DEVELOPMENT OF A CONUS MANUFACTURING CAPABILITY FOR FOX-7


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Funding provided by Joint Insensitive Munitions Technology Program (JIMTP)

- Develop a Continental United States (CONUS) manufacturing capability for FOX-7

- Demonstrate the capability to produce all 4 classes of FOX-7 currently available from EURENCO Bofors with equivalent purity/quality

EURENCO offers 4 different classes of DADNE (FOX-7) with different crystal sizes:
- Class 1: 20 – 40 μm
- Class 2: 50 – 100 μm
- Class 3: 100 – 200 μm
- Class 4: 250 – 300 μm
EURENCO Bofors FOX-7

- 1,1-Diamino-2,2-dinitroethene (FOX-7) is an energetic material developed by FOI Sweden in the late 1990s as an insensitive RDX replacement

- Technology was then transferred to NEXPLO Bofors AB (now EURENCO Bofors AB) for pilot/production scale manufacture

![2,2-dinitroethene-1,1-diamine](image)

2,2-dinitroethene-1,1-diamine

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Yellow crystals</td>
</tr>
<tr>
<td>Drop weight sensitivity</td>
<td>20-40 J (RDX 4-5J)</td>
</tr>
<tr>
<td>Friction sensitivity</td>
<td>&gt; 350 N (RDX 120 N)</td>
</tr>
<tr>
<td>Small Scale Gap Test at 1.63 g/ml</td>
<td>6.22 mm (RDX 9.33 mm, HMX 10.3 mm, TNT 6.4 mm)</td>
</tr>
<tr>
<td>ESD</td>
<td>&gt; 8 J (HMX 0.2 J)</td>
</tr>
<tr>
<td>Detonation velocity</td>
<td>8800 m/s</td>
</tr>
<tr>
<td>Density</td>
<td>1.885 (crystal)</td>
</tr>
<tr>
<td>Purity HPLC</td>
<td>&gt; 99%</td>
</tr>
<tr>
<td>Vacuum stability</td>
<td>0.1 – 0.4 ml/g,h at 120°C</td>
</tr>
<tr>
<td>Measured detonation pressure</td>
<td>34 GPa (RDX 35 GPa)</td>
</tr>
<tr>
<td>Appearance</td>
<td>Yellow crystals</td>
</tr>
</tbody>
</table>
Mixed acid nitration conducted at 10-30°C

- “b” is insoluble in mixed acid

Resultant slurry was poured into ice water

Delayed foaming (dinitromethane decomposition) and NOx generation

For larger scales, slurry will be dose-quenched into warm/hot water
Scale Up Protocol

Round-bottomed flask or MT EasyMax

- 5 gram theoretical yield
  - Small scale safety data
    - Impact, friction, ESD, DSC
  - Reproducibility & yield

- 20 gram theoretical yield
  - Small scale safety data
    - Impact, friction, ESD, DSC
  - Reproducibility & yield
  - Recrystallization
    - SEM & PSD
Selected DSC traces of NSWC IHEODTD FOX-7: 5g recrystallized batches. A) FOX-7 recrystallized from hot water at IHEODTD. B) FOX-7 recrystallized from NMP/water at IHEODTD.
Mettler Toledo RC1e Reaction Calorimeter

- 100 gram theoretical yield
- Small scale safety data
  - Impact, friction, ESD, DSC
- Reproducibility & yield
- Recrystallization
  - SEM & PSD
- Heat flow calorimetry
Scale Up: Nitration

1 kg theoretical yield

Solids mass increase x 2.4
Scale Up: Nitration & Quench

Solids mass decrease x 2.9
Scale Up: Quench

Nitration mixture is pumped into warm water

Yields typically ~70%
Scale Up: Nitration

2 kg theoretical yield

Solids mass increase x 2.4!
Scale Up: Nitration

Subsequent batches were limited to 1.5 kg
Second “upper” impeller was installed on agitator shaft
Recrystallization

2 L Scale; RC1

All four classes were obtained

At the 2 L scale
Recrystallization

2 kg scale

Recovery typically >80%
Recrystallization parameters did not scale!
Conditions that worked at the 2 L scale
Did not work at the 50 L scale (not surprising)

Class I: 20-40 µm

EURENCO Bofors Class I

CONUS Class I (bad solvent)
Recrystallization
CONUS FOX-7

Class I; too large!

Class IV; too large!
Recrystallization
CONUS FOX-7

Class I

Class IV

27 µm

279 µm
Safety and Thermal Analysis
CONUS Round-Robin Testing

- **Machine and Method**
  - **ERL Impact:**
    - Type 12 Tooling
    - Mass of drop weight: 2.5kg
    - Mass of Striker: 520-540gm
    - 180A Garnet Paper
  - **ESD:** SMS Equipment (ARDEC CL), ABL (IH)
    - Verify Ohm value of in-line resistor
    - Needle distance-position approximate 0.0020”
      - Hold voltage at 5.785Kv
    - Run using IH intervals – start at 0.326joules
  - **ABL Friction:** SMS Equipment
    - Wheels and Plates – verify hardness finish use hardened steel \( \approx \) 60microinch
    - Pressure Gauge Calibration – verify calibration is valid
  - **BAM Friction:**
    - Wheels and Plates – verify hardness finish (new wheels and plates)
    - Pressure Gauge Calibration – verify calibration is valid

- **Methods Stipulation:**
  1. All test masses will be 35mgs \( \pm \) 2
  2. All tests to proceed with ‘lights off’ for observational effect
  3. Testing to proceed at 45-55% relative humidity or as close to it as possible - note Hr and T
  4. DSC to be run at 5°C/min per ASTM E3537
  5. All sites use the older hemetic aluminum pans, closed pan vented

- **Data Analysis**
  - Provide both Bruceton 50% and 20 TIL
  - Provide description of reaction (sparks, pop, fire, consumption, smell) in comments section
## Safety and Thermal Analysis

### CONUS Round-Robin Testing

<table>
<thead>
<tr>
<th>Site</th>
<th>Sample Lot# Class</th>
<th>Impact (cm)</th>
<th>Friction</th>
<th>ESD (joules)</th>
<th>DSC (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Indian Head</strong></td>
<td>IHM170FX7-076 I</td>
<td>*55 51 32</td>
<td>708 (50%)</td>
<td>10/10 NF 216</td>
<td>TBD 0.095 0.037</td>
</tr>
<tr>
<td></td>
<td>IHM17FFX7-104 IV</td>
<td>*46 41 26</td>
<td>20/20 NF 1000</td>
<td>10/10 NF 216</td>
<td>TBD 0.095 0.037</td>
</tr>
<tr>
<td><strong>Picatinny</strong></td>
<td>IHM170FX7-076 I</td>
<td>**79 79 63</td>
<td>20/20 NF @ 1800</td>
<td>10/10 NF 324</td>
<td>TBD 0.095 0.037</td>
</tr>
<tr>
<td></td>
<td>IHM17FFX7-104 IV</td>
<td>*71 63 32</td>
<td>20/20 NF @ 1800</td>
<td>10/10 NF 360</td>
<td>TBD 0.095 0.037</td>
</tr>
<tr>
<td><strong>China Lake</strong></td>
<td>IHM170FX7-076 I</td>
<td>**71 63 50</td>
<td>20/20 NF @ 1000</td>
<td>10/10 NF 360</td>
<td>8.37 3.80 1.50</td>
</tr>
<tr>
<td></td>
<td>IHM17FFX7-104 IV</td>
<td>**51 40 32</td>
<td>20/20 NF @ 1000</td>
<td>10/10 NF 360</td>
<td><strong>20/20 NF @ 8.0</strong></td>
</tr>
</tbody>
</table>

*50% point via Bruceton Method  
** 50% point determined by Probit or Modified Bruceton Method

### Analysis:
- ERL Impact – IH values tend to trend lower, CL and ARDEC fairly similar
- ABL Friction – only IH Class I was observed to be lower than other sites
- BAM Friction – IH observations were a couple of logs lower, CL/ARDEC nearly the same
- ESD – IH and ARDEC same results, CL significantly different in observations (magnitudes)???

> ESD differences are likely due to the ‘interpretation’ of the description of a fire found in AOP-7 edition 2, not the method or machinery. Under NAWC method a fire consists of ‘flash, spark, burn, odor, or noise other than instrument noise’
# Safety and Thermal Analysis

**OCONUS vs. CONUS**

<table>
<thead>
<tr>
<th>Test</th>
<th>Class I</th>
<th>Class IV</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>OCONUS</td>
<td>CONUS</td>
</tr>
<tr>
<td>ERL Impact (cm) (50%/LF/TiL)</td>
<td>60/40/20</td>
<td>71/63/50</td>
</tr>
<tr>
<td>ABL Friction (lbf)</td>
<td>20/20 NF 1000</td>
<td>20/20 NF 1000</td>
</tr>
<tr>
<td>BAM Friction (N)</td>
<td>10/10 NF 360</td>
<td>10/10 NF 360</td>
</tr>
<tr>
<td>*ESD ( joules) (50%/LF/TiL)</td>
<td>&gt; 8.0/3.80/1.50</td>
<td>8.37/3.80/1.50</td>
</tr>
<tr>
<td>DSC (°C)</td>
<td>228, 286</td>
<td>231, 289</td>
</tr>
</tbody>
</table>

### Analysis:
- **ERL Impact** – Recent CONUS values appear to be slightly higher than the ‘as received’ OCONUS material but follow the general trend of Class I less sensitive to impact than the larger Class IV material – in general fairly good correlation.
- **ABL Friction** – Both CONUS and OCONUS tested out at the same level with no friction sensitivity up to 1000 pound of applied force.
- **BAM Friction** – All but the OCONUS Class IV tested out to no initiation (fires) up to 360 newtons of force.
- **ESD** – All materials tested to no ignition to spark at 0.25 joules of energy
  
  * The ESD testing used the NAWC method and description called out in AOP-7 edition 2. As noted prior site differences are likely due to the ‘interpretation’ of the description of a fire per the NAWC method.
Conclusions

A CONUS Manufacturing Capability for FOX-7 has been developed

Need to optimize nitration for larger scales

- Eliminate “yogurt” formation

Recrystallization to match EURENCO classes eventually successful

- Trial and error for conditions when changing scale and equipment

ROM cost estimate at this scale is inadvisable

- Economy of scale not yet realized for MDHP
- Economy of scale not yet realized for nitration/quench
- Economy of scale not yet realized for recrystallization
Acknowledgments

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- M24; Chemical Scale-up Group;
- Scientists, engineers and analysts at ARDEC, NAWC WD and NSWC IHEODTD for Round Robin testing
QUESTIONS?