DBX-1, A Potential Drop-In Replacement for Lead Azide

and an Extremely Brief Update on MTX-1, an Alternative to Tetrazene

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Michael Williams
Pacific Scientific EMC., Chandler, AZ USA

Magdy Bichay  Travis Thom
Charles Painter
NSWC-IH, Indian Head, MD USA

Cliff Bedford
ONR, Arlington, VA USA

"Distribution Statement A: Approved for public release, distribution is unlimited, IHDIV 12-058 & ONR 43216-12"
Green Energetics – Background

Lead Styphnate:
Major ingredient in stab and percussion primers, used as ignition element in hot-wire devices
KDNP (4,6-Dinitro-7-hydroxybenzofuroxan, salt) appears suitable as a drop-in replacement & offers high performance
KDNP was approved as safe and suitable for service use and qualified for weapons development in Feb 2009

Lead Azide:
DBX-1 appears suitable as a drop-in replacement and offers advantages over RD1333
DBX-1, under suitable conditions, may be an appropriate substitute for DLA
8020.5C Qualification Testing was completed in 2010 and approval is expected in 2012

Tetrazene:
Explosive high nitrogen material used for sensitization of a variety of priming compositions (mil/com ammunition)
Tetrazene is a material containing no heavy metals but has low hydrolytic and thermal stability
Extremely impact and friction sensitive
PSEMC is currently involved, with ONR, in a project to find a high stability replacement
MTX-1 has sensitivity nearly equivalent to tetrazene with much higher thermal and water stabilities

MTX-1

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**Physical Properties**

- **Physical State:** red, rust solid
- **Particle size:** 10-140 µm
- **Molecular weight:** 355.20 \( (C_2Cu_2N_{10}O_4) \)
- **Density:**
  - X-ray: 2.58 g/cc
  - He pycnometry: 2.59 g/cc
  - Tap density: 1.01 g/cc
- **Oxygen balance:**
  - 0% (to Cu), -9.01 (to Cu₂O), -18.02% (to CuO)
- **Hot Stage Ign Temp:** 1 sec – 356.2 ºC, 5 sec – 350.7 ºC, 10 sec – 345.2 ºC
- **Heat of Explosion:** 3816.6 J g⁻¹ (argon)
- **Heat of Formation:** 280.9 J g⁻¹
- **Critical Temp:** 256-281 ºC; 0.64cm dia. Cyl @ 80% TMD
  - \( E_A = 193.3 \text{ kJ mol}^{-1} \)
- **Thermal Conductivity:** 0.03 W m⁻¹ K⁻¹ powder
- **Vacuum Stability:** 0.47 mL gm⁻¹ 48hr⁻¹ (0.2g, 100 ºC) (<2)
- **VISAR (NASA-JSC):** DBX-1 2.3km/sec; RD1333 2.1km/sec

### Hygroscopicity

<table>
<thead>
<tr>
<th>Hygroscopicity at 25°C</th>
<th>Large Particle (EL3C106A)</th>
<th>Small Particle (EL3O009B)</th>
</tr>
</thead>
<tbody>
<tr>
<td>24 hrs @ 31% RH</td>
<td>0.01%</td>
<td>0.02%</td>
</tr>
<tr>
<td>72 hrs @ 31% RH</td>
<td>0.05%</td>
<td>0.07%</td>
</tr>
<tr>
<td>7 days @ 31% RH</td>
<td>0.07%</td>
<td>0.07%</td>
</tr>
<tr>
<td>24 hrs @ 74% RH</td>
<td>0.03%</td>
<td>0.03%</td>
</tr>
<tr>
<td>72 hrs @ 74% RH</td>
<td>0.03%</td>
<td>0.05%</td>
</tr>
<tr>
<td>7 days @ 74% RH</td>
<td>0.03%</td>
<td>0.06%</td>
</tr>
</tbody>
</table>

X-ray: Dr. D. Parrish at NRL
**DBX-1**

**Safety & Performance Properties**

VLPSC:
ZPP (24mg) was pressed into a header having a 1 ohm 0.0022” stablohm bridgewire at 10 kpsi. Materials were loaded into stainless steel cans* having a 7 mil wall thickness and pressed 5,10,20,40 kpsi. The units were loaded into fixtures and fired (4uf cap, 300V) onto 1” aluminum blocks.

* = considering redesign

Safety:
DSC: TA Instruments MDSC Q2000
Impact: Ball drop instrument/ Bruceton analysis
Friction: Julius-Peters Small BAM
Density: Micromeritics He Pyncnometry
TGA: TA Instruments TGA Q5000
ESD: Low Energy Electrostatic Analyzer

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>DSC (20ºC/minute)</th>
<th>IMPACT (J) (Ball Drop)</th>
<th>FRICTION (Small BAM)</th>
<th>DENSITY (g/cc)</th>
<th>High Res TGA Onset of Wt. Loss</th>
<th>ESD (LEESA)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Onset</td>
<td>Peak</td>
<td>No Fire</td>
<td>Low Fire</td>
<td>TMD</td>
<td></td>
</tr>
<tr>
<td>DBX-1</td>
<td>329ºC</td>
<td>337ºC</td>
<td>0g</td>
<td>10g</td>
<td>2.59 (Cu)</td>
<td>260 ºC</td>
</tr>
<tr>
<td>LA (RD1333)</td>
<td>332ºC</td>
<td>341ºC</td>
<td>0g</td>
<td>10g</td>
<td>4.80 (Pb)</td>
<td>166 ºC</td>
</tr>
</tbody>
</table>

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DBX-1 slowly dissolves and decomposes to 5-nitrotetrazolate when put in direct contact with water. Observed by ultraviolet absorption spectroscopy at 256nm. Pronounced for small particle DBX-1 samples. The residual undissolved solids were determined to be unaffected DBX-1 as demonstrated by FTIR and DSC.

Increased 2-propanol content suppresses the decomposition of DBX-1 with neat 2-propanol having no reactive effect. Currently revisiting with long duration testing & alternate solvents – IPA: 2 months, no change.
DBX-1 has demonstrated compatibility with:

<table>
<thead>
<tr>
<th>Metals (coupon test)</th>
<th>Metals (DSC)</th>
<th>Energetics</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>1100 aluminum</td>
<td>Tophet A</td>
<td>PETN</td>
<td>PEEK</td>
</tr>
<tr>
<td>6061-T6 aluminum</td>
<td>(StableOhm 650)</td>
<td>RDX</td>
<td>Mylar</td>
</tr>
<tr>
<td>2029-T3 aluminum</td>
<td>Tophet C</td>
<td>HMX</td>
<td>Mica</td>
</tr>
<tr>
<td>304 stainless steel</td>
<td>(Nichrome)</td>
<td>CL20</td>
<td>Isomica</td>
</tr>
<tr>
<td>Brass</td>
<td>EvenOhm</td>
<td>HNS (various)</td>
<td>Boron nitride</td>
</tr>
<tr>
<td>MK107 gilding metal</td>
<td>(StableOhm 800)</td>
<td>PYX</td>
<td>Stycast</td>
</tr>
<tr>
<td>(95% Cu, 5% Zn)</td>
<td></td>
<td>BI770</td>
<td>Hysol 2216</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ZPP</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>NOL-130</td>
<td></td>
</tr>
</tbody>
</table>

Housings | Bridgewires | Powder | et. al.

No incompatibility with any materials tested

LA is not compatible with some of the above metals/secondaries.
Preparation of DBX-1 from Cu(II)

Initial process from Cu(I):
~ 1 hour process with induction period

New process:
Low induction period for crystallization
Reaction time 10-15 minutes
Yield: 80-90%
Prep > 100 X

This process is suitable for scale-up
and is used in the current ManTech Program

Normal analysis for DBX1 indicates this material made
by this method is as good or better than previous

Particle size tends to be slightly smaller

Full Paper
# Process Development & Scale-Up of DBX-1

## Goals & Objectives

The goal of this initiative is to develop an optimized and reproducible method for production of the lead azide replacement material DBX-1. Particular attention will be given to DBX-1 particle size and morphology to insure that the final product is suitable for handling and loading in existing tooling. An objective is to scale production of this optimized DBX-1 to the 100 gram level and provide this material for testing in military hardware and devices.

## Initiative Information

- **Initiative Lead:** Michael Williams - PSEMC
- **Team Members:**
  - John Fronabarger
  - Jonathon Bragg
  - Stan Hartman
  - Lisa Dagostini – all of PSEMC
  - Nalas Engineering Services
- **Period of Performance:** 3Nov2010-29Nov2012

## Milestones & Technical Achievements

<table>
<thead>
<tr>
<th>Date</th>
<th>Event Description</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nov10</td>
<td>Kickoff Meeting - C</td>
<td>C</td>
</tr>
<tr>
<td>Jul11</td>
<td>Chemical Process Optimization - C</td>
<td>C</td>
</tr>
<tr>
<td>Aug11</td>
<td>NaNT (reactant) Evaluation - C DBX-1 lot Characterization - C</td>
<td>C</td>
</tr>
<tr>
<td>Sept11</td>
<td>100gm DBX-1 Process Validation - C</td>
<td>C</td>
</tr>
<tr>
<td>Apr12</td>
<td>DBX-1 Disposal Study - C</td>
<td>C</td>
</tr>
<tr>
<td>Oct12</td>
<td>100gm DBX-1 Process Review - S</td>
<td>C</td>
</tr>
</tbody>
</table>

**IP - in process, C- completed, S - scheduled**

## Implementation & Payoff

- **Schedule:** 31 October 2012.
- **Status:** On track

The initiatives successful completion will afford a reproducible scale-up process for DBX-1 (copper(I) 5-nitrotetrazolate). DBX-1 is a lead-free (green) replacement for the lead azide component of a variety of DoD devices.
Summary of Reaction Variables

variations from “standard”
yield, purity, particle size & distribution

• **pH** - pH should not be appreciably above 3.0 and preferably untreated (8, 0.99-5.00)

• **Concentration** - overall reaction concentration has little effect (6, 94-194 mL)
  high concentrations affords a larger, bimodal product

• **Counter-ion** - anions other than chloride result in smaller and multimodal particles (6, various)
  acetate and nitrate counter-ions afford products with lower DSC exotherms

• **Addition Rates** - ascorbate addition rates have little effect (5, 0.25 ml/min-10.0 mL/min)
  faster addition rates may increase DBX-1 particle size, narrow the particle distribution
  very fast addition provides larger particle/lower DSC

• **NaNT Stoichiometry** - lower NaNT/Cu(II) should be avoided – stoichiometric 5% (5, 0.84 eq. -1.12 eq.)

• **Ascorbate Stoichiometry** - initial 2 electron reduction occurs quickly while an additional 2 electron reduction
  of the didehydroascorbic acid (initial) oxidation product occurs more slowly
  and provides additional DBX-1. Changing stoi./rxn time gives larger size distribution

• **Alternate Reducing Agent** – replacement of ascorbate with α-D-glucose does not reduce Cu(II) to Cu(I)
Modified DBX-1 Process.

<table>
<thead>
<tr>
<th>Lot #</th>
<th>mL eq. Lot# NaNT</th>
<th>Add’n Rate Na Asc Pre/Post Hold</th>
<th>Add’n Hold (min)</th>
<th>Counterion</th>
<th>Water (mL) Total Volume</th>
<th>Yield* (gms/%) DSC exo†</th>
<th>Visual Observations (Particle size)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3Y114A</td>
<td></td>
<td>Identical</td>
<td></td>
<td>Cu(II)Cl₂</td>
<td>60mL 110mL</td>
<td>2.45/86% 336.1°C</td>
<td>Normal 111µm uniform</td>
</tr>
<tr>
<td>3Y119A</td>
<td></td>
<td>Total time ~ 35 minutes</td>
<td></td>
<td>Cu(II)Cl₂</td>
<td>60mL 110mL</td>
<td>2.37/83% 336.4°C</td>
<td>Normal 111µm uniform</td>
</tr>
</tbody>
</table>

Final Lab Scale Process

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Remote Control Operation
- Reactors – 1, 3, 20L capability
- Reaction –
  Addition and Reaction
  Filtration and Washing
  Dispensing and Weighing
- Computer controlled
- Heating and Cooling
- Remotely monitored

PSEMC/Franklin Engineering Scale-up Reactor

Currently used for large scale synthesis of:
DBX-1 – to 100+g lots
Silver azide – to 100g lots
## Preparation of DBX-1 – Initial Scale-up (Reactor)

<table>
<thead>
<tr>
<th>Lot #</th>
<th>Total Volume</th>
<th>Yield (gms/%) DSC exo</th>
<th>Particle Size</th>
<th>Additional Information</th>
</tr>
</thead>
<tbody>
<tr>
<td>3U085 (10g)</td>
<td>808mL</td>
<td>7.5gms/66% 337°C</td>
<td>95 um</td>
<td>&gt;1 eq. NaNT, various concentration, ascorbate addition rates varied, 550-600 RPM, granulation times: 9-17min.</td>
</tr>
<tr>
<td>3U087 (12g)</td>
<td>1212mL</td>
<td>11gms/65% 336°C</td>
<td>113 um</td>
<td></td>
</tr>
<tr>
<td>3U089 (25g)</td>
<td>1555mL</td>
<td>20gms/70% 336°C</td>
<td>104 um</td>
<td></td>
</tr>
<tr>
<td>3U113 (10g)</td>
<td>795mL</td>
<td>5.4gms/60% 336°C</td>
<td>97um</td>
<td></td>
</tr>
<tr>
<td>3U121 (25g)</td>
<td>1808mL</td>
<td>25gms/81% 336°C</td>
<td>95um</td>
<td></td>
</tr>
</tbody>
</table>

MicroTrac S3500 light scattering analyzer
Adding larger quantities of the reducing agent at the rate (or time) used in the lab scale is clearly untenable. During 25 gm scale-up experiments determined rate should be based on the amount of copper present in the reaction. Curve shows the rates for a variety of scales, extrapolation provided suitable addition rates at the 25/100 gm scale.
### Preparation of DBX-1 – 25gm Scale-up

<table>
<thead>
<tr>
<th>Lot #</th>
<th>Water (mL)</th>
<th>Yield* (gms/%) DSC exo †</th>
<th>Particle Size (mean)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4C061</td>
<td>1132 mL, 1742 mL</td>
<td>27.2/76% 336°C</td>
<td>140 µm</td>
</tr>
<tr>
<td>4C063*</td>
<td>1132 mL, 1700 mL</td>
<td>26.9/75% 336°C</td>
<td>110 µm</td>
</tr>
<tr>
<td>4C065*</td>
<td>1132 mL, 1700 mL</td>
<td>27.3/76% 336°C</td>
<td>117 µm</td>
</tr>
<tr>
<td>4C067</td>
<td>1382 mL, 1950 mL</td>
<td>23.3/65% 337°C</td>
<td>93 µm</td>
</tr>
<tr>
<td>4C069</td>
<td>1132 mL, 1700 mL</td>
<td>23.0/64% 337°C</td>
<td>116 µm</td>
</tr>
<tr>
<td>4C071</td>
<td>1382 mL, 1950 mL</td>
<td>26.3/74% 336°C</td>
<td>109 µm</td>
</tr>
<tr>
<td>4C073†</td>
<td>1286 mL, 1700 mL</td>
<td>18.5/71% 337°C</td>
<td>99 µm</td>
</tr>
<tr>
<td>4C075†</td>
<td>1286 mL, 1700 mL</td>
<td>16.5/63% 336°C</td>
<td>130 µm</td>
</tr>
</tbody>
</table>

~1 eq. NaNT, various concentration ascorbate addition rates varied 400-640 RPM granulation times: 12-18 min.

4C063 and 4C065 were run under identical conditions to confirm process reproducibility.

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SEM & Photomicrographs of 25gm Batches

EL4C067 – 94 µm

EL4C069 – 112 µm

EL4C071 – 109 µm

EL4C067 – 94 µm

EL4C069 – 112 µm

EL4C071 – 109 µm
Consistent Preparations of DBX-1 Lots at 25gm Level

DBX-1 25 Gram Batch Particle Size

Characteristic Bimodal Distribution

DSC (Peaks) – 336 °C
Based on reproducibility, continue at 100g scale

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Preparation of DBX-1 – 100gm Scale-up (20L)

<table>
<thead>
<tr>
<th>Lot #</th>
<th>Water (mL)</th>
<th>Yield gms/% DSC exo</th>
<th>Particle Size (mean)</th>
<th>Sur. Area (M²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Total Volume (mL)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4C077</td>
<td>8901 mL</td>
<td>95.5/58%</td>
<td>336°C</td>
<td>98 µm</td>
</tr>
<tr>
<td>100 gm</td>
<td>11571 mL</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4C079</td>
<td>9325 mL</td>
<td>113/74%</td>
<td>336°C</td>
<td>101 µm</td>
</tr>
<tr>
<td>100 gm</td>
<td>11690 mL</td>
<td></td>
<td></td>
<td>0.39</td>
</tr>
<tr>
<td>4C081</td>
<td>9324 mL</td>
<td>118/77%</td>
<td>336°C</td>
<td>70 µm</td>
</tr>
<tr>
<td>100 gm</td>
<td>11691 mL</td>
<td></td>
<td></td>
<td>0.36</td>
</tr>
<tr>
<td>4C083</td>
<td>8594 mL</td>
<td>147/78%</td>
<td>336°C</td>
<td>104 µm</td>
</tr>
<tr>
<td>100 gm</td>
<td>11690 mL</td>
<td></td>
<td></td>
<td>0.35</td>
</tr>
<tr>
<td>4C085</td>
<td>8480 mL</td>
<td>129/68%</td>
<td>336°C</td>
<td>87 µm</td>
</tr>
<tr>
<td>100 gm</td>
<td>11639 mL</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Sept-Oct 2011

~1 eq. NaNT, various concentration
ascorbate addition rates varied
340-640 RPM
granulation times: 24-35 min.

stirring rate, NaNT concentration, total volume modified to assess the effect on particle size with results as shown

100 gm scale takes longer than 25 gm scale due to the additional heating time required to get the mixture to 90°C.
Initial EL4C077 had an extended time to granulation minutes in un-seasoned reactor, subsequent reactions were seeded & had normal time to granulation of ~25 minutes.
Overall reaction time (or amount of time to run the DBX-1 synthesis once the reactants are at temperature) is less than 1 hour start to filter, even at the 100 gm scale

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SEM & Photomicrographs of 100gm Batches

EL4C077 – 98 µm (77X)  EL4C079 – 101 µm (77X)  EL4C081 – 70 µm (77X)

EL4C077 – 98 µm (500X)  EL4C079 – 101 µm (500X)  EL4C081 – 70 µm (500X)
Particle size range of interest (60-140µm) versus stirring speed. 
Line color shows effect of overall NaNT concentration on particle size.

Either of these variables alone or in conjunction will afford control over particle size of the DBX-1 produced.
Particle Size/Distribution of 100gm Batches

Moving from a bimodal distribution (light blue & light green) to a “normal” distribution. This gives lower average particle size.
DBX-1 Characterization

Heat Flow (W/g) vs Temperature (°C)

- EL4C085
- EL4C077
- EL4C079
- EL4C081
- EL4C083

Temperature range: 100 to 400 °C

Exothermic (Exo Up) and endothermic reactions observed.
Consistency of Process at 100g Level

Demonstrates control over particle size and distribution
Demilitarization/Decomposition Studies on DBX-1

PSEMC has been assessing a variety of methods for disposal of DBX-1 utilizing a number of procedures.

**NaNT:**
Treatment of DBX-1 with 10% sodium hydroxide decomposes the DBX-1 into an inert copper containing solid (oxide) and the sodium 5-nitrotetrazolate (NaNT) starting material which may be recovered.

**Decomposition to inert materials:**
Treatment of DBX-1 with 5N HCl and (inexpensive) granular Zn metal further reduces the NaNT to inert 5-aminotetrazole and successfully reduces any of the azotetrazole formed during the process (to 5-AT)

Currently getting > 1:1000 NaNT:5-AT with NO azotetrazole, works under cold & hot conditions

**Dissolving DBX-1:**
DBX-1 is very insoluble under most conditions.
Treatment with aqueous sodium thiosulfate reacts with the Cu(I) and leaves the NT in solution. (photographic fixer, binds soft metals)
May suggest a recrystallization method

Primary analysis in solution by Raman spectroscopy
Secondary analyses HPLC, ion chromatography, FTIR and DSC.

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PSEMC has completed Compound Qualification Testing per NAVSEAINST 8020.5C – (J. Laib input) DBX-1 data pack forwarded to NSWC-IH for submission to NAVSEA Systems Command for Military qualification

Current Program Complete Nov12

Performance testing on 100g level samples
Safety
Output
  Strong confinement
  202 Detonator
Additional compatibility studies
Additional test at NSWC-IH & elsewhere
Final Specification (Preliminary Specification complete)
Current Studies using DBX-1

Completed Testing:
Prepare PSEM C 104477-202 detonators with LA (DLA) and DBX-1 transfer charges for comparison
successful: DBX-1 functions faster and with a greater output “dent” at -65 F, 200 F and ambient
Prepare NOL-130 primer mix with both DLA and DBX-1 and perform side by side safety tests
Alex Woods, NSWC-IH – successful: replaced RD1333 in Mk125-1 Stab primer in both NOL-130 & output
NOL-130 to DBX-130: > output; RD1333 to DBX (same volume): > output
Sensitivity data for NOL-130 vs. DBX-130 (impact, friction, etc.)

Preliminary Tests with the 2-Piece Stab Detonator
Stu Olson, Stresau – successfully utilized DBX-1 as a “drop-in” replacement for RD-1333
same loading pressure, same height, NOL-130 unchanged – 89 dets from 2 lots, 88 normal

Current Testing:
PSEM C – Chandler - JL42 Firex Cartridge (~120g)
PSEM C – Hollister - ZY56 Drogue Severance Assembly (~60g)
Stresau – 2-Piece Det. then to General Dynamics - Army (~100g)
NSWC-IH – J. Laib for additional qualification tests (~95g)
Action Manufacturing – M100 Detonators – S. Marino
ARDEC – M55 Detonators – N. Mehta, G. Cheng
Army Institute of Public Health – Toxicity – William Eck

Nalas Engineering – synthesis, seeding studies, NaNT
NSWC-IH – synthesis (T. Ricks)
ARDEC – synthesis (N. Mehta, K. Oyler)
The PSEMC/I2Chem/MIT NaNT microreactor system continuously converts 5-AT to NaNT which is the starting material for DBX-1 and BNCP.

**Benefits over current batch process:**

- **Safety:** minute quantities of unstable intermediates, safe aqueous solvent system
- **Quality:** fast heat and mass transfer rates means no lot variability
- **Efficiency:** computer monitored and autonomous

I2Chem delivered 2 systems to PSEMC

Expect production of NaNT from 1 reactor line to be ~20 gms/hr minimum continuous

Scale-out (add more reactor lines) to increase capability to meet needs

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Acknowledgments

- Jeff Dehart, Tracy Smith - PSEMC
- Dr. Bill Sanborn - PSEMC
- Dave Grum, Diane Ross, Paul Garber - PSEMC
- Dr. Alfred Stern - NSWC-IH
- Dr. Brad Sleadd - NSWC-IH
- Dr. Pete Ostrowski - Energetic Materials Technology
- Mike Sitzmann - NSWC-IH (Ret.)
- Gerald Laib - NSWC-IH
- John Hirlinger – ARDEC/Picatinny
- Alex Schuman - NSWC-IH
- Frank Valenta - NSWC-IH (Ret.)
- Dr. Robert Chapman - NAWC-CL
- Dr. Farhad Forohar - NSWC-IH
- Dr. Phil Pagoria - LLNL
- Dr. Mike Hiskey
- Dr. Jeff Bottaro
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