This report documents demonstration testing of MIC primer compositions designed to eliminate lead from the primers used in small arms ammunition and cartridge actuated devices. This effort has shown that an Al/Bi$_2$O$_3$ composition can be successfully mixed and wet-loaded into existing US Army and US Navy primer hardware. Subsequent testing in M855 5.56 mm ammunition as well as a variety of Navy impulse and delay cartridges has shown that the primer provides performance essentially equivalent to the lead-based compositions presently in use. Thus, the new primer composition meets the objective for a drop-in replacement for the lead compounds. While the test results are positive and the MIC primer meets all performance specifications, additional work is needed to refine the primer composition to achieve faster action time in small arms cartridges and to eliminate occasional misfires.

**Introduction**

1.1 Background

Current percussion primers in small and medium caliber ammunition (i.e. 5.56mm-40mm) use a lead styphnate based primer formulations that pose a long term hazard to the environment and the operator of the weapon since airborne vaporized lead results from each successfully fired cartridge. Lead styphnate based primer compositions are currently specified in all of the US Army’s combat small caliber ammunition and in many Cartridge Actuated Devices and Propellant Actuated Devices (CAD/PAD) used in US Navy aircraft ejection systems, countermeasure applications, and stores release systems. The CAD/PAD devices are used by all DOD components and foreign military that utilize US manufactured aircraft. Lead is a known toxic material, which pollutes test ranges and exposes the manufacturers and users of these devices to serious health hazards liabilities. Lead is regulated by the Environmental Protection Agency (EPA) and the Occupational Safety Health Administration (OSHA). Current EPA and OSHA regulations are directly impacting range and testing operations. Stricter regulations in the future will seriously impact or force closing of production, testing and range operations. With the current production rate for all small caliber ammunition (less than 20 mm), the quantity of lead to be consumed for percussion primer production alone is well over 23,686 pounds or nearly 12 tons annually.

Small caliber percussion primers generally consist of a brass cup loaded with the charge composition and a brass anvil pressed into the charge. Figure 1 shows the US Army No. 41 primer prior to inserting the anvil. When the cup is struck by a firing pin, the friction and impact sensitive charge is crushed between the bottom of the cup and anvil, causing
ignition. The hot ignition products flow out of the cup around the legs of the anvil to ignite the next element in the ignition train of the weapon system. For the Army No. 41 primer, this would be the ball powder main charge in small caliber ammunition. For the Navy PVU-1/A primer, this would be either a transition charge or an output charge, depending on the particular application.

![Figure 1 – No. 41 Small Caliber Percussion Primer Showing Loaded Cup and Tripod Anvil](image)

While the No. 41 and PVU-1/A primers are similar in size, there are some important differences. The No. 41 primer is designed to provide sufficient pressure to quickly ignite the double base ball powder main charge, and thus utilizes a tripod anvil and the FA-956 primer composition, which contains PETN and aluminum powder for added brisance (Table 1(a)). The PVU-1/A is designed for “soft” ignition of delay cartridges, and uses a bipod anvil and the 5086 primer composition (Table 1(b)), which provides a lower output pressure than FA-956. Figure 2 is a photograph of a loaded PVU-1/A, illustrating the small size.

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Weight %</th>
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<tbody>
<tr>
<td>Normal Lead Styphnate</td>
<td>37.0</td>
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<tr>
<td>Tetracene</td>
<td>4.0</td>
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<tr>
<td>Barium Nitrate</td>
<td>32.0</td>
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<tr>
<td>Antimony Sulfide</td>
<td>15.0</td>
</tr>
<tr>
<td>Aluminum Powder</td>
<td>7.0</td>
</tr>
<tr>
<td>PETN</td>
<td>5.0</td>
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</table>

(a) FA-956 Primer Mix (No. 41 Primer) Primer

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Weight %</th>
</tr>
</thead>
<tbody>
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<td>Normal Lead Styphnate</td>
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<tr>
<td>Barium Nitrate</td>
<td>41.5</td>
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<tr>
<td>Tetracene</td>
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<tr>
<td>Calcium Silicide (Treated)</td>
<td>10.5</td>
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<tr>
<td>Antimony Sulfide</td>
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</tbody>
</table>

(b) 5086 Primer Mix (PVU-1/A)

| Table 1. Primer Mix Compositions for No. 41 and PVU-1/A Primers |
Metastable Intermolecular Composite (MIC) material has the potential to replace the current conventional energetic composition in the initiation subcomponents of ammunition and cartridge actuated devices known as the percussion primer. The novel properties associated with nanostructure materials have resulted in the development of thermite-like formulations of energetic materials by Los Alamos National Laboratory (LANL). These materials being of nano-sized particles offer the possibility of tunable energy release and high temperatures without appreciable gas generation and attendant high pressures. There are various examples of MIC applications that attracted a great deal of interest recently for weapon enhancement. One unique feature of MIC materials is its ability to produce particles hot enough to ignite a bed of propellant. Additionally, the MIC materials are impact sensitive which makes them a good percussion primer mix candidate. MIC can be utilized as an initiation composition for replacing the current established FA-956 and 5086 primer formulations which are based on lead styphnate, barium nitrate and antimony sulfide. The MIC mixture is an environmentally friendly, lead free composition.

In general terms, the MIC material is an engineered energetic composition consisting of a metal fuel (most often nano-scale aluminum) and metallic oxidizer that are exothermically reactive with each other. By utilizing nano-sized particles, the near atomic scale proximity of the reactants minimizes distances over which the fuel and oxidizer molecules must diffuse in order to reach each other, resulting in a dramatically increased reaction rate relative to that of conventionally sized mixtures. Two of the most commonly used MIC compositions have the following chemical reactions:
For Army small caliber ammunition applications, the MIC primer must meet #41 primer all-fire and no-fire energy requirements and also ignite the propelling charge rapidly enough to meet the action time requirement for each individual cartridge application.

For CAD/PAD applications, the MIC primer must meet the PVU-1/A primer all-fire and no-fire energy specifications and also must function such that the performance requirements for each individual application are met. Because the applications chosen for the demonstration represent a cross-section of the CAD/PAD spectrum, the performance requirements vary considerably from one application to another.

The current primer mix for all 5.56mm 7.62mm and 0.50 cal ammunition is the Frankford Arsenal composition FA 956. 20mm ammunition uses a primer mixture of slightly different amounts of the same basic components plus a carbon compound to make the mixture electrically conductive. These primer mixtures are manufactured by Lake City Army Ammunition Plant (LCAAP). The manufacturing process used in producing the FA 956 primer mix is basically a five step process that includes the manufacture of trinitroresorcinol (TNR), lead styphnate, tetracene, pentaerythritol tetranitrate (PETN), wash, and a final wet mix operation.

- Lead styphnate is formed by mixing TNR with magnesium oxide to form magnesium trinitroresorcinol. The magnesium trinitroresorcinol is in turn mixed with lead nitrate to form lead styphnate.

- Other heavy metals chemical compositions besides lead that are added during the final mixing process of producing the FA 956 primer mix are antimony sulfide and barium nitrate.
The following flow chart summarizes the current manufacturing process for the FA 956 formulation:

- **AMINE GUANIDINE BICARBONATE**
- **TETRACENE**
- **SODIUM NITRITE**
- **LEAD NITRATE**
- **NORMAL LEAD STYPHNA TE**
- **MAGNESIUM TRINITRORESORCINOL**
- **PETN**
- **GUM ARABIC SOLUTION**

The wet-mixing procedure consists of the following steps:

Lead styphnate is placed into the mixer. Tetracene is then added followed by the gum Arabic solution and the PETN. The dry fuels (antimony sulfide and aluminum) are added to the mixture followed by the oxidizer (barium nitrate). The finished wet primer mix is transferred to a conductive container and transported to the primer pelleting. These are labor intensive processes.

Pellets are made by hand-pressing the wet primer mix onto a plate with holes that correspond to the number of primer cups that are to be filled. The pellets are then transferred to primer cups and the mix is consolidated. The anvils are then inserted into the consolidated mix and the finished primer is moved to a drying area for removal of water. After a period of time suitable to ensure the mixture is dry, the primers are tested for sensitivity and taken to the bullet assembly line to be inserted into cartridge cases.

The PVU-1/A primer has been designed for use in aircrew escape systems for Army, Navy, and Air Force aircraft. Delay cartridges are used extensively in these systems, and because delay columns cannot tolerate high impact forces, it is necessary to use a primer mix that is less brisant than the FA 956 used in the #41 primer. In addition, to attain the high reliability required for man-rated systems, the PVU-1/A hardware has been designed.
for increased sensitivity to friction and impact. Thus, the output pressure generated by the PVU-1/A is roughly 25% of that for the #41, and the all-fire energy is considerably less (25.5 inch-ounces versus about 46 inch-ounces for the #41). These differences between the PVU-1/A and #41 primers highlight the difficulty in creating a common mixture which can be used in both.

The process for manufacturing the 5086 mix is specified in the PVU-1/A drawing package (NAVAIR 851AS110). The process is similar to that used for the FA 956, and is briefly summarized below:

- Inert ingredients (barium nitrate, calcium silicide, and antimony sulfide) are dried and sieved.
- Lead styphnate and tetracene are weighed out to the correct proportions for the mix, and then re-wetted with ethanol.
- Mixing speed is adjusted, the inert ingredients are added, and mixed.
- The lead styphnate and tetracene are added separately and mixed.
- The wet mix is stored in polyethylene bottles for later use.
- Prior to loading, the wet mix is dried and then sieved.

PVU-1/A primers are hand-loaded in manufacturing facilities using the dry loading procedure illustrated in Figure 3. Lot sizes are typically 10,000 primers. After a lot has been completed 900 are withdrawn for sensitivity and dud testing in a ball-drop apparatus as per WS 21535B.

The following flow chart characterizes the current MIC manufacturing process demonstrated at Armament Research, Development & Engineering Center (ARDEC), Los Alamos National Laboratory (LANL) and Naval Surface Warfare Center/Indian Head Division (NSWC/IHDIV):

![Flow Chart]

A brief description of the current procedure for manufacturing the MIC primer mix is illustrated in Figure 4. Recent technical advances in the MIC program have shown that MIC mixtures utilizing bismuth trioxide as the oxidizer can be wet mixed and loaded without significant degradation of the mix. Bismuth trioxide is currently being used as the oxidizer in all MIC development programs conducted at ARDEC and NSWC/IHDIV. Many of the details of the process have been omitted due to the sensitivity of the
information. Qualified organizations with a bona fide need-to-know can contact ARDEC or NSWC/IHDIV directly for access to this information.

The correct amounts of nano aluminum, oxidizer (bismuth trioxide) and other additives are placed into a mixing vessel. The optimum amount of solvent (water, hexane, cyclohexane or isopropyl alcohol) is added, and the composition is then mixed with either an ultrasonic probe or in an ultrasonic bath. The mixing time is dependant on the method used. After mixing, the material is loaded into a primer cup, the anvil is inserted, and the loaded primer is placed into a vacuum oven for drying.

**Steps where personnel may be exposed to nano sized materials**

![Proposed MIC Primer Manufacturing Process Diagram](attachment:image.png)

Figure 4 - Proposed MIC Primer Manufacturing Process