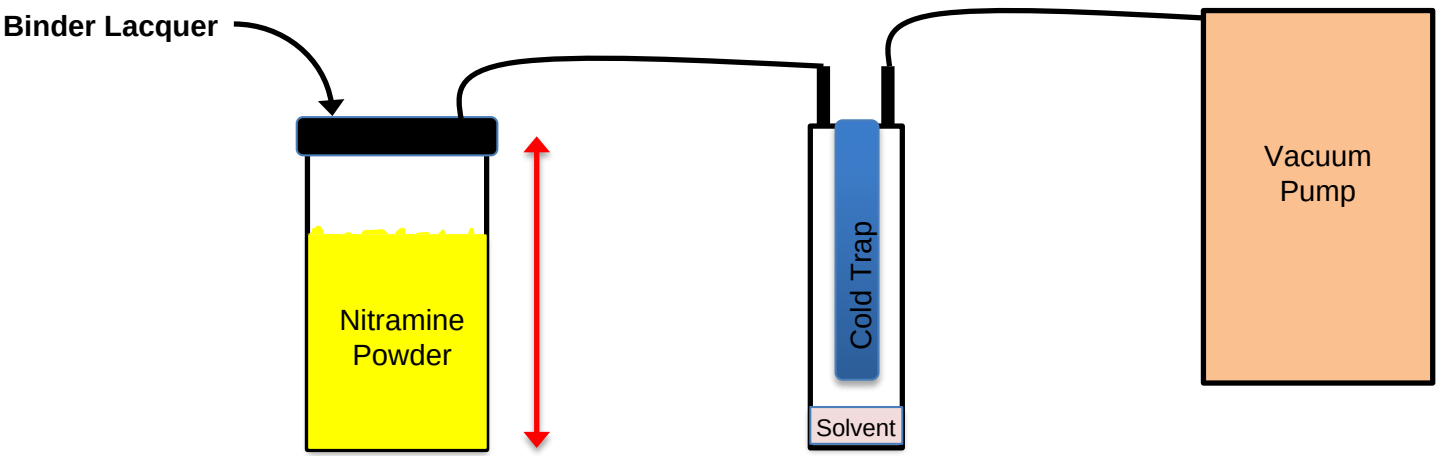
Public Health Service

Agency for Toxic Substances and Disease Registry

June 1995

Pressing Powder Water Elimination

- RAM techniques fluidize material and may allow binder to adhere to nitramine surface upon slow removal of solvent from system
- Designed an experiment to determine optimal processing parameters



Waterless Slurry

- Chose PBXN-5 as nominal formulation
  - 95% HMX, 5% Viton
- Identified five factors for the design of experiment
  - Particle size distribution (HMX Class 1 through 5)
  - Lacquer concentration (Tetrahydrofuran (THF) : Viton)
  - G-Force
  - Mix time (minutes)
  - Vacuum level (inches of Hg)
- Identified four responses for the design of experiment
  - Particle size distribution
    - Sieve #5 (4 mm) Sieve #10 (2 mm), Sieve #20 (0.841 mm), Sieve #40 (0.400 mm) and residual
  - Composition analysis
  - Safety data (impact, friction and Electrostatic Discharge (ESD))
  - Percent solvent collected in cold finger

Design of Experiment

- Chose a Resolution 4 fractional factorial experiment
  - Limited confounding with second order interactions
- Performed all runs with standard mixing vessels, 1.5:1 Height: Diameter (H:D) ratio

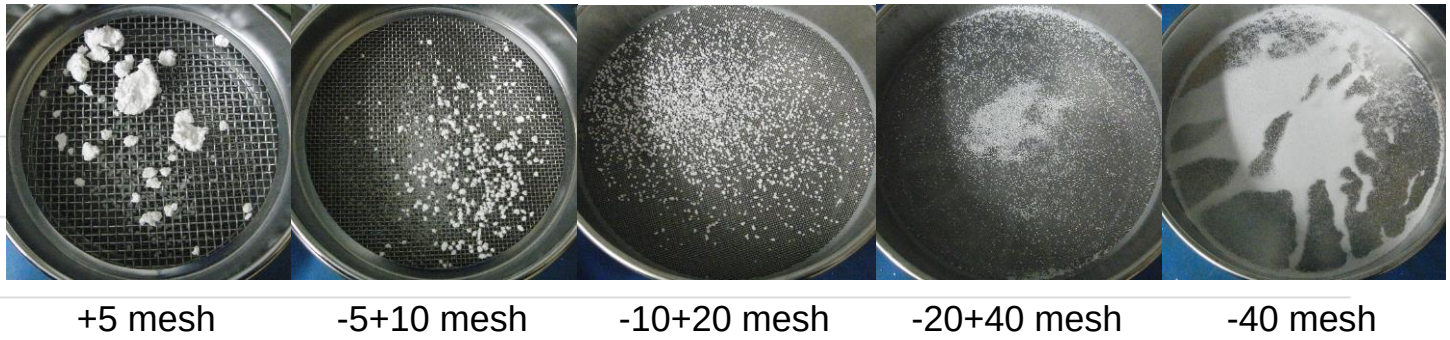
Run	Particle size (CL1:CL5)	Lacquer conc (THF:Viton)	G-Force	Mix time (min)	Vacuum (in. Hg)
1	3	4	40	10	12.5
2	3	10	40	60	12.5
3	1.665	7	60	35	25.0
4	3	4	40	60	25.0
5	0.33	4	80	10	12.5
6	0.33	10	80	60	12.5
7	0.33	10	80	10	25.0
8	3	10	80	10	12.5
9	0.33	10	40	10	12.5
10	3	10	40	10	25.0
11	3	4	80	60	12.5
12	0.33	4	40	10	25.0
13	1.665	7	60	35	25.0
14	0.33	4	80	60	25.0
15	0.33	10	40	60	25.0
16	1.665	7	60	35	12.5
17	1.665	7	60	35	12.5
18	0.33	4	40	60	12.5
19	3	10	80	60	25.0
20	3	4	80	10	25.0

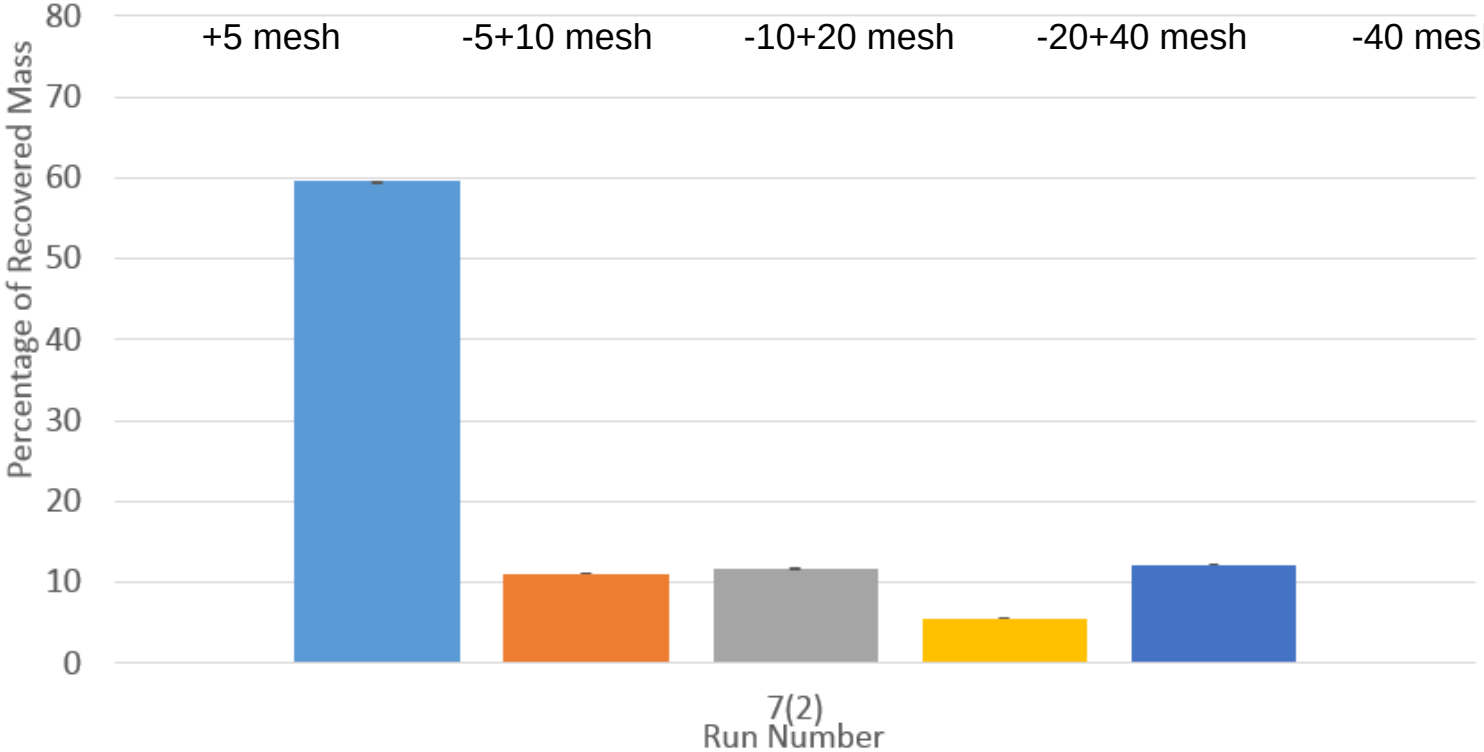
Best Results from Initial Trials

- Large, irregularly shaped, particles dominated collection fraction

- 41% THF recovered
- 80 weight % recovered as granules
- 70 - 80 G within 5 minutes
- High residual THF

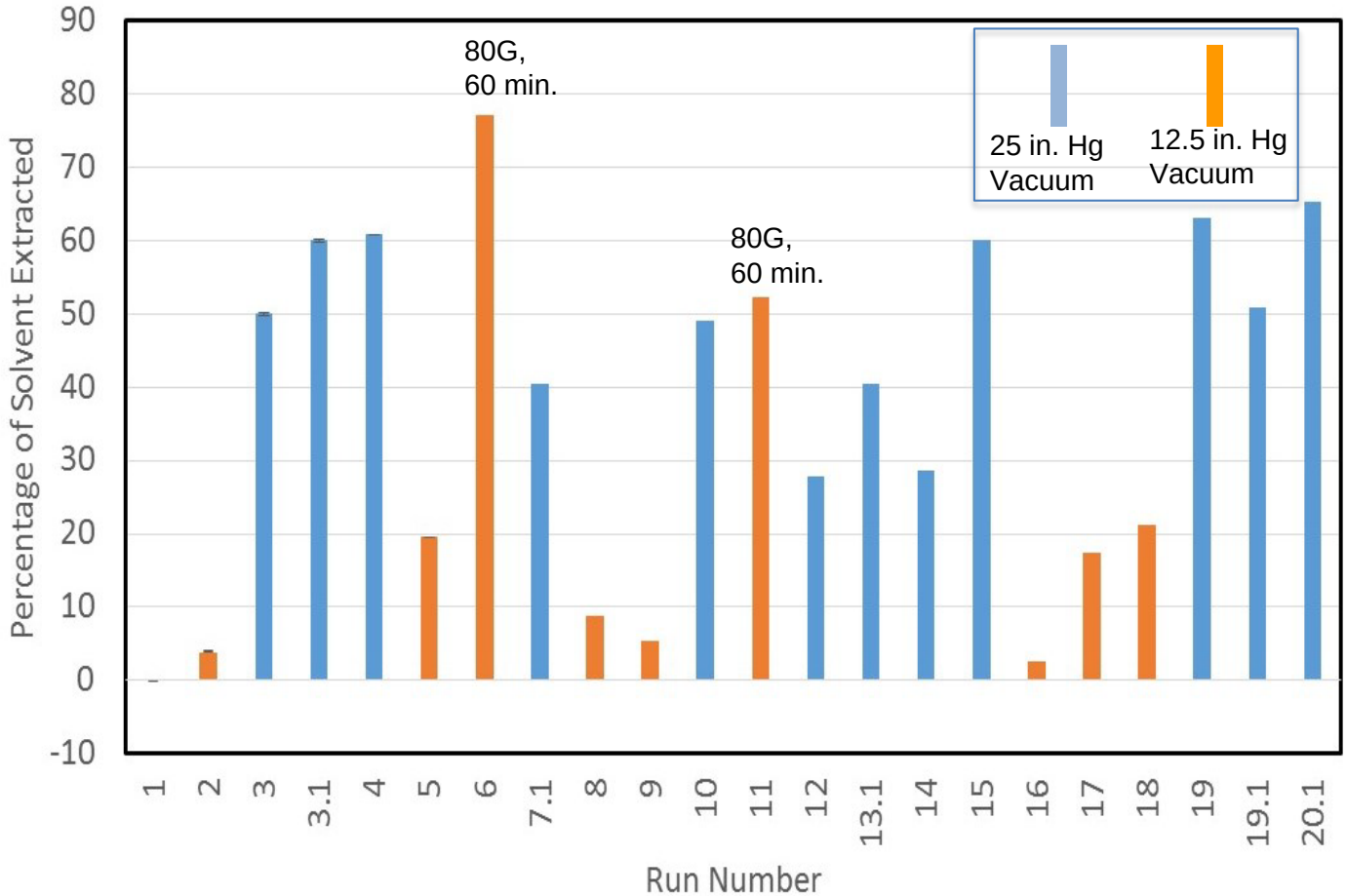
Best Results from Initial Trials





Solvent Extraction

Full vacuum needed to extract most solvent*



*Full vacuum numbers may be slightly lower due to boil-off in solvent trap

Lessons Learned from Initial Trials

- Full vacuum needed to quickly vaporize solvent to form granules
- High H:D ratio mixing vessels may not allow rolling action to round and polish the granules
  - Performed inert studies with lower H:D ratio vessels

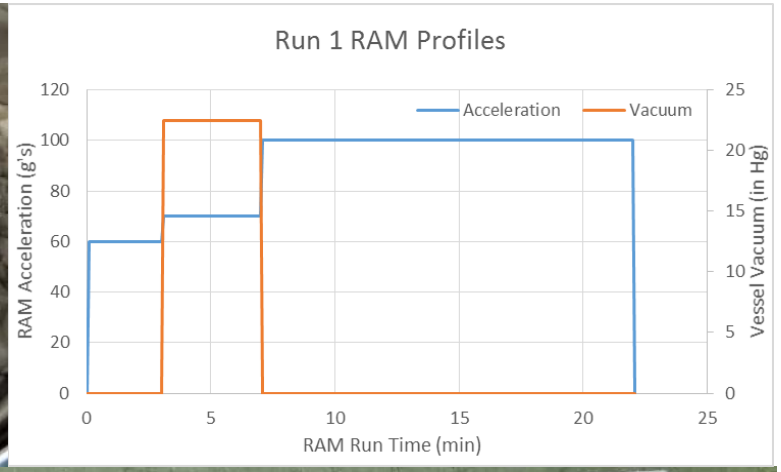
Optimized H:D Ratio Vessel Live Runs

- Determined optimal H:D ratio was 0.38 for forming narrow distribution granules
  - Stainless steel mixing vessel
  - Slightly rounded bottom
- Performed small scale run of PBNX-5
  - HMX:Viton was 90:10 (not the desired 95:5)
  - Binder lacquer concentration was 4:1 THF:Viton

Run 1: Mix 22 min, High Vacuum 4 min

Recovered 68% as particles, 95% in correct bins

- Over-dried, irregular granules, vessel wall residue

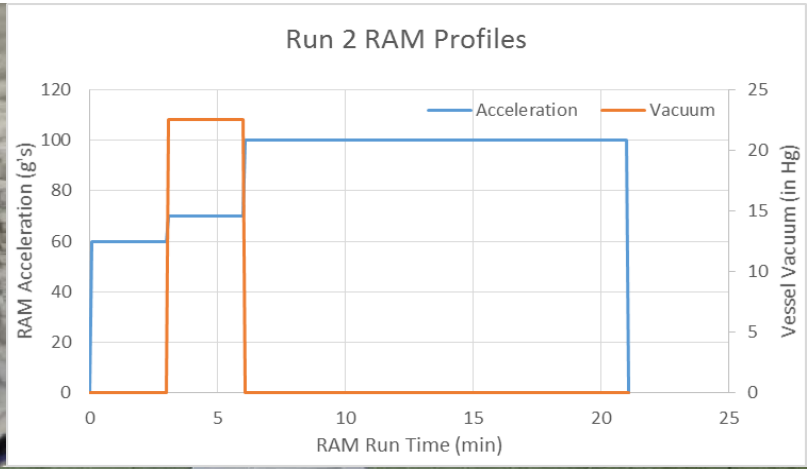


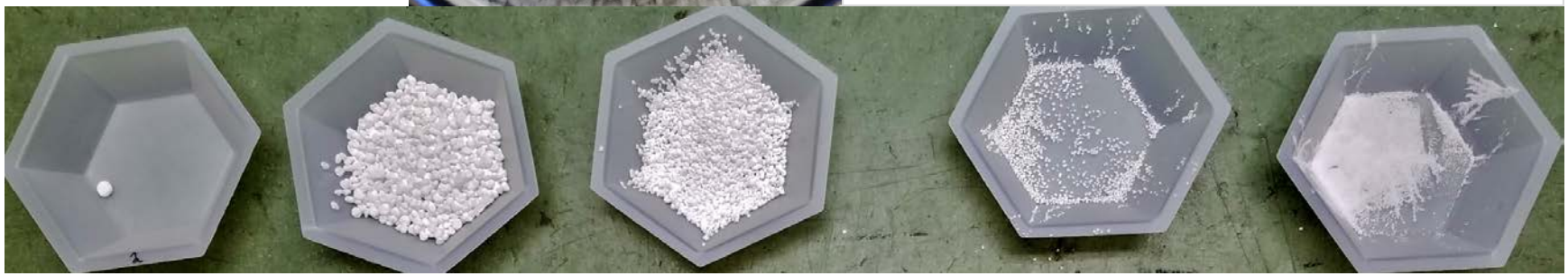
+5 mesh | -5+10 mesh | -10+20 mesh | -20+40 mesh | -40 mesh

Run 2: Mix 21 min, High Vacuum 3 min

Recovered 82% as particles, 96% in correct bins

- Slightly over-dried, slightly irregular granules, vessel partially self-cleaned



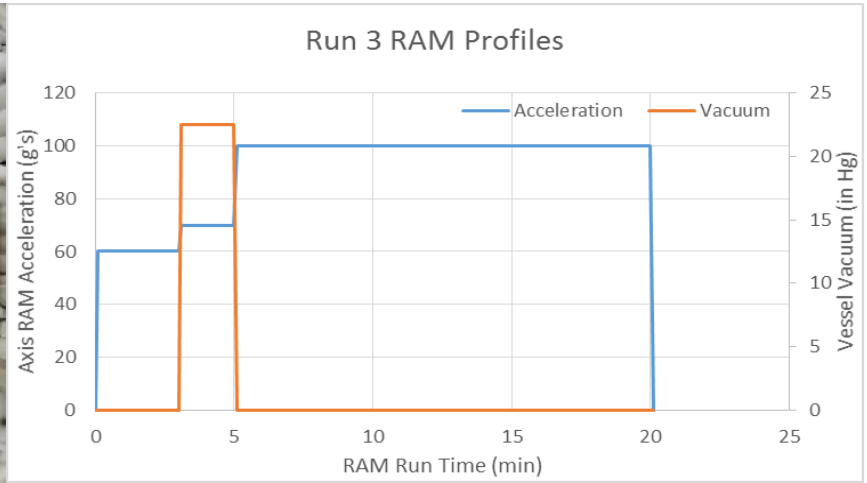


+5 mesh -5+10 mesh -10+20 mesh -20+40 mesh -40 mesh

Run 3: Mix 20 min, High Vacuum 2 min

Recovered 94% as particles, 99% in correct bins

- Nearly ideal, highly spherical granules, vessel mostly self-cleaned, may completely clean with longer run time

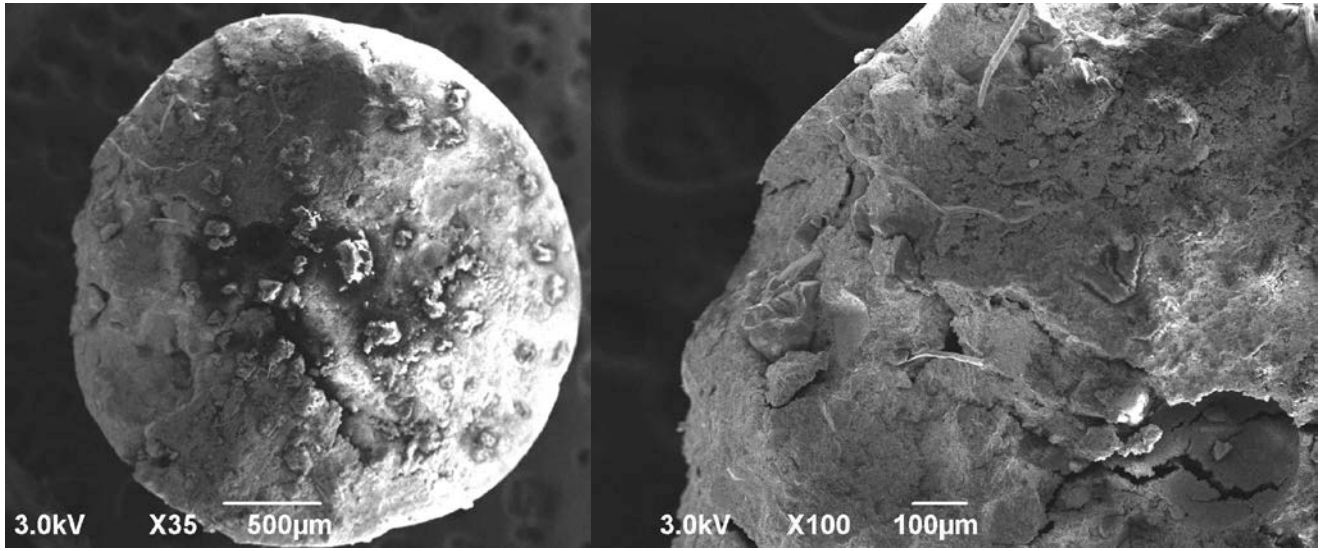


+5 mesh -5+10 mesh -10+20 mesh -20+40 mesh -40 mesh

Optimized Live Run Results

Close match between actual and nominal composition

Material	Nominal Composition	Actual Composition
HMX	90%	90.05% ± 0.03%
Viton	10%	9.95% ± 0.03%



Scanning electron microscope (SEM) analysis shows good coating of energetic material

DoD Benefits

- Alternate manufacturing method for producing pressing powder formulations established
  - Manufacturing risk reduction
- Increased throughput for pressing powder formulations
  - Decrease batch time from 4 to 5 hours to 30 minutes

Conclusions

- Wide range of benefits for RAM energetic processing
  - Reduces waste via mix in-item and continuous mixing
  - Eliminates contaminated water for pressing powder generation
- Demonstrated a waterless process for pressing powder generation
  - Narrow distribution granule formation
  - High yield
- Scale process to larger quantities to further demonstrate proof-of-concept

Acknowledgements

- Rocky Mountain Scientific Laboratory
  - Ross Nellums
  - Jon Kosak
- U.S. Army Armament Research, Development and Engineering Center
  - Explosives Research Branch
    - Paul Anderson
    - Christopher Pizzo
  - Propellant Surveillance Branch
    - Henry Grau
    - Aleksander Gandzelko
    - Robert Decker
  - Explosives Development Branch
    - Philip Samuels

For additional information, please visit

https://www.serdp-estcp.org/Program-Areas/Weapons-Systems-and-Platforms/Energetic-Materials-and-Munitions/Explosives/WP-2604

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Q&A Session 1

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Mix-in-Case Green Processing With Resonant Acoustic Mixing

Andrew Nelson
Naval Air Warfare Center
Weapons Division

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Agenda

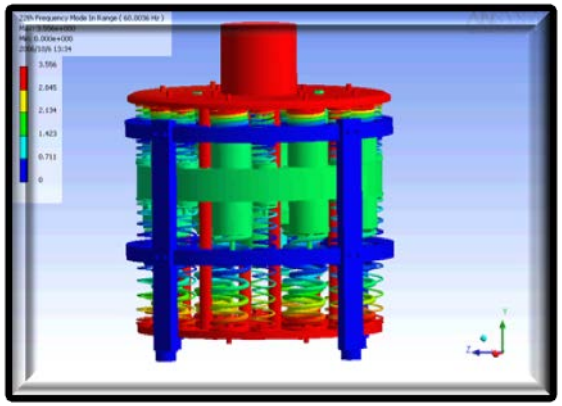
- Problem statement
- What is Resonant Acoustic® Mixing (RAM)?
- RAM machines
- Advantages of RAM
- Old mix and cast routine
- RAM mix-in-case paradigm
- Conclusions

Problem Statement

- High energy composite materials are currently produced in batch style mixers, kettles, tumblers, extruders etc.
- "Mix" and "cast" process
  - Requires solvent, energy, and intensive labor
  - Produces large quantities of hazardous waste
  - Imposes significant risk to the health and safety of operators
- New processing techniques are needed to eliminate the use of batch style equipment
- DoD benefits
  - Cost
    - Less wasted material and material disposal fees
  - Agility
    - Infrastructure not dedicated to a single production line
  - Safety
    - Reduced exposure of personnel to high energy materials and volatile organic chemicals

What is RAM?

- Mixing with:
  - High intensity [5-100 x Force of Gravity (Gs)]
  - Low frequency (60 hertz)
  - Vibration waves
- If you can bolt it to the platform you can mix in it!



Acoustic resonator platform

Solid-solid

Liquid-liquid-gas

Liquid-solid (pastes)

RAM Machines

LabRAM

- 1 pint mixer
- 500 gram capacity
- Separate vacuum and temperature control

LabRAM II

- 1 quart mixer
- 1 kg capacity
- Integrated vacuum and temperature control

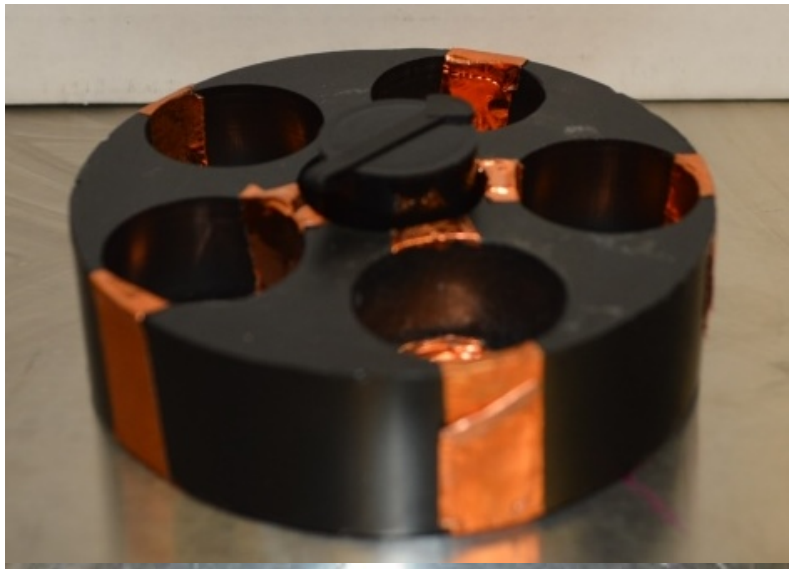
RAM5*

- 5 gallon mixer
- 37 kg capacity
- Integrated vacuum and temperature control

*A RAM55 (55 gallon mixer) is also available

Advantages of RAM – Safety

- No blades
  - No pinch points
  - No blades to scrape clean
- New materials can be dangerous
  - Use a remote control machine
- Start small to reduce risk
  - Navy regulation
    - No more than 5 grams
  - Smallest remote control bladed mixers – 25 to 50 grams
  - Historically mixed by hand
- RAM mixes as little as a gram with remote control!



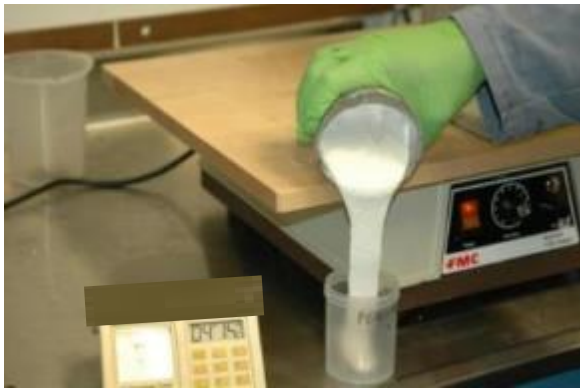
Advantages of RAM – Speed

- RAM
  - Put it all in the pot at once!
  - Single step mix routines
  - 5-15 minute process time
  - NOT limited by material viscosity
- Bladed mixer
  - Multiple material additions
  - Multiple 20-30 minute mixing cycles
  - 4-6 hour process time
  - Limited by material viscosity

Increased production rates = Increased profits and decreased cost

Extreme Viscosity = New Materials

- No blades to mix means no blades to stop
- Increased viscosity means increased performance and decreased sensitivity



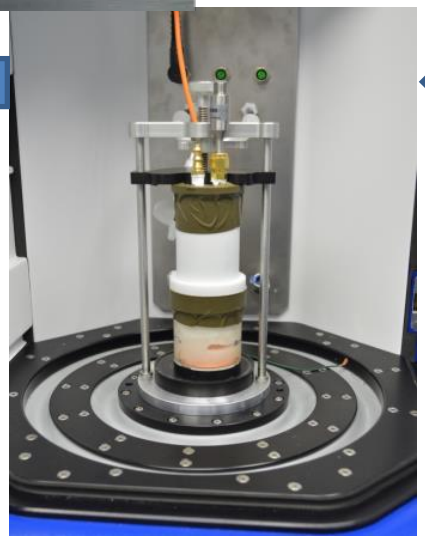
Old Mix and Cast Routine

- Mix in one facility and cast in another
- Clean up the mixer and the bowl at the end of the mix

Mix-in-Case Paradigm

Load all raw ingredients

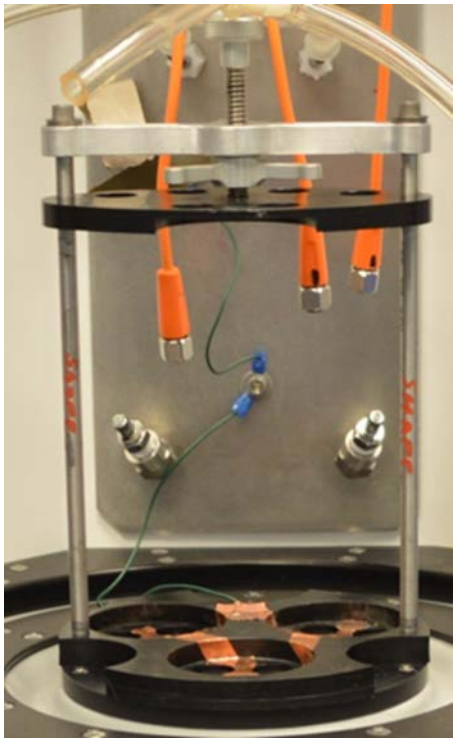
1. Binder ingredients
2. Explosive solids
3. Curative and catalysts

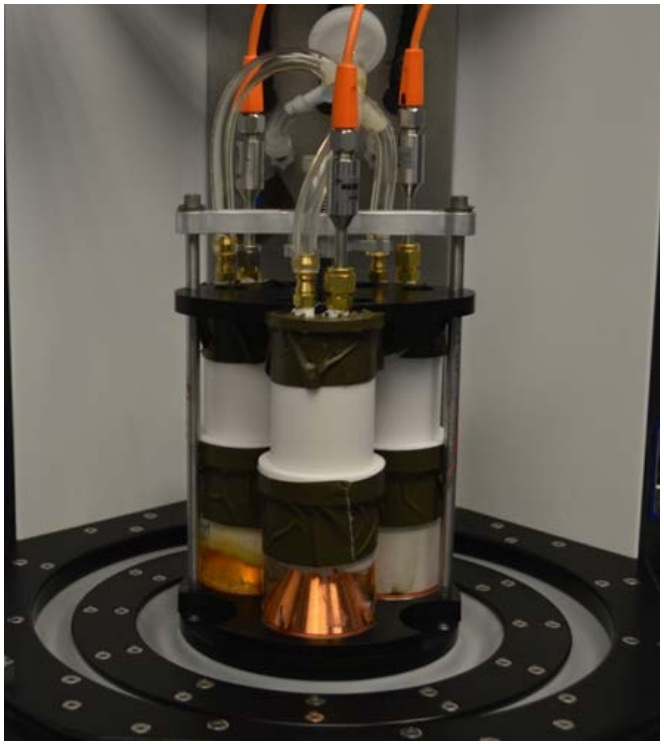


Curing oven

Increased Throughput

- LabRAM II has increased payload capacity
  - Mix multiple warheads in one routine





How Good is the Mixing?

- Composition analysis from the top, middle and bottom of each warhead
  - Global standard deviation for solids content PBX-A ± 0.217 wt.%
  - Global standard deviation for solids content PBX-B ± 0.09 wt.%
  - Mil-spec normally ± 2.0 wt.%

Lab-Scale Production Demo

- Station 1
  - Pre-polymer, plasticizer, and wetting agent
- Station 2
  - Explosive CXM
- Station 3
  - Isocyanate curative, catalysts, and anti-oxidant
- Station 4
  - Vacuum cap assembly
- Station 5
  - LabRAM II operator (not pictured)

One gallon mix and cast operation was conducted for comparison to the RAM operation

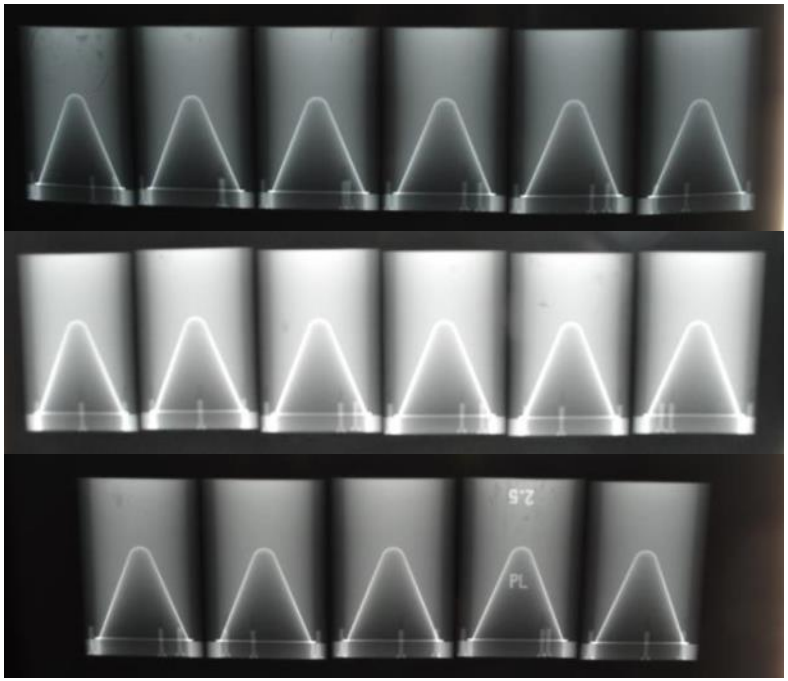
Lab-Scale Production Demo

- Total solid waste produced measured by weighing scrap bag
- Volume of cleanup solvent measured by refilling each bottle to the red line

Higher Quality Products

- Mix-in-case
  - 18 warheads prepared
  - 3 would likely be rejected for void content
  - 1 questionable

- Mix and cast
  - 17 warheads prepared
  - 7 would likely be rejected for void content
  - 2 questionable



Faster Production

Mix-in-case

- 18 Shape Charge Jet warheads
  - 4500 grams of material
  - 3.5 hr operation
  - 17.5 man-hours
  - 41 warheads/8 hr shift
  - 0.97 man-hour/warhead (5 man crew)
  - 0.77 man-hour/warhead (4 man crew)

Mix and cast

- 17 Shape Charge Jet warheads
  - 4500 grams of material (1 gallon mixer)
  - 8.5 hr operation
  - 26 man hours
  - 16 warheads/8 hr shift
  - 1.53 man-hours/warhead

Reduced Environmental Impact

Mix-in-case

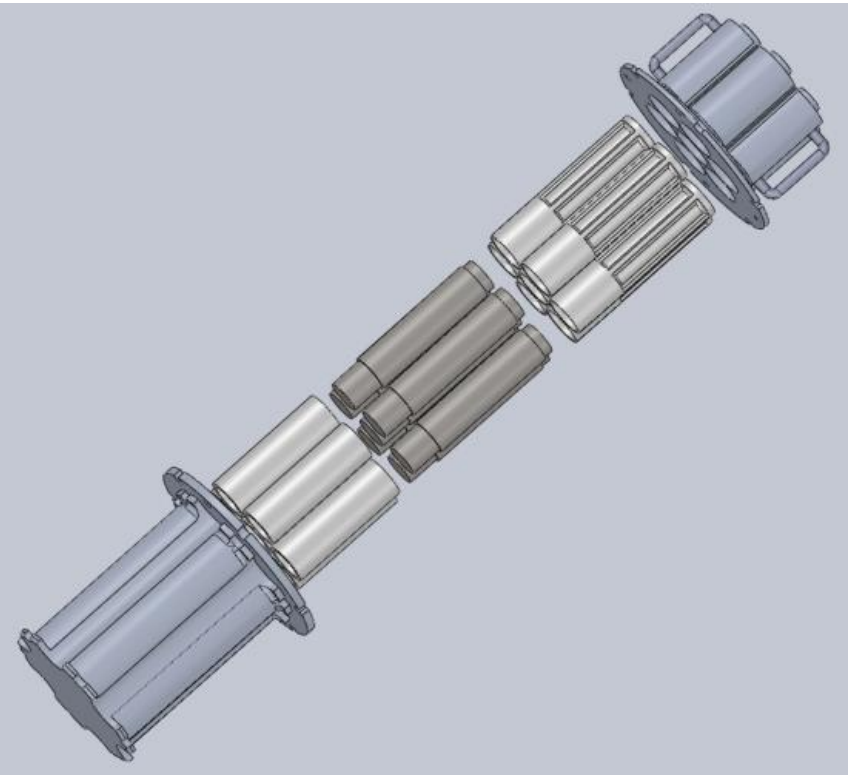
- Total cleanup solvent
  - 115 ml ≈ 6.5 ml solvent / warhead
- Total mass of hazardous waste
  - 672 grams = 37 grams waste / warhead

Mix and cast

- Total cleanup solvent
  - 519 ml ≈ 30.5 ml solvent / warhead
- Total mass of hazardous waste
  - 1,712 grams = 101 grams waste / warhead

RAM operation decreased the cleanup solvent by 79% and the hazardous waste by 63%

Future Full-Scale Warhead Production



- RAM5 warhead fixture
- 7 warheads / routine

Conclusions

- RAM uses high intensity, low energy vibration to mix everything without blades
- RAM advantages
  - Improved personnel safety
  - Improved production speed
  - New high viscosity materials
  - Mix-in-case
- RAM mix-in-case benefits
  - Improved production
  - Decreased environmental impact
  - Decreased facility infrastructure

For additional information, please visit

https://serdp-estcp.org/Program-Areas/Weapons-Systems-and-Platforms/Energetic-Materials-and-Munitions/Explosives/WP-201507

Speaker Contact Information

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Q&A Session 2

SERDP DOD • EPA • DOE | ESTCP

The next webinar is on June 14, 2018

Approaches to Managing Threatened, Endangered and At-Risk Bird Species

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Survey Reminder

Please take a moment to complete the survey that will pop up on your screen when the webinar ends

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