



## AVT-340 Research Workshop on Preparation and Characterization of Energetic Materials

Strategic Environmental Research and Development, Environmental Security Technology Certification Program Energetic Material Overview

11 February 2021

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## Strategic Environmental Research and Development Program (SERDP)

- Established by Congress in FY 1991
  - ♦ DoD, DOE, and EPA partnership
  - Advanced Development Program with statutory authority to support basic research through advanced development
- High-priority environmental science and technology areas that address
  - DoD unique issues
  - Environmental issues with large costs to DoD



# **Environmental Security Technology Certification Program (ESTCP)**

- Established by DoD in FY 1995
  - ♦ 6.4 Demonstration/Validation program
- Demonstrate innovative and cost-effective environmental and energy technologies
  - Capitalize on past investments
  - Transition technology out of the lab
- Promote implementation
  - Facilitate regulatory acceptance



# **Environmental Drivers**

## Sustainability of Ranges, Facilities, and Operations

90N



Threatened and Endangered Species Maritime Sustainability



**Toxic Air Emissions and Dust** 

8



Noise



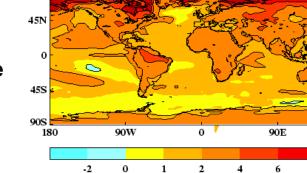


UXO & Munitions Constituents



Sustainable FOB



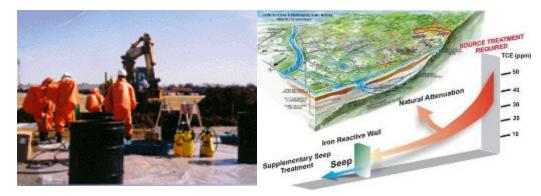




# **Environmental Drivers**

## **Reduction of Current and Future Liability**

### **Contamination from Past Practices**



- Groundwater, Soils and Sediments
- Large UXO Liability
- Emerging Contaminants

Pollution Prevention to Control Life Cycle Costs





- Elimination of Pollutants and Hazardous Materials in Manufacturing, Maintenance, and Operations
- Achieve Compliance Through Pollution
   Prevention
- Develop and Assess Alternative Technologies



# Program Area Management Structure

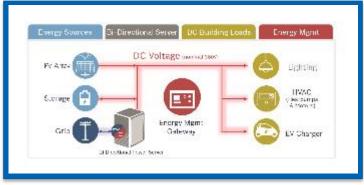
### Weapons Systems & Platforms





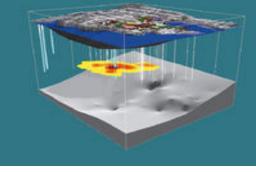
Resource Conservation & Resiliency

## **Energy and Water**



**ESTCP** Only

### Environmental Restoration





## Munitions Response



# **Investment Strategy**

- Longer term strategic plans to address highpriority requirements
  - groundwater contamination in Environmental Restoration
  - elimination of hexavalent chromium, cadmium, and lead in Weapons Systems and Platforms
  - T&E species management in Resource Conservation and Resiliency
  - synthesis and formulation of energetic materials
- Nimble enough to respond to high-priority emerging needs
  - perchlorate alternatives in pyrotechnics
  - ♦ infrastructure resilience
  - PFOS/PFOA



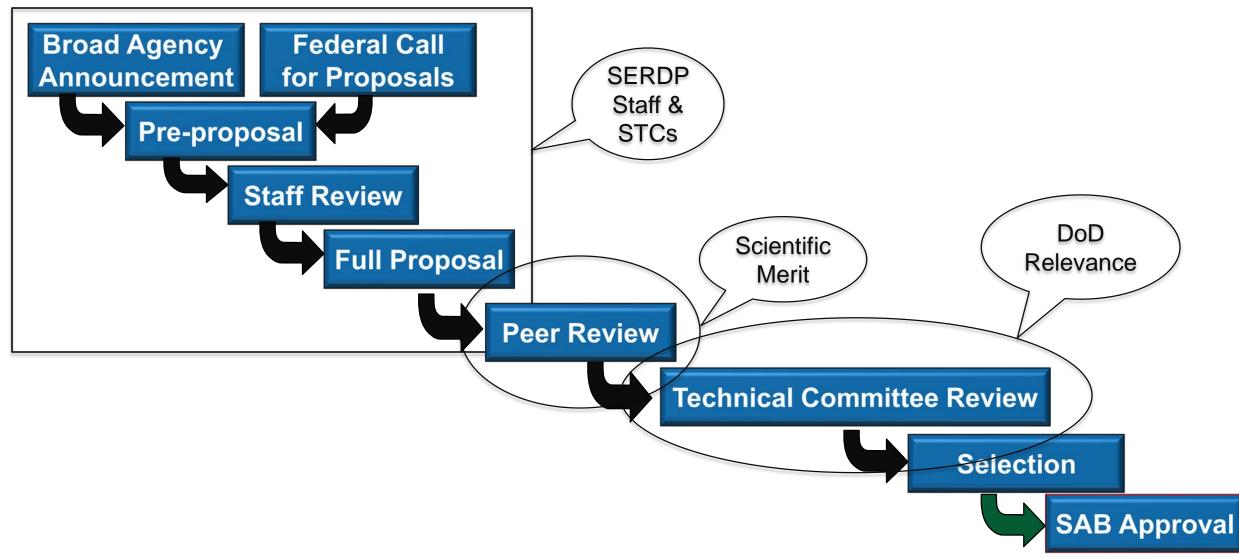


# **SERDP Technical Committees**

- Members represent technical staff and research management from SERDP Federal Partners, DoD Services, EPA, and DOE
- Responsible for first level planning, review and development of program
- Provide significant time to SERDP
  - Key to the effective execution of SERDP
- Provide method for coordination across agencies



# **SERDP Proposal Selection Process**





# **Recent SERDP WP Statements of Need**

- FY 19-Aircraft Engine Noise Reduction Technology
- FY 19-Predictive Corrosion Models to Mitigate Environmental Hazards
- FY 19-Additive Manufacturing of Gun Propellants with Reduced Environmental Impact (4)
- FY 19-Novel Pyrotechnics that Reduce Environmental Impact (5)
- FY 19-Multifunctional Fibers and Textiles for Warfighter Integrated Protection

FY 20-Development of Advanced Coating Systems

FY 20-Advanced Synthesis Techniques for Military-Relevant Energetic Materials or Significant Precursors (4)

FY 20-Novel Solutions for Prevention of CMAS Accumulation in Gas Turbine Engines

- FY 20-Environmentally Benign Rocket Propellants (1)
- FY 20-Development of New Approaches for Demilitarization of Conventional Military Munitions
- FY 20-Reduction of Hazardous Waste Streams from Composite Manufacturing and Repair
- FY 20-Supplemental-Innovative Approaches to Fluorine-Free Fire Fighting Agents

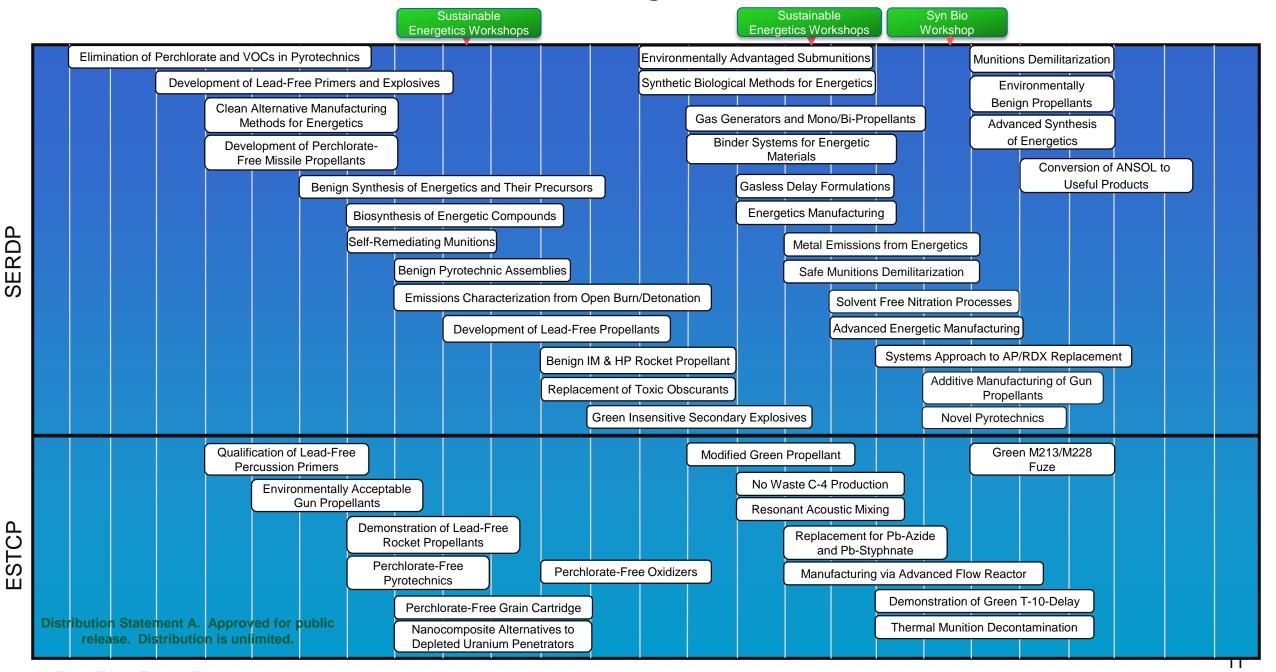
#### FY 21-Conversion of Ammonium Nitrate Solutions to Useful Products

- FY 21-Optimization of Advanced Battery Processing and Recycling Technologies
- FY 21-Development of Chromium-Free Post Treatment Sealers
- FY 21-Structural Repair of Defense Assets

#### SERDP & ESTCP

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### **SERDP and ESTCP Energetic Materials Efforts**



FY01 FY02 FY03 FY04 FY05 FY06 FY07 FY08 FY09 FY10 FY11 FY12 FY13 FY14 FY15 FY16 FY17 FY18 FY19 FY20 FY21 FY22 FY23 FY24 **FY25** 



# **Synthesis Methods**



## WP20-1391: Sustainable, Low Cost Production of Biosynthetic 1,2,4-Butanetriol and Butanetriol Trinitrate

#### **Performers:**

- Benjamin Harvey-Naval Air Warfare Center, Weapons Division (NAWCWD) China Lake
- Michael Lynch-Duke University, Dept. of Biomedical Engineering

### **Technology Focus**

Biosynthetic production of energetic plasticizers for low smoke propellants

### **Research Objectives**

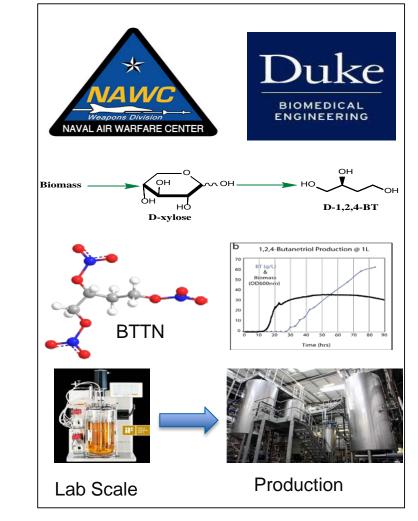
 This project will seek to develop a scalable, low cost process for the production of the energetic plasticizer, butanetriol trinitrate (BTTN), from biosynthetic 1,2,4-butanetriol (BT). BT will be synthesized from a biomass derived sugar with metabolically engineered E. coli.

### **Underlying Technology**

Duke University has developed a cell-free process to generate BT from xylose at over 60 g/L.
 NAWCWD has extensive experience in the purification and chemical transformation of biosynthetic substrates

### **Technology Transition**

 BTTN generated during the course of this project will be used to prepare propellant formulations for tri-service demonstration programs focused on testing propellants in realistic environments.



# WP20-1110 Precursors of the High Explosive Hexanitroffexaazaisowurtzitane (CL-20)

Performers: Nigel Scrutton-The University of Manchester

Benjamin Harvey-Naval Air Warfare Center Weapons Division

### **Technology Focus**

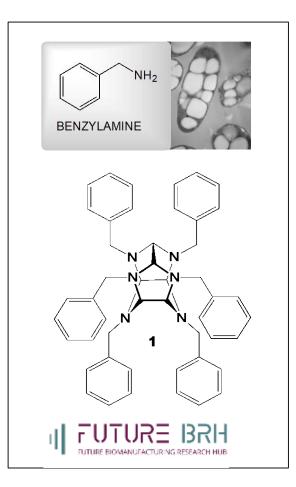
• Develop innovative, scalable and environmentally sustainable bio manufacturing routes to precursors of the high explosive CL-20

#### **Research Objectives**

- Develop innovative, scalable and environmentally sustainable bio-manufacturing routes to benzylamine.
- Enable development of a more cost effective and semi-synthetic route to the high explosive hexanitrohexaazaisowurtzitane (CL-20).
- Assist the DoD by generating an alternative strategy that will reduce overall costs and environmental challenges associated with production of CL-20

#### **Technology Transition**

Engage industrial end-users and chemical manufacturers, in partnership with the FBRH. Ensure
processes being developed are "fit for purpose" and respond to industrial need. Identify commercial
partner to co-develop the scale-up to make it economically viable and pull benzylamine production
through to larger-scale.



## WP20-1125 Retrobiosynthetic Design and Genome Mining for Renewable Energetic Materials

#### **Performers:**

- Dr. Ben Gordon (MIT, Director of MIT-Broad Foundry)
- Dr. Deepti Tanjore (LBNL, Director of DOE ABPDU)

### **Technology Focus**

• Development of biosynthetic routes to precursors to energetic targets, which will have greatly reduced waste streams compared to current chemical processes.

#### **Research Objectives**

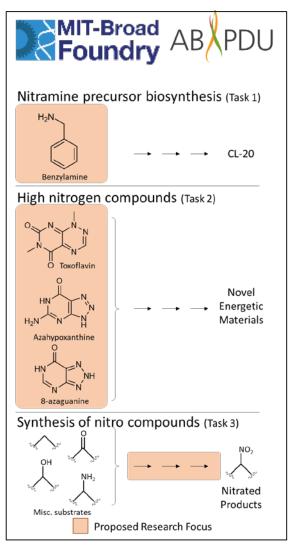
- Design, prototype, and debug biological systems to produce benzylamine and high-nitrogen compounds.
- Develop a biocatalysis toolset for enzymatic nitration
- POP process development and scale-up (>100L) to produce testable quantities of materials.

### **Project Progress and Results**

N/A – [Starts Spring of 2020]

#### **Technology Transition**

Materials: Will be tested by DoD research partners at service labs, in coordination with SERDP.
 Production processes: Pilot-scale methods and techno-economic analysis results will be available to license to commercial partners.





## WP20-1010 Reduction of Acidic and Toxic Waste Streams in Explosives Manufacturing Using Electrochemical Nitration

**Performers:** *Matthew Burke-Nalas Engineering Services* 

Prof. Paul Anastas group (Yale University)

#### **Technology Focus**

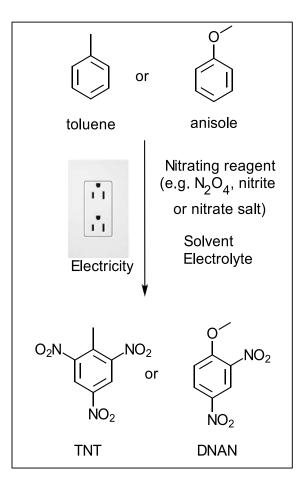
• Use of electricity to replace or supplant conventional nitration methods, which require large excess of toxic and corrosive materials.

#### **Research Objectives**

- Develop electrochemical nitration methods for aromatic nitration (TNT, DNAN)
- Adapt methods to O-nitration (NG), N-nitration (TAT/TRAT  $\rightarrow$  HMX/RDX)

#### **Technology Transition**

• Select most promising example for 6.3 followup, including scale-up, high-throughput reactor design, and environmental impact; could be transition to ESTCP or JEMTP



### WP20-1215: Reaction Acceleration of Military Relevant Energetic Compounds and Precursors through Confined Volume Methodologies

#### Performers: Patrick W. Fedick NAWCWD

### **Technology Focus**

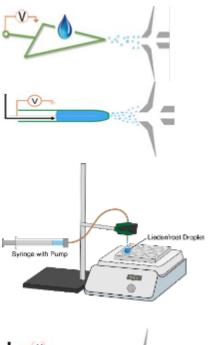
 Confined volume reactors, which encompass microdroplets, emulsions, and thin films, are synthetic methodologies that have shown to accelerate product formation compared to their counterpart traditional bulk reactions (i.e. a round-bottom flask).

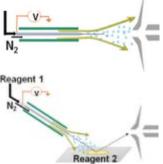
#### **Research Objectives**

- Accelerate product formation of highly desired energetic compounds that customarily have lengthy, lowyield or cost-ineffective syntheses through confined volume reactors.
- Systematically answer what physical processes cause reactions to accelerate in confined volume systems and to what extent each process is responsible for acceleration.
- Create desired energetics and energetic precursors in the fastest and most effective manner possible while decreasing hazardous waste generated.
- Rapidly screen reactions through environmentally friendly methods.

#### **Technology Transition**

• These accelerated synthetic confined volume techniques can be leveraged to shorten the development of novel energetic materials and may be applied to scale-up processes for energetics.





## **WP-201623: Energetic Manufacturing via Advanced Flow Reactions**

**Performers:** Omar Abbassi, Eric Gauthier, Daniel Iwaniuk-US Army CCDC-Armaments Center Jerry Salan, Matt Jorgenson, Matt Burke, Patrick Staiber, Andrew Pearsall-Nalas Engineering Matthew Zolnowski, Christian Footman-Alion Science and Technology

#### **Technology Focus**

• To demonstrate a continuous process for the manufacture of bis(2,2-dinitropropyl)acetal/formal (BDNPA/F) utilizing Continuous Advanced Flow Reactor technology.

#### **Demonstration Site**

• Nalas Engineering, Centerbrook CT, 2-3Q FY20

#### **Demonstration Objectives**

• **Reduction** of Aqueous and Organic Waste, **Reduction** of Operator Exposure

#### **Project Progress and Results**

- Developed dinitropropanol (DNPOH) process, waste reduction of 24% expected
- Recycling of dichloromethane extraction solvent, >80% recovery, >95% reduction expected
- Reduction of organic solvents in BDNPA/F by 57% by increased concentration of DNPOH stream

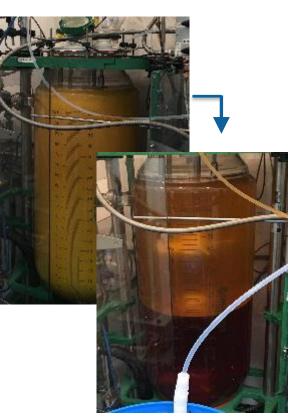
#### **Implementation Outlook**

- LCPP supporting continued process optimization at pilot-scale
- CCDC AC, PM-MAS enabling transition to scale-up for annual production











# **Formulation**

SERDP & ESTCP

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## WP-2605 Environmentally Conscious Process Development for the Production of Composite Propellants and Explosives

Performers: Mike Miller-Resodyn Corp.

Andrew Nelson-NAWCWD China Lake

#### **Technology Focus**

• Develop and Demonstrate an energetics rated Continuous Acoustic Mixing System with Clean-in-Place (CAM-CIP) and Temperature Control Capability

#### **Research Objectives**

- Develop a Surrogate PBX Material to Model Prototype CAM-CIP Production Capability and CIP Efficiency
- Design, Fabricate, and Test an Energetics Rated CAM-CIP system
- Demonstrate Energetic PBX Production at NAWCWD China Lake Using the Energetics Rated CAM-CIP System

### **Project Progress and Results**

- CAM-CIP Module Completed and Tested
- Design and Fabrication of Energetics Rated Ancillary System Underway
- Expect to Produce Energetic PBX Material in Q3 2020

### **Technology Transition**

• Interest in the Technology is High, Application Engineering for Customers is Underway



Phase 3: In Progress Transition to and Demonstration at China Lake with Energetic Material



### WP19-1246 Additive Manufacturing of Gun Propellants Using Vibration Assisted Direct Write Printing Printing Resolution Quantification

**Performers:** S.F. Son (PI), I.E. Gunduz, and J.F. Rhoads, S. Isert, and J. Ciezak-Jenkins (co-PI's)

## **Technology Focus**

Novel additive manufacturing (AM) approaches for gun propellants

### **Research Objectives**

 Implement new AM to printing viscous propellants, characterize "inks", investigate resulting structure properties, quantify propellant performance, and assess the reduction in environmental impact and lifecyle.

### **Project Progress and Results**

 Three inert ink systems developed, vibration assisted printing (VAP) and commercial systems contrasted and characterized, initial mechanical properties (dog bone testing) and printing performance characterized, novel grain configuration explored, propellant ingredients obtained and live propellant printing being planned.

### **Technology Transition**

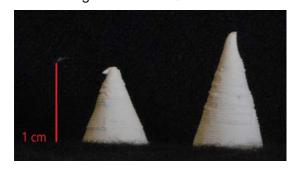
• Communicating with Picatinny Arsenal and working with ARL to make technology transition seamless

Print turning Characterization



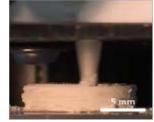
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#### Gun Propellant Model (100% infill)







Unique Grain configuration developed that cannot be fabricated without additive approach



Purdue Energetics Research Center





# **Pyrotechnics**

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## WP19-1287: Lithium Based Red Colorants in Environmentally Friendly Pyrotechnical Illuminants

Performer: Prof. Dr. Thomas M. Klapötke

#### **Technology Focus**

- Synthesis and characterization of new lithium salts
- Screening of literature-known as well as unknown lithium materials for red light-production in "drop-in" formulations
- Formulation optimization for the most promising candidates
- Prototyping

#### **Research Objectives**

- Focus on nitrogen-rich, nitro-substituted and multivalent counterions
- Use of commercially available neutral compounds or such accessible by highly scalable, lowstep syntheses without toxic reagents or solvents
- Study of the influence of the particle size and amounts of magnesium on the light intensity

#### **Project Progress and Results**

• Preparation, analysis and determination of emissive properties of new lithium materials with high nitrogen content and nitro groups

#### **Technology Transition**

- Scale-up of the lithium material to the multi-pound scale
- Dynamic function test of the M126A1 hand-held signal at a prime flare manufacturer







## WP19-1255 High Color Purity Low Smoke Solvent-free Multi-color Signal Flares

#### **Performers:**

- TNO: Richard Bouma (PI), Martijn Zebregs, Michiel Straathof
- South Dakota School of Mines and Technology: Lori Groven
- Chemring Ordnance: Matt Rexford

### **Technology Focus**

 Novel pyrotechnic formulations, novel production technologies and engineering solutions that produce multi-color signals

#### **Research Objectives**

• Advancement of production technologies for multicolor signal, using sustainable NC-based compositions, and reduced environmental and health impact. Success is based on demonstration with proof of concept and quantification of ESOH performance.

#### **Project Progress and Results**

 Requirements document is under review, NC-based colors are selected, reference colors need to be defined.

#### **Technology Transition**

• Will provided at a later stage in the project.

Chemring Ordnance







## **WP-2518: Environmentally Sustainable Gasless Delay Compositions For Fuzes**

**Performers:** Dr. Jay Poret (U.S. Army CCDC-AC),

Dr. Anthony Shaw (U.S. Army CCDC-AC),

Prof. Lori Groven (SDSMT),

Dr. William Eck (U.S. Army PHC)

#### **Technology Focus**

 Develop new, environmentally sustainable chemical delays that are reliable and adaptable so they can be used in several different fuzes, especially ones for hand grenades.

#### **Research Objectives**

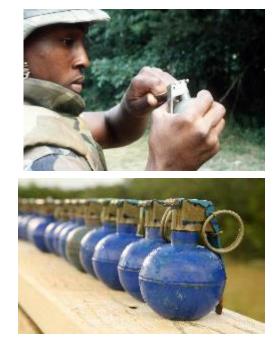
 Develop and test safer and more sustainable compositions that meet the requirements of the M201A1, M213, and M228 fuzes.

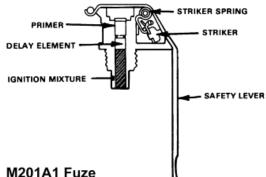
#### **Project Progress and Results**

 Delay formulations for both M201A1 and M213/M228 fuzes have been successfully developed and tested in fuze hardware. Studies on the role of perchlorate and chromate oxidizers in the legacy compositions have yielded insightful results.

#### **Technology Transition**

 The relevant stakeholders (U.S. Army CCDC-AC Fuze Division and PM-CCS) are being briefed and kept informed.





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## WP19-1372 Solving Ambient Performance Sea Salt Obscurants

#### **Performers:**

- TNO: Joost van Lingen (PI), Denise Meuken, Dinesh Ramlal
- NSWC Crane: Mackenzie Alameda

### **Technology Focus**

• Development of an environmentally benign obscurant (improve performance of 14WP04-002 candidates).

#### **Research Objectives**

 Improve the obscurant performance of compositions 15EM0281 and 15EM0282 (from 14WP04-002) at ambient temperature conditions and, if possible, also at cold conditions.

#### **Project Progress and Results**

 Testing composition 15EM0281 and 15EM0282 in laboratory scale setting in the smokebox at hot, ambient and cold conditions; Root Cause Analysis used to identify possible causes for the lower performance at ambient and cold; Laboratory analysis to determine effects of identified causes.

#### **Technology Transition**

 Once prototype is improved at ambient conditions, this technology will be proposed as an ESTCP project for final development testing and demonstration











## WP19-1196 Feasibility of a Thermoelectric Generator for a Reduced Environmental Impact Electronic Flare

#### **Performers:**

• TNO: Joost van Lingen (PI), Denise Meuken, Jos van den Elshout, François Bouquet

### **Technology Focus**

• Development of an E-Flare technology demonstrator.

### **Research Objectives**

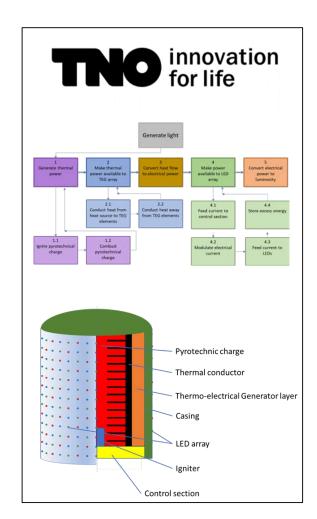
 Identify the technical and environmental feasibility of an electronic signal flare, with a specific focus on the feasibility of pyrotechnic power source; Pyro compositions will be selected and tuned to the optimal working temperatures of the Thermo-Electrical Generator.

### **Project Progress and Results**

 Definition of System requirements; Design of the E-flare technology demonstrator; Procurement of COTS Thermo-Electrical Generators (TEGs); Determination of appropriate pyrotechnic compositions for the power source;

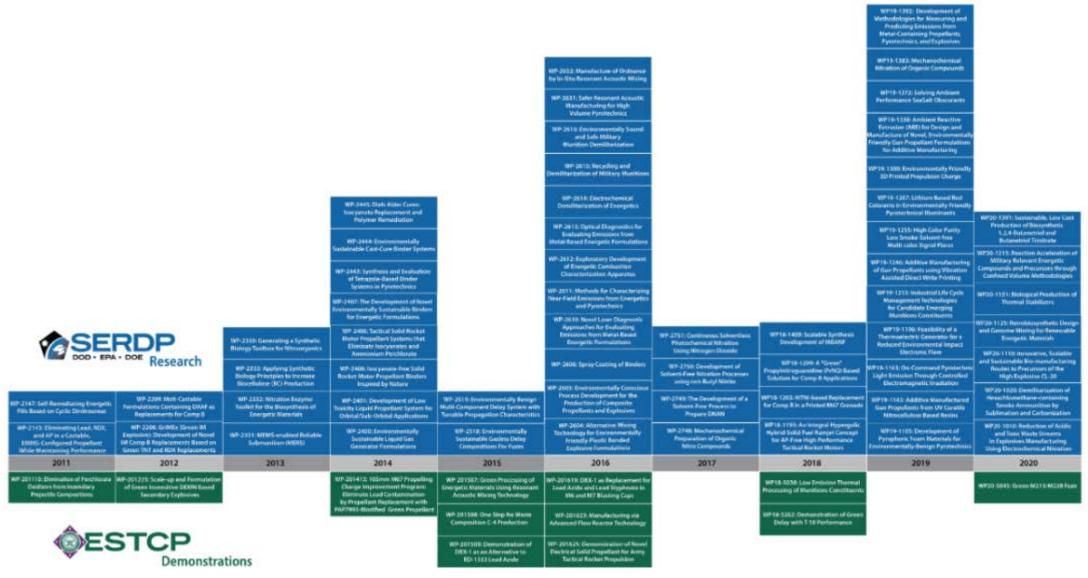
### **Technology Transition**

 If the E-flare technology demonstrator proves to be feasible, this technology will be proposed for further development, testing and demonstration





#### **SERDP and ESTCP Energetics Timeline**

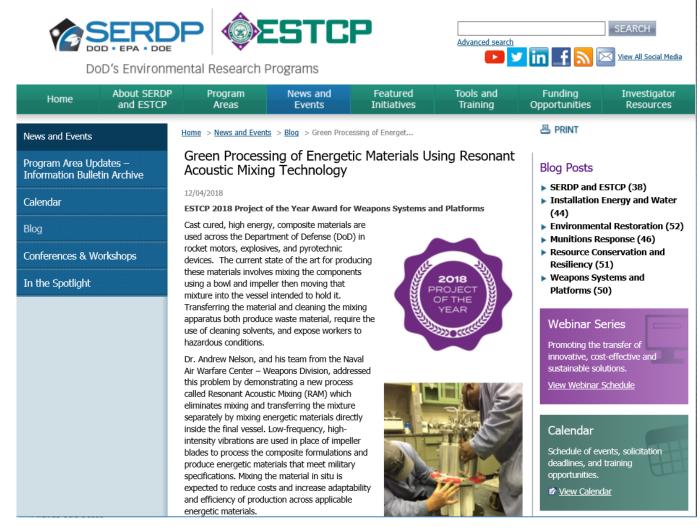


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# https://www.serdp-estcp.org



- The SERDP and ESTCP website can be used to search for individual projects or authors.
- Project overviews related to energetic materials are available at https://www.serdp-

estcp.org/Program-Areas/Weapons-Systems-and-Platforms/Energetic-Materials-and-Munitions

 The SERDP ESTCP Annual Symposium is scheduled to be held in Washington DC 30 November-2 December, 2021