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Lead Free Electric Primer
(SERDP Project WP-1331)

Naval Air Warfare Center, Weapons Division

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NAWCWD overcame some major obstacles, meeting both the low-temperature firing requirement (-65°F) and AUR action time (less than 4ms). Many reformulations were required to optimize the LFEP composite. To date there has not been a single incident with the current aluminum/molybdenum formulation. This demonstrates that the LFEP can be used with suitable safety guidelines. The final formulation consisted of 76% Al(50nm)/MoO_3(45nm), 20% BTATZ, 2% Kel-F, and 2% Carbon Black. A critical parameter for nano aluminum powder stability in air was a passivation oxide thickness of at least 2.7 nm. Al powders with thinner oxide layers aged badly resulting in poor primer performance.

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CONTENTS

Acronyms 4

Executive Summary 6

Objective 7

Background 7
- SEED Program 7
- Follow-On Program 9

Materials and Methods 10
- Technical Approach 10
- Materials Characterization 10
- Formulation 11
- Safety Characterization 11
  - Human-Body Static Energy 12
- Production Methods 13
- AUR Test Setup 13

Accomplishments 15
- Materials Characterization 15
  - Aluminum 15
  - Molybdenum Trioxide 18
  - Bismuth 21
  - Metastable Interstitial Composite (MIC) 21
- Formulation 22
- Alternative Oxidizers 29
- Safety Characterization 31
  - Handling Safety 34
- Production Methods 40
  - Mixing 40
  - Loading 46
  - Solvent-Based Loading 46
  - Puszynski Method 49
  - Solvent-Based Method Optimized 51
  - Production Processes 52
- AUR Results 52

Partnering 57
- Collaboration/On-Site Training 58

Summary and Conclusions 63

Figures

Figure 1 – Conventional Primer Configuration 8
Figure 2 – Exploding Foil Primer Configuration 9
Figure 3 – ESD Test Apparatus 12
Figure 4 – Cold-Temperature Test Shroud 15
Figure 5 – SEM Photographs of MoO_3 Morphology 20
Figure 6 – Scanning Electron Micrographs of Bi_2O_3 From Different Vendors 21
Figure 7 – Bismuth (III) Oxide Ignition Testing 25
Figure 8 – Pressure Cell Versus Pan Dent Optimization 27
Figure 9 – Pan Dent Optimization of Al (50 nm)/MoO_3 Composite 28
Figure 10 – Ignition of I_2O_5 On Aluminum Pan 29
Figure 11 – Percentage of Primers Fired Versus Voltage 34
Figure 12 – Maximum Pressure Versus Gas Additive 44
Figure 13 – Pressurization Rate Versus Gas Additive 44
Figure 14 – MIC Pressing Operation 48
Figure 15 – Rams and Dies Used to Load MIC Electric Primers 48
Figure 16 – Separated Al and Bi_2O_3 50
Figure 17 – Pressure Versus Time Curve for Typical FA 956 Primer Energetics 61
Figure 18 – Pressure Versus Time Curve for MIC +10 Primer Energetics 61

Tables

Table 1 – NTI/NovaCentrix Versus NAWCWD Powder Characterization 16
Table 2 – Particle Size Growth Versus Atmospheric Exposure Time 18
Table 3 – Effects of Heat Treatment on the Particle Size of MoO_3 19
Table 4 – Effect of Temperature and Dwell Time on Crystal Structure and Particle Size of MoO_3 19
Table 5 – Results of Informal Binder/Solvent/Formulation Study 24
Table 6 – Alternative Fuel/Oxidizer Combinations 24
Table 7 – Aluminum Pan Dent Results (80nm Al/320nm Bi_2O_3) 26
Table 8 – Archival M52A3B1 Data 32
Table 9 – M52A3B1 Data 33
Table 10 – Experimental Primer Data 33
Table 11 – MIC/BTATz Ignition Tests 35
Table 12 – ESD Mitigation Experiments For Nano Al Powders 36
Table 13 – ESD Data for Nanocomposites & Coated Nanocomposites 37
Table 14 – BAM Friction and Hammer-Impact Test Results 39
Table 15 – ESD and BAM Friction Test Results (MIC) 40
Table 16 – MIC Formulations and Results of Mixing Experiments 41
Table 17 – Comparison of Mixing Techniques with Alternative Fuels and Oxidizers 42
Table 18 – Al/Oxidizer Material Mixing 43
Table 19 – Samples Prepared Using Different Mix Times and Amplitudes 46
Table 20 – Modified Water Loading Method Results 50
Table 21 – Alternate MIC Formulations Investigated During FY04 53

Table 22 – Phase III AUR Firing Test Data Summary 53
Table 23 – AUR Firing Test Data Summary 55
Table 24 – AUR Test Results 56
Table 25 – Time to Peak Pressure (milliseconds) 60
Table 26 – MIC +10 Conductive Mix Results Data 62

Appendices

Appendix A – Updated Schedule/Milestones A-1
Appendix B – Primer Mix Formulation Data B-1
Appendix C – Primer Fabrication Information C-1
Appendix D – Functional/Firing Test Data D-1

ACRONYMS

AB	Acetylene Black
ADN	Ammonium dinitramide
Al	Aluminum
ATK	Alliant Techsystems Corporation
AUR	All Up Round
BET	Brunauer, Emmett and Teller Method
Bi_2O_3	Bismuth (III) Oxide
BTATz	3,6-bis(1H-1,2,3,4-tetrazol-5-ylamino)-1,2,4,5-tetrazine
BTL	Ballistics Test Laboratory
CAD	Cartridge Actuated Devices
CRADA	Cooperative Research and Development Agreement
DOD	Department of Defense
DOE	Department of Energy
ESD	Electro-Static Discharge
ESTCP	Environmental Security Technology Certification Program
FTIR	Fourier Transform Infrared Spectroscopy
GDOTS	General Dynamic Ordnance and Tactical Systems
HAP	Hazardous Air Pollutant
HBSES	human-body static energy simulator
IPR	In-Process Review
JPWG	Joint Primer Working Group
JWG	Joint (Service) Working Group
Kel-F	fluorocarbon-based polymer (PolyChloroTriFluoroEthylene)
LANL	Los Alamos National Laboratory
LCAAP	Lake City Army Ammunition Plant
LFEP	Lead Free Electric Primer
LN2	Liquid Nitrogen
M/O	Metal to Oxidizer Ratio
MIC	Metastable Interstitial Composite
MICWG	MIC Working Group

MoO_3	Molybdenum Trioxide
MOU	memorandum of understanding
NAWCWD	Naval Air Warfare Center Weapons Division
NMC	New Materials Committee
NMR	Nuclear Magnetic Resonance
NSWC-IH	Naval Surface Warfare Center, Indian Head
NTI	Nano Technology, Inc.
PAD	Propellant Actuated Devices
PEG	Polyethylene Glycol
PETN	Pentaeryhritol Tetranitrate
SEED	SERDP Exploratory Development
SEM	Scanning electron microscopy
SERDP	Strategic Environmental Research & Development Program
SOP	Standard Operating Procedure
TCG	Technical Coordinating Group
TGA	Thermogravimetric Analysis
TTU	Texas Tech University
VOC	Volatile Organic Compound
WO_3	Tungsten Trioxide

EXECUTIVE SUMMARY

The overall objective of the Lead-Free Electric Primer (LFEP) Program was to determine if metastable interstitial composite (MIC) material could be used as a lead-free alternative to existing primary energetic components in electric primers for medium caliber ammunition.

The use of ammunition and other ordnance components containing heavy-metal-based energetics threaten the environment and the well-being of persons involved in production, testing, training, combat use, and disposal of such items. The LFEP Program, funded by the Strategic Environmental Research and Development Program (SERDP) Office, was a continuation of the successfully transitioned SERDP Exploratory Development (SEED) Program (Project Number 1183). Both the SEED and the follow-on LFEP efforts focused on validating the feasibility of replacing lead-containing materials with MIC materials in electric primers for medium caliber gun systems.

The overall approach for the follow-on LFEP program was to thoroughly investigate all aspects of MIC material in the intended application from the perspectives of safety, reliability, production feasibility, cost, shelf life, and its ultimate performance. For the purposes of this program, MIC was defined in its simplest form as a mixture of nano aluminum (Al) particles as the fuel and an appropriate oxidizer. The majority of the efforts associated with this program involved the use of molybdenum trioxide (MoO_3) as the oxidizing agent. However, in an effort to produce increased gas pressures and thereby improve primer performance, a number of different additives to the basic MIC material were evaluated.

Independent characterization of commercial nano material was a critical effort to ensure material quality. Aluminum, molybdenum, and bismuth powders were obtained from various vendors. The samples were characterized and performance was evaluated. Investigation of aging properties and subsequent degradation of MIC was also a strong focal point for all efforts.

Once materials were characterized and their properties were investigated, several tasks were identified in order to smoothly transition a MIC formulation into a primer and eventually all-up round (AUR) for military use. The first and most critical task was to test the MIC formulation for reproducibility using different manufacturers' aluminum and gas generants and to characterize performance.

The down-selected candidate for the Lead-Free Electric Primer Program was KTHU-22. This baseline primer mix consisted of 76% MIC (Technanogy 50-nm Al powder), 20% BTATZ, 2% Kel-F and 2% carbon. The action time for ambient and cold temperatures fell consistently within specifications.

OBJECTIVE

The overall objective of the Lead-Free Electric Primer (LFEP) Program was to determine if metastable interstitial composite (MIC) material could be used as a lead-free alternative to existing primary energetic components in electric primers for medium caliber ammunition.

A number of additional critical parameters were also investigated. These included life cycle cost; end-item safety production issues, including safety in handling, producibility, and unit cost; long-term environmental issues; and performance in a broad range of environments.

BACKGROUND

Lead-containing primers are widely employed in U.S. military ammunition, and use of these items and other ordnance components containing heavy-metal-based energetics, continue to threaten the environment and the well-being of persons involved in the production, testing, training, combat use, and disposal of such items. The LFEP Program, funded by the Strategic Environmental Research and Development Program (SERDP) Office, was a continuation of the successfully transitioned SERDP Exploratory Development (SEED) Program (Project Number 1183). Both the SEED and the follow-on LFEP efforts focused on validating the feasibility of using MIC materials in electric primers for medium caliber gun systems.

This section contains a very brief description of some of the basic elements of both the SEED program and the phases of the follow-on LFEP program. A more complete description of these completed phases of the program is included in later sections of this report.

SEED PROGRAM

The twelve-month SEED program demonstrated that the standard M52A3B1 electric primers for 20-mm ammunition could be replaced by using MIC as the principal energetic component and the replacement successfully initiated using conventional means. However, there were a number of unexplained phenomena and unresolved issues brought to light during this early feasibility effort. It was observed that the electrical conductivity of the MIC material used in the primer mix significantly changed over a relatively short interval of time. The initiation of MIC-based primers was likewise highly variable, working one day and not the next. Resolution of these and other fundamental questions was critical to the success of the follow-on effort. Consequently, the early stages of the follow-on effort focused on improving the basic understanding of MIC and its behavior over time and when exposed to varying environmental and operational conditions.

The SEED program initially started with the concept of simply substituting an electrically conductive MIC material for the normal lead based energetic material currently used in the

M52A3B1 medium caliber electric primer. The basic configuration of this conventional electric primer design is shown in Figure 1. Investigators discovered early in the SEED effort that simple substitution of a MIC-based primer mix for the normal lead based formulation did not produce reliable "ohmic heating" ignition.

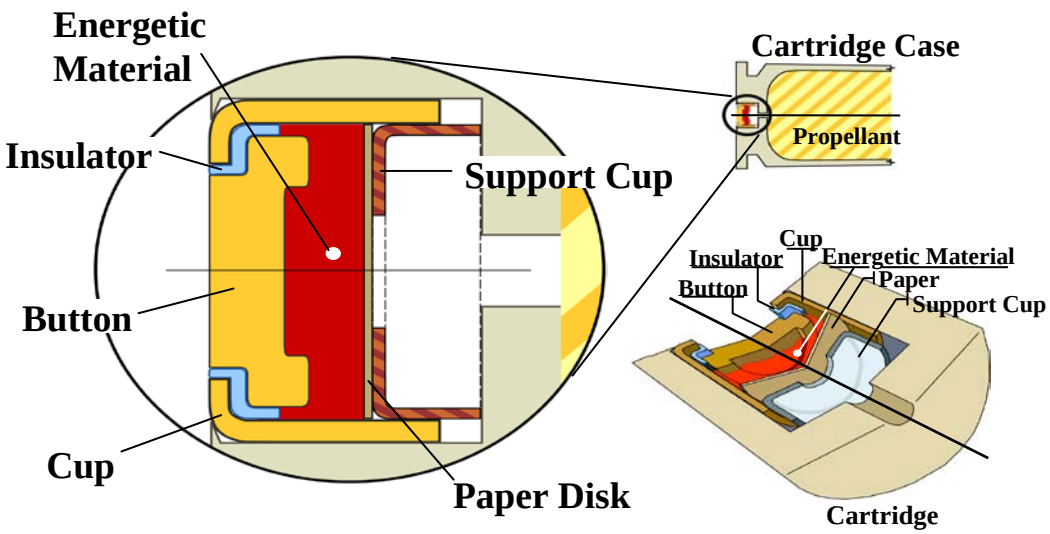


Figure 1 – Conventional Primer Configuration

Due to these primer initiation issues, an alternative ignition method was investigated. The physical configuration of this alternative design, before the MIC was consolidated into the cup, is illustrated in Figure 2. The principal feature of this design was the placement of an aluminum-foil-coated Mylar "ribbon" within the primer cup in such a way as to create an electrical "bridge" through which the primer firing current would pass. The metal film portion of the bridge was evaporated when the high voltage (300 – 450 VDC) pulse was applied. The resulting plasma was very effective in igniting the MIC primer mix, which was consolidated on top of the Mylar/metal foil. This concept was evaluated and determined to be an impractical ignition approach and possibly more costly to implement.

Some of the earliest activities of the SEED Program were associated with meeting mandatory safety tests. A small number of different MIC formulations were prepared and evaluated to determine the basic sensitivity to friction, impact, and electrostatic discharge (ESD) of these materials. These tests led to the conclusion that MIC materials are highly sensitive to friction and ESD, but less sensitive to impact. The results of these tests have directly affected the local handling and processing of MIC materials at NAWCWD China Lake.

Figure 2 – Exploding Foil Primer Configuration

Although the work accomplished during the SEED Program left many questions unanswered, the final result of the effort was the identification of two candidate LFEP primer designs that could be successfully initiated using conventional electric primer voltages and current levels. Both of these candidate designs used MIC as the primary energetic component, with one depending on ohmic heating of the primer mix as the initiation process and the other using the very robust exploding foil approach. This success led to the transition of the program into the follow-on LFEP Program.

FOLLOW-ON PROGRAM

The planning for the follow-on effort on the LFEP Program was completed during the winter and spring of 2002. The plan included the active participation of the U.S. Army as well as other U.S. Navy and Department of Energy (DOE) laboratories. The Navy was to focus their attention on establishing a lead-free primer design as a substitute for the current 20-mm M52A3B1 primer. The Army was, in a similar fashion, going to investigate the use of MIC-based primer material in the low impedance PA520 electric primer used in the lightweight 30-mm gun system, employed on the AH-64 Apache helicopter. DOE and Navy laboratories took advantage of their academic contacts to leverage the expertise and capabilities of the organizations involved in MIC research and served as a valuable resource of information on MIC technology for the Navy and Army. The overall LFEP program was structured around three nominally twelve-month long phases with relatively simple objectives. Phase I was principally focused on acquiring the necessary materials to produce several different MIC compositions and evaluate their basic properties through laboratory testing and limited test firing operations. Phase II was designed to thoroughly evaluate the candidate primer configurations under laboratory conditions and develop safe mixing and pressing procedures as well as perform a limited number of all-up-round (AUR) firing tests. The final Phase III effort was designed to continue the evaluation process and, through statistically significant test processes, ultimately arrive at a

single primer design that could serve as a suitable entry point into a subsequent design and development program leading to production and introduction into military ordnance systems. The schedule and milestones for this program can be found in Appendix A.

MATERIALS AND METHODS

TECHNICAL APPROACH

The overall technical approach for the follow-on LFEP program was to thoroughly investigate all aspects of MIC material for use in medium caliber ammunition primers from the perspectives of safety, reliability, production feasibility, cost, shelf life, and its ultimate performance in the intended application. For the purposes of this program, MIC was defined in its simplest form as a mixture of nano aluminum (Al) particles as the fuel and an appropriate oxidizer. The majority of the efforts associated with this program involved the use of molybdenum trioxide (MoO_3) as the oxidizing agent. However, in an effort to produce increased gas pressures and thereby improve primer performance, a number of different additives to the basic MIC material was evaluated. These alternate formulations will be discussed in greater detail in appropriate sections of this report.

To help ensure the successful accomplishment of program objectives, the Navy LFEP Team worked closely with other Navy labs as well as Army and DOE personnel from the Picatinny Arsenal and the Los Alamos National Laboratory (LANL), respectively. There was successful collaboration in the evaluation of a number of different aspects of MIC formulations. In particular, the Navy LFEP Team benefited, because the Army was able and willing to share knowledge gained through their investigation of the use of MIC for small-caliber percussion primer applications.

In addition to working closely with the China Lake LFEP Team, the LANL group also established independent working agreements with the Naval Surface Warfare Center, Indian Head Division (NSWC-IH), to conduct analyses and tests in connection with NAVSEA's lead-free cartridge-actuated devices (CADs) and propellant-actuated devices (PADs). At the same time, LANL was also supporting the Army's work on the 25-mm M115 percussion primer. From the Navy's perspective, everyone benefited from these cooperative and collaborative efforts, because knowledge gained on the characteristics and behavior of MIC material was appropriately shared in a mutually helpful environment.

MATERIALS CHARACTERIZATION

The independent characterization of commercial nano material was a critical effort to ensure material quality. Aluminum, molybdenum, and bismuth powders were obtained from various vendors. The samples were characterized and performance was evaluated. Aging properties and subsequent degradation of MIC was also a strong focal point for all efforts. The Navy's China Lake Chemistry Division devoted a significant amount of laboratory time to studying this factor.

Several tests were conducted to characterize nano materials, as well as study aging and performance of the individual nano powders and overall MIC formulation.

Thermogravimetric Analysis (TGA) was used to determine the active Al content by determining mass increase as the temperature was raised and the Al was oxidized. Particle size was determined using Brunauer-Emmett-Teller (BET), x-ray defraction techniques, and scanning electron microscopy (SEM). Once particle size and active Al content were determined, the average oxide thickness was determined. TGA, Fourier Transform InfraRed (FTIR), and solid state Nuclear Magnetic Resonance (NMR) techniques were used to characterize acetylene black (AB) used to make the MIC mixes, all of which are discussed in the Accomplishments Section of this report.

MIC performance characterization was also addressed. Establishing a fast and reliable method for assessing the properties of MIC and primer composites was essential. To address this need, the pan dent test was developed. Nano material was placed on a disposable 44-mm diameter aluminum dish and ignited. The dent created was measured using a Scherr-Tumico micrometer. The dent deflection in millimeter per gram of material was calculated and evaluated for performance.

FORMULATION

Once materials were characterized and their properties were investigated, several tasks were identified in order to smoothly transition a MIC formulation into a primer and eventually all-up-round (AUR) for military use. The first and most critical task was to test the MIC formulation for reproducibility using different manufacturers' Al and gas generants and to characterize performance discrepancies. Second was the evaluation of the safety characterization. Third was the evaluation of safe mixing and loading methods including potential use of Puszynski's water-loading method as a technique to allow for water loading of the molybdenum oxide-based-primer formulation with minimal impact on performance. Also, there was a need to characterize other potential oxidizers, including bismuth (III) oxide, for performance and manufacturability comparison as alternative production options to ensure program continuation as discussed in a subsequent section.

SAFETY CHARACTERIZATION

The use of MIC and specifically nano phase materials has led to several safety concerns because of the high sensitivity associated with the materials. There have been several incidents in DOD, DOE, and industry activities in recent years. The need to establish safety data and handling procedures has been a critical part of this program since its inception.

The optimization of a primer formulation to desensitize ESD sensitivity and friction without affecting performance was a priority. ESD tests suggested that LFEP sensitivity would be comparable to or lower than the current M52A3B1 primer under proper handling and assembly conditions. However, due to limited test equipment with a fine enough resolution to gather safety and sensitivity data for the MICs and base material, an ESD test apparatus, shown in Figure 3,

was developed. The ESD tester was equipped with a 5kV power source, exchangeable capacitors, and variable gap. Discharge energies ranged from 0.00002 to 2.25 Joules. This system was setup to evaluate changes in ESD sensitivity for each new formulation and to help characterize materials. The spark energy was calculated by the formula $E = 0.5CV^2$, where C is capacitance (F), V is voltage, and E is energy (J). The human body ESD capacity was estimated at 1 mJ, and a material was considered a non-ESD hazard at 250mJ.

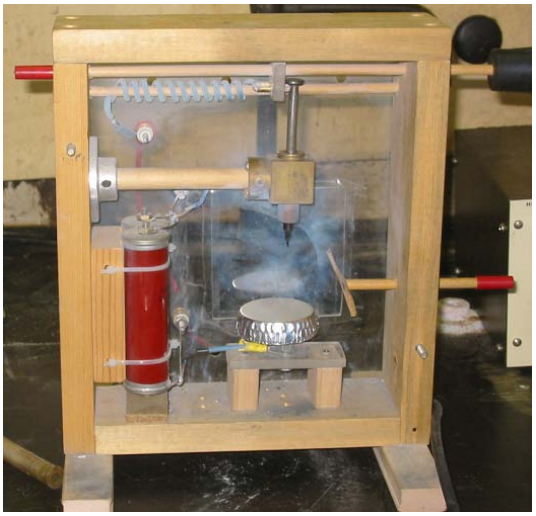


Figure 3 – ESD Test Apparatus

Friction was believed to be the most likely cause of the incidents at DOD, DOE, and industry facilities. As a result, BAM Friction Tester and Hammer-Impact testing was conducted using both ceramic and steel plates.

Human-Body Static Energy

Due to the highly sensitive nature of the BTATz MIC, the New Materials Committee (NMC) at China Lake determined that further safety testing needed to be conducted on all-up loaded primers. The NMC wanted to assure that assembled primers and loaded cartridge cases could be safely handled. It was decided to conduct experiments using a human-body static energy simulator (HBSES). The equipment to perform this test had been previously set up and used at China Lake. However, its condition had deteriorated, and it needed to be reconditioned and recalibrated before it could be used. This test device was relocated to the quality analysis (QA) lab where it underwent a significant amount of work to make it usable. Following this extensive reconditioning effort, the HBSES was found to be capable of delivering a measured amount of energy simulating a static discharge. Voltage was varied across the gap to determine the voltage capable of a human body to simulate a static discharge. The results of the ESD, bam friction, hammer-impact, and HBSES testing are discussed in the accomplishments section.

PRODUCTION METHODS

Primer mixing processes, loading procedures and all-up-round (AUR) testing were investigated with the objective of reducing the hazards associated with material preparation. With regard to mixing, the China Lake Chemistry Division performed a series of qualitative laboratory tests. The objective of these tests was to investigate the effects of alternative preparation (mixing) techniques. The three mixing methods include dry mixing, ultrasonic bath for up to one hour, and an ultrasonic horn for up to two minutes.

Efforts were made to find a solvent to facilitate handling of the MIC based primer mix in a production environment. There were some considerations given to how to consolidate a wet slurry mix into a primer cup. Two potential methodologies were evaluated. The first alternative loading procedure involved the use of a solvent such as hexane, heptane, or a fluorocarbon. The procedure involves the incremental loading of a hexane-based paste using special tooling that allows a sufficient amount of material to be loaded into the primer in a minimum number of steps. The loading procedure is conducted in explosives pressing room and is performed in accordance with the Standard Operating Procedure (SOP) titled: "Pressing and Weighing of Experimental Energetic Materials" (SOP 10090-03). This procedure has facilitated the performance testing of various MIC primer mixes and is expected to help address issues related to large-scale processing and eventually lead to a method of producing LFEPs in a production environment. The second alternative loading procedure involved Puszynski's water loading method (Jan A. Puszynski, et al, South Dakota School of Mines and Technology).

The LFEP program opted to use AUR testing to evaluate primer functionality and performance. The reasoning was that if a primer could function reliably under typical environmental conditions in its final configuration, then it would be much more likely to meet the specifications and be easily transitioned into production. The standard PGU-27A/B 20-mm specification was used to provide the performance metrics for which the primer was evaluated. The rounds were assembled using industry-supplied hardware components and WC 868 propellant. Each round was assembled according to the drawing specification modifying only the primer formulation. The mixing techniques, proposed solvent loading and water loading techniques, and AUR testing are discussed in detail in the Accomplishments Section.

AUR TEST SETUP

The AURs were test-fired from a single-shot 20-mm Mann Barrel using an electrically charged firing pin. The pressure-velocity and action-time data were taken for each round and compared to PGU-27 A/B checkout rounds to evaluate the performance of these different compositions. All of the reduced performance data is available in Appendix D.

One very important functional feature of any ammunition primer is the need to meet the all-up round action time. Action time is defined as the interval between the point in time at which the primer is struck, or in the case of electrical primers, the time when the fire volts pulse is applied to the primer, and when the projectile exits the muzzle of the barrel. This is a vitally important feature of any externally powered gun since delays in action time can result in a round being initiated after the gun bolt has unlocked and the round is being extracted from the

chamber. This type of event typically destroys major components of the gun and produces seriously dangerous conditions for the aircraft and the personnel manning the aircraft. In the case of the 20-mm round of ammunition, the maximum allowable action time, according to specification MIL-P-1394, is 4.0 milliseconds. Although precise action times for the M52A3B1 primers were not measured, it was noted that the time for first light and maximum light output, as measured with a photo-transistor was on the order of 0.085 and 0.600 milliseconds, respectively. The equivalent times for the non-optimized MIC primers were on the order of 0.140 to 0.250 milliseconds for first light and approximately 1.0 to 1.1 milliseconds for maximum light output. These slightly longer primer action time indicators for the non-optimized MIC primers were very encouraging and lead the investigators to the conclusion that the times could be reduced to meet the specification requirements for cartridge action time

Part of the firing evaluation for candidate primer formulations included low-temperature (-65ºF) conditioning of the rounds for 24 hours and firing from a cold-conditioned barrel. The low-temperature firing tests were intended to provide some initial indication of how the MIC-based primers would perform under these extreme environmental conditions, which is typically a challenge even for conventional lead-based primers. The temperature requirement was determined by the 20-mm lot acceptance specification.

The cold-barrel test stand was fabricated by wrapping half-inch diameter copper tubing around the most rearward 12 inches of the breech end of the Mann barrel. Insulation was placed around the copper tubing to reduce the influx of heat from the surrounding environment. A liquid nitrogen tank (LN2) was connected to the copper tubing using a flexible line. Liquid nitrogen circulated through the copper tubing wrapped around the breech end of the Mann barrel. A shroud was constructed using 1-inch thick Polystyrene foam for insulation and mounted to the breech end of the Mann barrel stand as shown in Figure 4. The LN2 vented from the exit end of the tubing into the shroud to further cool the barrel, stand and the breech between shots. The breech temperature was continuously monitored using a thermocouple and the LN2 application was controlled to maintain a constant temperature for up to 5 minutes prior to the shot. The breech and barrel temperatures were recorded for each test and the breech cooled back to approximately -65ºF before the next round was loaded. Performance results for the AUR testing are given in Appendix D.

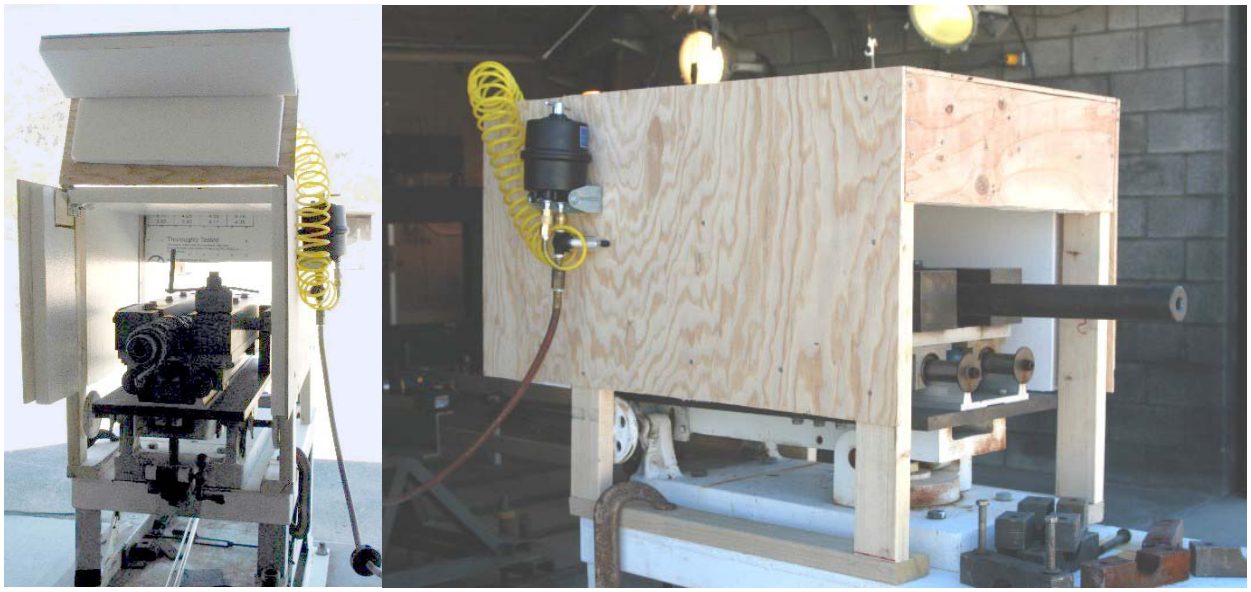


Figure 4 – Cold-Temperature Test Shroud

ACCOMPLISHMENTS

MATERIALS CHARACTERIZATION

Aluminum

The independent characterization of commercial nano materials was a critical effort to ensure material quality. Aluminum powders, 33 and 50 nm in diameter, were obtained from the Technanogy Company. The samples were characterized and performance evaluated. The use of the 50-nm Al proved very effective and resulted in the development of several formulations that met the necessary performance requirements. Unfortunately, Technanogy went out of business, and this required the evaluation of alternative nano aluminum sources.

In 2005, a shipment of 80-nm Al powder was received from Nanotechnologies, Inc. (now NovaCentrix since July 2006), and it was characterized by the China Lake Chemistry Division prior to being used in formulation. Results are located in Table 1. The vendor sent four sealed packages of Al 80 P (Lot Number M2210) labeled M2210A, M2210B, M2210C, and M2210D, each containing about 125g of 80-nm Al with a 2.5-nm oxide passivation layer. The characterization of lot M2210 by Nanotechnologies, Inc; (NTI) was 87% (plus or minus 2%) active Al based on hydrolysis, 85-nm (plus or minus 8.5 nm) particle size based on a weighted average between BET measurements and TGA and an oxide layer between 1.5-2.5-nm. Characterization by China Lake's TGA gave an active Al content to 82.7%. This corresponded to a spherical particle with a diameter of 116 nm, not 80 nm, assuming a 2.5-nm oxide passivation layer. However, the initial oxidation step and particle distribution of this material was far superior to that observed for the 50-n-nm Al from Technanogy. This indicated that the 80-nm Al

had fewer large particles and should be more reactive. In addition, the active Al content of 82.7% was much higher than for the 50-nm Al (65%). Formulations using the 80-nm Al would have less dead weight (Al oxide) and should have exhibited superior performance. Aging studies of this material indicated that the oxide layer thickness was not 2.5-nm thick. The Al particle size was closer to 80 nm with an oxide thickness of 1.1 nm. The thin oxide passivation layer led to severe aging of the Al powder and deterioration of primer performance.

Table 1 – NTI/NovaCentrix Versus NAWCWD Powder Characterization

Sample Number	NTI/NovaCentrix Data				NAWCWD Data			
	Calc'd Al Size (nm)	BET Area (m^2/g)	Active Al %	Calc'd Oxide Thickness (nm)	Calc'd Al Size (nm)	BET Area (m^2/g)	Active Al %	Calc'd Oxide Thickness (nm)
M2443	48	42.7	57	3.5				
M2444	47	43.6	59	3.3				
M2445	48	43.6	63	2.9				
M2446	46	44.4	57	3.4				
M2447	47	43.6	59	3.3				
M2448	51	40.4	64	3.0				
M2449	49	41.9	62	3.1				
M2450	50	41.9	64	2.9				
M2451	**50**	**41.9**	**66**	**2.8**			**61.4**	**3.11**
M2452	52	39.7	65	3.0				
M2453	**74**	**28.9**	**75**	**2.8**			**81.1**	**2.05**
M2454	**78**	**27.1**	**72**	**3.4**			**74.5**	**3.02**
M2455	88	24.4	79	2.8				
M2210D	**85**	**25.1**	**87**				**82.7**	**1.15**
Aged M2210D							**74.1**	

A 400-Watt Branson 450 Digital Sonifier (horn) was acquired in 2005 to support the various mixing-process evaluations and preparation of higher quality primer mixes with accordingly higher levels of performance. Power levels, duty cycles and timing were varied to determine its effects on the quality of the end product, as described in a subsection below. In order to study aging effects induced by sonication, the active Al content of M2210D was characterized by TGA upon exposure to air by China Lake. The Al powder was sonicated using the 400-W horn under different conditions, varying amplitude and sonication time. The conditions for the 400-W sonication of the 80-nm Al (M2210D) with a 0.5-sec pulse were sonication at 75% amplitude for 2, 1, and 0.5 minutes and 50% amplitude for 2, 1, and 0.5 minutes. The sonicated powders were stored at 20°C in air at 25% controlled humidity. TGA was performed weekly for 6 weeks. The baseline corrected data consisted of storage of the materials in the air and checked every 7 days for a total of 51 days. The active Al content based on weight percent (wt%) started at 85 wt% and decreased to 73.8 wt%. In the first 14 days of storage the active Al content decreased by 4 wt%, then an additional 3 wt% loss occurred during the next 7 days, with the active Al content decrease leveling off at approximately 1 wt% every seven days. Since the untreated Al-80-P powders continued to age after 7 weeks, the sonication data may have been ambiguous.

To verify the results, a sealed bag of the Al-80-P Al powder (M2210A) was sent to Los Alamos National Laboratory (LANL) for aging studies. The ambient temperature and humidity conditions at LANL were not controlled resulting in fluctuating humidity and temperatures that may have produced dramatically different aging behavior. In general, the humidity at LANL is less than 5% except when it rains.

The NTI Al-80-P, 80-nm Al powder was aged to a much greater extent than Technanogy's 50-nm Al powder (with a 2.5 nm oxide passivation layer) that lost only 2.5 wt% active Al content after 1 year in air. The active Al content of Al-80-P dropped from 85% to 74%, an 11 wt% loss after just 7 weeks of exposure to air. This behavior was consistent with a thinner oxide passivation layer. The TGA of active Al content of 85.0% was consistent with an 85 nm Al particle with a 1.6-nm oxide shell. The hydrolysis number of 87% active Al stated by NTI indicated an even thinner oxide shell of 1.35 nm. In discussions with NTI, investigators were told that the oxide thickness was targeted at 1.5 to 2.5 nm. According to their analysis, their material was within their tolerances. They were also aware of the aging issue when their Al was exposed to air, but not the extent. However, they agreed to provide a new batch of 80-nm Al with 2.5-3.0-nm oxide coating. NTI appeared to be willing to resolve the aging issue. They planned to do an aging study at their facility. They did not have control over their environment and the Texas humidity is higher than LANL's. The three aging studies (China Lake, LANL, and NTI) with very different environments provided a better understanding of the importance of humidity on aging of nano aluminum powders. An alternative approach may have been to coat the Al with a long-chain organic compound to inhibit hydrolysis. Investigators planned to evaluate the effects of coating the NTI 80-nm Al with 5 wt% of a perfluoroacid. It was anticipated that the acid would inhibit the hydrolysis/aging process.

Subsequent nano aluminum from NovaCentrix was also characterized in 2006. NovaCentrix was asked to prepare 12 nano aluminum powders with thick oxide layers specifically for the primer program. Nine powders, included in Table 1, were 50-nm Al with oxide thicknesses of 2.8 to 3.5 nm and three were 80-nm Al powders with oxide thicknesses between 2.8 and 3.4 nm. Nano aluminum powders with at least a 2.7-nm oxide layer were found to age less than powders with thinner oxide layers. To date, NAWCWD has characterized only three samples by TGA. All samples were tested within hours of opening the sealed bags. The active Al content in samples M2451, M2454, and M2453 were all different than reported by the vendor. The sample M2453 was particularly disturbing since it was 6.5% higher than the vendor reported. If the size reported by the vendor was correct, the oxide thickness was calculated to be less than 1.1 nm but reported as 2.8 nm. NovaCentrix was informed of the results. NAWCWD characterized nano Al powders using TGA, while NovaCentrix used base hydrolysis. The poor correlation of the characterizations was due to errors in the hydrolysis performed at NovaCentrix. A problem was identified with NovaCentrix's standard hydrolysis method used to determine active Al content. NovaCentrix performed a base hydrolysis on micron-size Al (H3) and determined it to be 68% active Al. It should have been greater than 98%). NAWCWD worked with NovaCentrix to correct this problem.

Molybdenum Trioxide

In 2004, a fresh batch (2 kilograms) of MoO_3 was purchased from the Climax Company. Although Climax MoO_3 had been previously characterized, it was material that had been obtained indirectly through other sources (Technanogy, LANL, NSWC-IH, etc.). The condition of the MoO_3 was critical to the performance of MIC, and it had been shown to degrade when exposed to atmospheric moisture and light. Exposure to moisture caused the formation of hydrates, and storage in light changed the color from off-white to a bluish green. For these reasons, the container containing this fresh material was wrapped in aluminum foil and stored under argon gas in a glovebox.

The BET surface area measurements of the MoO_3 immediately after it was removed from the glovebox indicated an average particle size of 25.5 nm. Table 2 describes the particle size growth when exposed to the atmosphere over the passage of time.

Table 2 – Particle Size Growth Versus Atmospheric Exposure Time

Exposure Time (Days)	Particle Size (nm)
0	25.5
1.5	35.4
14	41.0
30	120.0

Upon aging, the MoO_3 color changed from pale yellow to dark green. This change in color was due to the formation of sub-oxide species but the change in particle size may or may not be associated with the color change.

TGA of the MoO_3 showed an approximate 2% weight loss, and x-ray diffraction showed that the material consisted of the monoclinic and orthorhombic phases of MoO_3 and the MoO_3 hydrate. The 2% weight loss was due to the dehydration of the MoO_3 hydrate. The monoclinic phase of MoO_3 was also found to readily absorb water to form the hydrated phase, while the orthorhombic phase did not form the hydrate. The significant amount of the monoclinic form of the material marked a potential aging problem for this material, dictating that it be heat treated prior to use in any mixing operations.

It was also recognized that heat treatment of the MoO_3 produced a growth in particle size, and these larger sized particles of MoO_3 produce slower reaction times that adversely affect the action time of AURs. Two separate studies were undertaken to determine the relationship between heat treatment times and temperatures.

In the first series of tests, samples of Climax's nano phase MoO_3 were heated to 400 and 500 ºC for periods lasting from 15 minutes to 3 hours. The results of this initial study are highlighted in Table 3.

Table 3 – Effects of Heat Treatment on the Particle Size of MoO_3

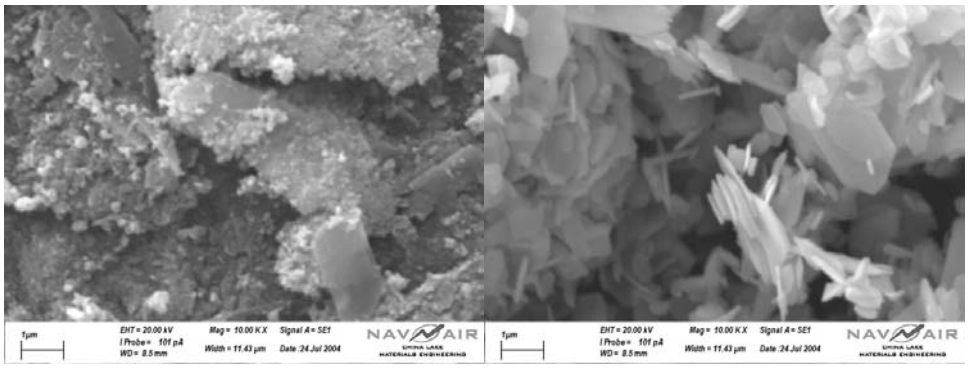
Heat Treatment Parameters	**Results of Heat Treatment on Mixtures**
Heat treatment @ 400ºC for thirty minutes	Partially converts monoclinic form to the orthorhombic form with the monoclinic form still being dominant
Heat treatment @ 400ºC for one hour	The monoclinic form is less prevalent, but is still the dominant form
Heat treatment @ 400ºC for a period of two hours	Completely converts all of the MoO_3 to the orthorhombic form – Exact time for total conversion is still being studied, but lies somewhere between one and two hours
Heat treatment @ 500ºC for fifteen minutes	Completely converts all of the MoO_3 to the orthorhombic form

The second laboratory study related to heat treatment and particle size growth of MoO_3 was similar, but used slightly different parameters. The specific objective of this second heat treatment study was to identify the exact steps required to convert the MoO_3 to the desirable orthorhombic phase with minimal particle size growth. Studies showed that conversion of the MoO_3 from the monoclinic to the orthorhombic phase required temperatures of at least 300ºC, but these conditions also produce larger particle sizes that may adversely affect primer action times. In this study, the samples of material were heated to 400, 443 and 500ºC for periods ranging from 15 minutes to 3 hours. X-ray diffraction techniques were used to monitor the conversion of the monoclinic phase to the orthorhombic phase. Table 4 summarizes the results of this second heat treatment study.

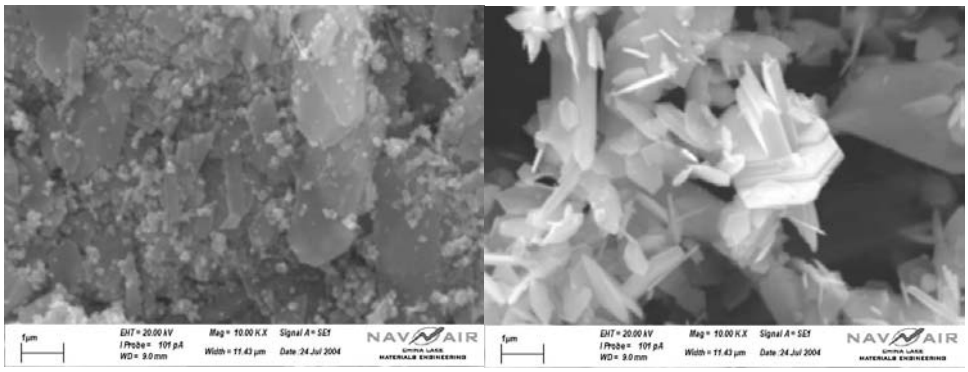
Table 4 – Effect of Temperature and Dwell Time on Crystal Structure and Particle Size of MoO_3

Time (Mins)	Temperature					
	400°C		443°C		500°C	
	Crystalline Form	Size (nm)	Crystalline Form	Size (nm)	Crystalline Form	Size (nm)
0	Mono/Ortho	25	Mono/Ortho	25	Mono/Ortho	25
15	Mono/Ortho		Mono/Ortho		Ortho (100%)	220
22	Mono/Ortho		Ortho (100%)		Ortho (100%)	
30	Mono/Ortho		Ortho (100%)		Ortho (100%)	
60	Mono/Ortho		Ortho (100%)		Ortho (100%)	
120	Ortho (100%)		Ortho (100%)		Ortho (100%)	
180	Ortho (100%)	198	Ortho (100%)		Ortho (100%)	

Scanning electron microscopy (SEM) was used to determine the morphology of the MoO_3 at different temperatures and exposure times. The photographs included in Figure 5 illustrate the observed effects.



Untreated MoO_3 after 24 Days in Air MoO_3 Heated to 500ºC for 15 Mins



MoO_3 Heated to 400ºC for 15 Mins MoO_3 Heated to 400ºC for 180 Mins

Figure 5 – SEM Photographs of MoO_3 Morphology

These heat treatment studies resulted in the following observations and conclusions. It took 1-2 hours for the complete conversion of monoclinic to orthorhombic MoO_3 at 400°C. Complete conversion took less than 22 minutes at 443°C and less than 15 minutes at 500°C. There was significant particle size growth during the conversion. Orthorhombic crystalline structure appeared to be exclusively in plate morphology. Heat treatment at lower temperatures for longer times had less impact on particle size.

It was also observed that the molybdenum trioxide (MoO_3) had shifted in color from a near white hue to a blue-green color. It was thought that it had been significantly affected by the absorption of water from the atmosphere. X-ray analysis by China Lake revealed that the container of MoO_3 received from the Climax Company contained three distinctly different crystalline forms of the material, the monohydrate ($MoO_3 \cdot H_2O$) form, as well as the orthorhombic and monoclinic forms. A preliminary heat treatment method was shown to convert the Climax MoO_3 to the pure orthorhombic phase. This data was reported to the MIC Working Group (MICWG), and the Air Force used this data to develop a large-scale thermal treatment process. This process involved placing the MoO_3 in an oven and heating it to a specific threshold temperature (500ºC) for an appropriate period of time (4 hours). The Air Force disseminated heat-treated MoO_3 samples to MICWG members for testing and evaluation. The orthorhombic phase was reported to be water stable. This was verified in the laboratory by placing

orthorhombic MoO_3 samples in water and in air, by monitoring them over time by X-ray analysis. The heat treated MoO_3 (orthorhombic) also lost its photosensitive behavior and did not undergo the color change even after storage in air for 6 months.

Bismuth

In 2005, material characterization was conducted on nano Bismuth (III) Oxide (Bi_2O_3) as a possible alternative to MoO_3. Samples from Sigma-Aldrich and Skylighter were characterized. Aldrich claimed an average spherical particle size of 90 nm. However, BET measurements performed at NAWCWD China Lake gave an average particle size of 320 nm. TGA exhibited only a very small weight loss (0.09%) due to adsorbed species. SEM of the material exhibited a wide distribution of spherical particle sizes in the range of 50 to 500 nm. Samples of Skylighter CH8040 material were received for characterization from LANL and directly from the vendor. SEM of the material revealed rectangular prism shaped particles in lengths ranging from 1 to 10 microns. However, LANL reported the average particle size of the Skylighter CH8040 material to be between 1 to 3 microns. The material received directly from Skylighter exhibited a BET surface area of approximately 0.27 square meters per gram as compared to 0.9 square meters per gram for the Skylighter sample from LANL. This indicated high size variability from the vendor. A performance difference was also noted for MICs prepared from the two Skylighter Bi_2O_3 samples due to the difference in particle size. Figure 6 shows SEMs of vendors' samples.

Sigma-Aldrich 10 micron | Skylighter 2.5 micron | Aldrich 320 nm

Figure 6 – Scanning Electron Micrographs of Bi_2O_3 From Different Vendors

The Skylighter Bi_2O_3 is the cheapest and most readily available material, but there is wide variation in lot-to-lot particle size.

Metastable Interstitial Composite (MIC)

The aging properties and subsequent degradation of MIC was a strong focal point for all of the efforts. In 2003, the Navy's China Lake Chemistry Division devoted a significant amount of laboratory time to studying this factor and arrived at some very important conclusions. In the early stages of this investigation, a number of potential causes of the observed aging/ deterioration were examined. These potential causes included exposure to the atmosphere, chemical reactions between the MIC materials and the metal parts used in the fabrication of the

electric primers, and potential chemical reactions between the constituents of the MIC, including the possibility of introducing contamination (moisture and other chemicals) into the MIC by having added carbon, in the form of acetylene black (AB), to the mixture for enhancement of the electrical properties of the primer mix.

The thickness of the protective oxide layer on the nano aluminum was a large factor in the behavior of the MIC. Tests verified that an oxide layer less than 2.7-nm thick was not effective in protecting the Al from further oxidation through contact with the atmosphere, reduction in the percentage of active Al, and subsequent degradation in reliability of initiation and performance. It was determined that nano aluminum particles having an oxide layer of at least 2.7-nm thick adequately protected the aluminum.

Other observations with respect to aging were that untreated MoO_3 was sensitive to light and should be stored in a light free environment; ultrasonic processing of the materials during the mixing process might adversely affect the oxide layer of the nano aluminum and promote degradation of the sensitivity and energy content of the final MIC product; the chemical compatibility of the metal parts comprising the primer cup and the constituents of the MIC did not appear to be a problem; and other constituents of the primer mix, such as AB carbon, did not appear to have any observable short-term negative effect on the primer mixture.

In general, vendors of nano materials appear to have a problem with lot-to-lot variances that can have dramatic impact in performance. Material characterization and standardization of material characterization is essential for DOD weapons systems. Failure of DOD systems due to defective vendor materials could be catastrophic. These issues have been relayed to the members of the Joint Primer Working Group (JPWG) and are continually being addressed.

FORMULATION

With the nano powder characterization issues being addressed, focus then shifted to MIC performance characterization. In 2003, the use of micron-sized Al particles in lieu of nano particles was investigated to reduce sensitivity and at the same time improve handling properties of the MIC. Subsequent tests of primers fabricated using these larger particles indicated that this particular formulation was difficult to initiate and resulted in unacceptably long cartridge action times. Cartridge action time is defined as the interval of time between the instant that the primer is initiated and the projectile exits the muzzle of the gun system.

While photos and videos of the MIC-based ohmic-initiated primers indicated a significant reaction, it was noted that they did not consistently provide action times that met specifications. One of the possible explanations of the shortcoming was that the MIC combustion reaction produced virtually no gaseous by-products and the absence of the gas pressure explained the less than desirable performance. This precipitated an effort to identify a gas-producing additive that would help resolve the problem.

Army scientists and engineers working on the earlier MIC-based lead-free percussion primers for small caliber ammunition had encountered similar difficulties in finding a formulation that consistently produced action times within the interval of time specified in the

appropriate requirements documents. It was concluded that the absence of a gas-producing ingredient in their formulation did explain the difficulties being observed.

Ignitability was also a concern when dealing with performance characteristics. The LANL Organization executed a series of MIC ignition tests using a CO_2 laser. Results of these tests clearly showed the strong correlation of particle size to ignitability, with the larger size particles being exponentially more difficult to ignite.

Ignition temperature measurements were also made by LANL, with the cooperation of Texas Tech University (TTU). Again, there was a strong correlation between particle size and the temperatures at which the material reacted, with the smaller particle size materials reacting at lower temperatures than the larger size particles. Using a slightly modified test set-up that involved the use of smaller thermocouples, LANL duplicated these tests.

LANL studied the ignition/combustion of two samples of nano aluminum in air. These nano aluminum samples were obtained from the Technanogy Company and had particle-size characteristics of 33 nanometers and 134 nanometers. These tests demonstrated two stages of burning in air. The initial burning was the normal reaction to the oxygen in air. The secondary burning reaction was with the nitrogen in air.

Other burning tests of nano aluminum were conducted on 40-nm sized particles contained in a Plexiglas box. The Al material was exposed to atmospheric air, nitrogen, argon/nitrogen, argon/oxygen, and pure oxygen gases. The material was ignited using a hot nichrome wire. These tests further highlighted the importance of nitrogen in the reaction of nano aluminum.

Using funding from NSWC-IH, LANL investigated the use of alternative oxidizers in MIC formulations. Both tungsten oxide (WO_3) and bismuth (III) oxide (Bi_2O_3) demonstrated excellent performance in initial tests using peak pressure measurements as a comparative metric.

Transitioning to 2004, in an initial study, a small number of different binders and solvents were studied to determine their applicability to the LFEP application. The results of this relatively simple effort are shown in Table 5. Kel-F contains chemical energy and serves as both a binder and an energetic component of the primer mix. However, it was learned as part of this program that the use of this material is sensitive to how it was blended into the overall primer mix. Laboratory testing showed that it was important to add the AB carbon to the mixture as a final step, since a Kel-F coating on the AB negatively affected its electrical properties and subsequently had an adverse effect on the ignitability of the primer mix. Properly blended into the primer mix, the Kel-F material demonstrated improved cartridge action time consistency and will be a constituent in the final MIC formulation.

Table 5 – Results of Informal Binder/Solvent/Formulation Study

Binder/Solvent/Formulation	Performance
Polyethylene glycol (PEG) binder W/ hexane solvent – MIC (56%) + BTATz (40%) + PEG (2%) + AB (2%)	Spark produced loud 'Pop' similar to Kel-F formulation
Kel-F + hot heptane (75 ºC)	Nice homogeneous solution
Kel-F + hot toluene (75 ºC)	Nice homogeneous solution
BTATz = bistetrazolylaminotetrazine	

Beginning in 2005, alternative fuel/oxidizer combinations were also investigated in order to develop a more effective energetic nano composite material for the LFEP application. The samples prepared for comparison are listed in Table 6. The fuels used in this investigation included: 50-nm Al (R20, R22) from Technanogy, 80-nm Al from NanoTechnology, 208-nm Al made at NAWCWD, 5-mm Al from Valimet, and lithium Al hydride from Aldrich. Oxidizers of interest included: MoO_3 from Climax (Cl), 50-nm bismuth (III) oxide from Nanophase, 320-nm bismuth (III) oxide from Aldrich, micron bismuth (III) oxide from Skylighter, 10-micron bismuth (III) oxide from Sigma-Aldrich, nickel oxide, tellurium dioxide, and iodine pentoxide.

Table 6 – Alternative Fuel/Oxidizer Combinations

Sample	Date	Fuel	Oxidizer (mmol)	M/O Ratio	Comments
KTHU-12	1/18/2005	Al (R20)	MoO_3 (Cl)	2.747	
KTHU-13	1/18/2005	Al (R22)	Bi_2O_3 (1 μm)	2.054	
KTHU-14		Al (R22)	MoO_3 (Cl)	2.985	
KTHU-15		Al (R22)	MoO_3 (Cl)	3.035	
KTHU-16		Al (R22)	MoO_3 (Cl)	3.011	
KTHU-17		Al (R22)	MoO_3 (Cl)	3.017	
KTHU-18	1/20/2005	Al (R22)	MoO_3 (Cl)	3.024	
KTHU-19	1/21/2005	Al (R22)	MoO_3 (Cl)	3.017	
KTHU-21	1/24/2005	Al (R22)	Bi_2O_3 (1 μm)	3.005	Poor
KTHU-23	1/24/2005	Al (R22)	MoO_3 (Cl)	3.033	
KTHU-24		Al (R22)	MoO_3 (Cl)	2.967	
KTHU-25		Al (R22)	MoO_3 (Cl)	2.952	
KTHU-26		$LiAlH_4$	MoO_3 (Cl)	1.269	
KTHU-29		Al (R22)	I_2O_5	5.815	Need to use 3.33 M/O

The use of lithium Al hydride with molybdenum trioxide gave poor results. The material did not pop when ignited. The alternative micron sized oxidizers, nickel oxide, tellurium dioxide, and iodine pentoxide, also gave poor results.

The bismuth (III) oxide formulations appeared to be very promising. These formulations gave a loud bang when stimulated in almost every formulation, regardless of fuel. The reaction rate/audio report appeared to be dependent on both the Al particle size and the bismuth (III) oxide particle size. MIC formulations using Al (80 nm) powders with bismuth (III) oxide gave decreasing audio reports in the order of 320 nm, greater than 1 micron, and greater than 10 microns. Pan dent testing on lightweight aluminum foil pans was used to help discriminate material performance. In all cases, between 50 to 55 mg of MIC was placed on the aluminum pan and ignited. Formulations using the 80-nm Al with either the 320-nm or 1-micron bismuth (III) oxide ignited with a very loud bang and produced large holes in the aluminum pans. The 80- nm Al plus 10-micron bismuth (III) oxide dented the pan without penetration (Figure 7). The damage to the aluminum pans was probably due to higher reaction velocities that impart higher product impulse.

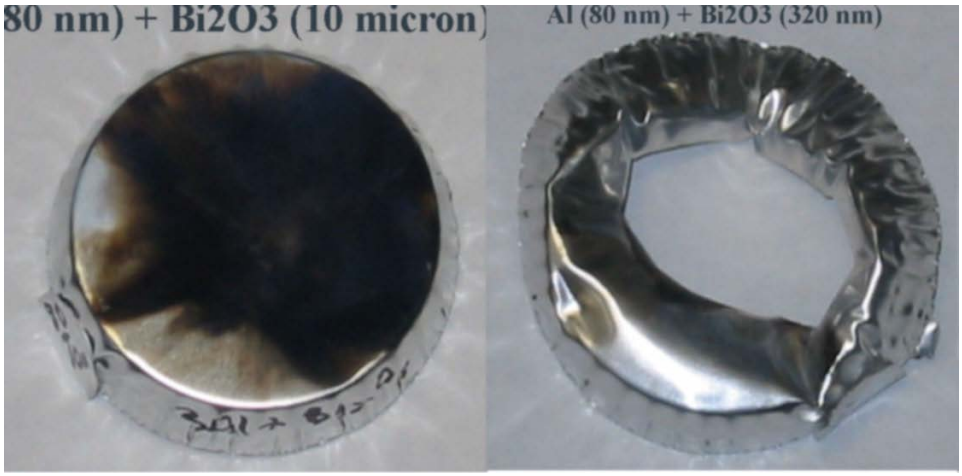


Figure 7 – Bismuth (III) Oxide Ignition Testing

In order to determine the best composition of 80-nm Al/320-nm Bismuth (III) Oxide composition optimization, a series of composites were prepared with varying metal to oxidizer (M/O) ratios ranging from 0.782 to 3.165 (see Table 7). The performance was measured by the impulse damage to the aluminum pan. The minimum amount of composite needed for penetration through the aluminum pan was determined. In summary, at low M/O ratios (0.78 and 1.39, KTHU-79 and KTHU-87, respectively), pan penetration was not achieved indicating poor performance. Pan penetration was observed for M/O ratios between 2.0 and 3.2 (KTHW-3, KTHU-93, KTHU-90, and KTHU-88, respectively) with the best performance observed at 2.05 (KTHW-3). A M/O ratio of 2.0 is the balanced metal to oxide ratio for the reaction of Al and bismuth (III) oxide to form Al oxide and bismuth gas.

Table 7 – Aluminum Pan Dent Results (80nm Al/320nm Bi_2O_3)

Formulation	M/O	Weight of MIC (mg)	Result
KTHU-79	0.782	158	Dent
KTHU-87	1.389	64	Dent
KTHU-88	3.165	38	Penetration
KTHU-90	2.799	22	Penetration
KTHU-93	2.329	18	Penetration
KTHW-3	2.049	15	Penetration

MIC composites using NTI's 80-nm M2210D Al and Skylighter's CH8040 bismuth (III) oxide received directly from vendor were prepared. Although the composites gave good audio reports, 50 mg of composite dented rather than punctured the aluminum pans. Similar MIC composites using Al and bismuth (III) oxide received via LANL consistently penetrated the aluminum pans. The reduced performance is believed to be due to the aging Al (80-nm M2210D), the larger bismuth (III) oxide (CH8040), or both. A composite using the Al (80-nm LANL) and bismuth (III) oxide (CH8040) also gave a loud audio report but failed to puncture the aluminum pan. Unfortunately, the LANL sample of bismuth (III) oxide (Skylighter via LANL) had been expended. A composite using the new Al with the old bismuth (III) oxide (Skylighter via LANL) could not be prepared and tested. The BET surface area measurement of the two batches of Skylighter Bi_2O_3 gave average particle sizes of 1.5 microns (LANL's sample) and 2.5 microns (China Lake's sample). The larger particle size explains the difference in performance observed between the MIC prepared using the two different batches. This raised a question of the vendor's ability to provide a consistent product. The vendor provides materials for pyrotechnic applications where quality control is not nearly as stringent. In order for their material to be used by the DOD, additional ball-milling may be required.

A scaled-up 10-g sample of 50-nm Al (Technanogy) and molybdenum trioxide (Climax) was sonicated using the 400-W horn at 100% amplitude, 0.5-sec pulse for two minutes in 75 ml of hexane. There appeared to be very little movement of the solid material during the sonication process. The viscosity appeared to be too high. This material was isolated by filtration and vacuum dried. When initiated, it gave a good loud pop, but the audio frequency was lower than usual. The lower frequency sound could be an indication of lower reaction velocity.

Energetic nano composites using bismuth (III) oxide and molybdenum trioxide, as a second nano oxidizer, were evaluated. This was done in an attempt to mitigate the high electrostatic discharge (ESD) sensitivity of the Al/bismuth (III) oxide composites and also to increase the energy content, while maintaining the high reaction rate and gas generation. The Al/bismuth (III) oxide/molybdenum trioxide MIC generated a loud audio report, but 50-mg of material only dented the aluminum pan. Further material characterization is in progress.

Sample KTHU-62 was a new Al/bismuth (III) oxide/BTATz/Kel-F primer formulation that was prepared for safety testing and evaluation. However, due to the ESD sensitivity of the bismuth (III) oxide and recent reports of incidents at other test facilities, the safety testing and

evaluation of the new primer formulation was delayed. The handling process was evaluated and safety concerns were addressed. It was also noted that humidity level had a notable affect on ESD sensitivity of the material and that testing would need to be done under low humidity conditions for reliable results.

In 2006, a series of MIC composites were prepared varying the fuel-to-oxidizer ratio. Composites with stoichiometric ratios from 0.75 (oxidizer rich) to 1.5 (fuel rich) were prepared using NovaCentrix's 80-nm Al and Climax's MoO_3. The samples were sonicated using the 400-W ultrasonic horn for 2 minutes at 75% amplitude with a 0.5-second pulse. Using the pan dent test, between 37 and 41 mg of material was placed on disposable 44-mm diameter aluminum dishes and ignited. The dent deflection was used to screen the formulation.

Al (80 nm)/MoO_3 composites with Al/MoO_3 molar ratios from 1.5 to 3.5 were prepared and evaluated using the pan dent test. The data was normalized, graphed (Figure 8), and compared to LANL optimization (P_{max}) of the Al/MoO_3 composites. Although the curves did not exactly match, they were similar and both maximized at a molar ratio of Al to MoO_3 of 2.5.

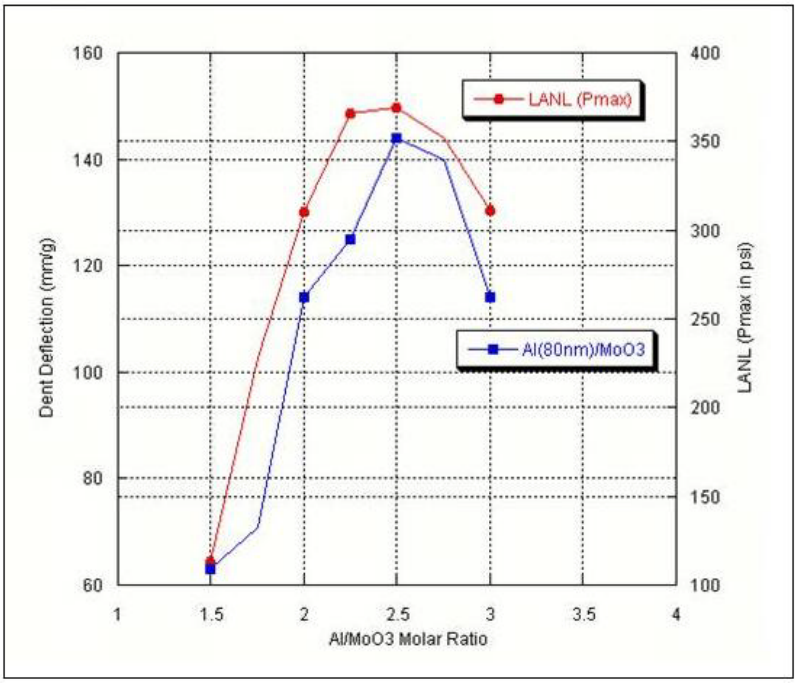


Figure 8 – Pressure Cell Versus Pan Dent Optimization

The Al (50 nm)/MoO_3 composite was optimized by the pan dent test. Optimum performance was observed at an Al/MoO_3 molar ratio of 3.15 as compared to 2.5 for the Al (80 nm) composite (Figure 9).

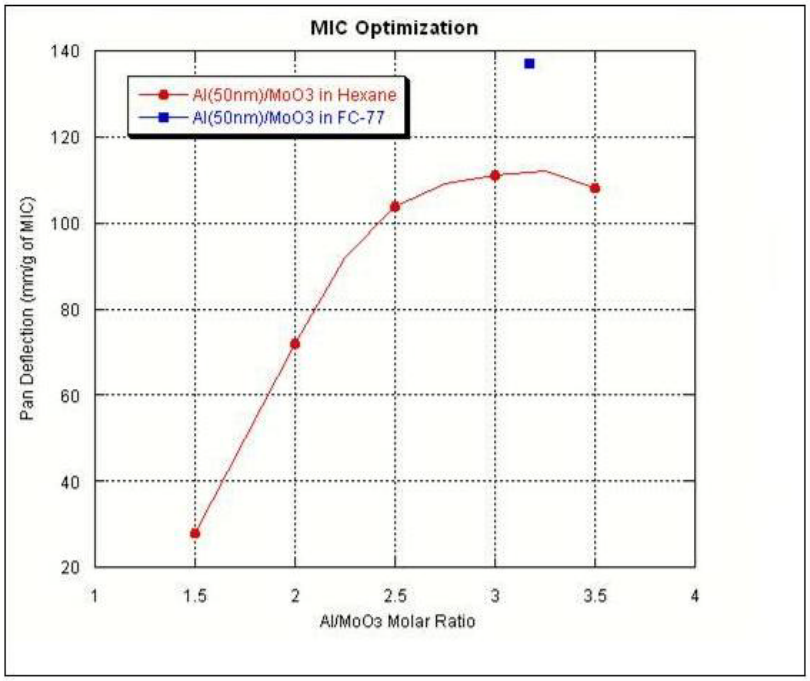


Figure 9 – Pan Dent Optimization of Al (50 nm)/MoO_3 Composite

The optimized Al (50 nm) composite gave a lower maximum deflection (112 mm/g) than the Al (80 nm) composite (144 mm/g). This was expected since the Al (50 nm) powder contained more dead weight Al_2O_3 than the Al (80 nm) powder. The use of the pan dent test led to an unexpected but beneficial result. The sonication of MIC powders in higher boiling solvents such as FC-77 and heptane gave higher performance material (14% performance improvement). The pan dent test also provided a tool to evaluate and compare MIC from water-based prepared MICs. As a baseline, the Al (80 nm)/Bi_2O_3 (320 nm) MIC formulation was prepared by sonication in heptane and characterized with the aluminum pan dent test. This MIC gave a deflection value of 513 mm/g.

ALTERNATIVE OXIDIZERS

Further efforts were made to evaluate an alternative oxidizer to MoO_3 for the purposes of improving action time results. The alternative oxidizers evaluated were Ag_2O, I_2O_5, WO_3, Bi_2O_3, and $NH_4N(NO_2)_2$.

$$2\ Al + 3\ Ag_2O \rightarrow Al_2O_3 + 3\ Ag \qquad \Delta H = -2.16\ kJ/g \tag{1}$$

Submicron AgO was prepared from silver nitrate and potassium hydroxide. The AgO was washed with distilled water, then acetone, and dried at 100°C for 8 hours. A composite was prepared (KTHW-23) by standard methods. The composite gave a mild pop on ignition. The nano Al/Ag_2O composite did not perform as well as the Al/MoO_3 system, and was not a good primer component. No further work was done on this system.

$$10\ Al + 3\ I_2O_5 \rightarrow 5\ Al_2O_3 + 3\ I_2 \qquad \Delta H = -6.21\ kJ/g \tag{2}$$

The energy content (-6.21 kJ/g) of the Al/I_2O_5 system versus Al/Bi_2O_3 (-3.01 kJ/g), and the fact that gaseous iodine is formed, made this system very attractive. This system had twice the energy content on a weight basis than the Al/Bi_2O_3 system and produces a gas with a boiling point of 184ºC, as compared to bismuth (1564°C). The Al (80 nm)/I_2O_5 composite was examined and found inferior. Ignition of this material gave a flash and purple smoke. The BET surface area of the commercial grade iodine pentaoxide was determined and found to have an average particle size of 26 microns. The I_2O_5 was ball-milled under hexane for 1 day. The BET gave an average particle size of 1.68 microns. A composite of Al (80 nm) and I_2O_5 (1.68 μ) was prepared by our standard technique. The ignition of this composite on an aluminum pan is shown in Figure 10.

Figure 10 – Ignition of I_2O_5 On Aluminum Pan

The purple cloud was gaseous iodine and the composite dented the aluminum pan. This system was very promising since the energy content was more than twice that of the Al/Bi_2O_3, it produced 33% more gas with a boiling point significantly lower than for bismuth. Gas generating additives such as BTATz may not be needed for small caliber and possibly large caliber ammunition. An additional application of this material could be as a countermeasure for chem/bio weapons (CBW). Iodine is sometimes used to kill bacteria in drinking water. Iodine is also chemically reactive and could be used to chemically degrade chemical weapons.

$$2\ Al + WO_3 \rightarrow Al_2O_3 + W \qquad \Delta H = -2.91\ kJ/g \tag{3}$$

Nanophase tungsten trioxide was acquired from LANL for Al/ WO_3 evaluation. The WO_3 was approximately 40 nm in diameter. A composite was made using 80 nm Al (KTHW-29). This composite gave a mild pop when ignited on an aluminum pan. Although LANL has been a strong proponent of this composite, it pales in performance comparison to the Al/Bi_2O_3 MIC discussed below. No further work was done on this system.

$$2\ Al\ (80nm) + Bi_2O_3\ (40nm) \rightarrow Al_2O_3 + 2\ Bi \tag{4}$$

Bismuth (III) oxide (40 nm) was obtained from Nanophase. This material enabled NAWCWD to make a true Al/Bi_2O_3 nano composite that was comparable to the Al/MoO_3 composite. However, this material exhibited extremely high ESD sensitivity. Currently, $Al(80nm)/Bi_2O_3$ (2 μ) composites used in primers by the Army have resulted in a number of accidental ignitions. The true nanocomposite is significantly more ESD sensitive. Normally, a Tesla coil will set off the $Al(80nm)/Bi_2O_3$ (2 μ) composite at a distance of 2 inches. The nanocomposite ignites when the Tesla coil gets within 10 to 12 inches. While the nanocomposite may give heightened performance, the severe safety hazard precludes its use in the field.

$$8\ Al + 3\ NH_4N(NO_2)_2 \rightarrow 4\ Al_2O_3 + 6\ N_2 + 6\ H_2 \qquad \Delta H = -8.52\ kJ/g \tag{5}$$

Ammonium dinitramide (ADN) and Al composites were made and ignition properties evaluated. The composite just burned rapidly, but this may be due to the ADN large particle size. The products of this system are nitrogen and hydrogen gas and a large amount of energy. If a source of nano ADN or 1 micron ADN can be found, the Al/ADN composite may be better than any material evaluated. Although ADN is not yet commercially available, it may be soon. It is being examined by a number of DOD sites for different applications. It is also a viable ammonium perchlorate (AP) replacement.

The investigation into alternative oxidizers was conducted specifically to mitigate risks associated with the primary molydenum trioxide LFEP configuration. Replacement of the current oxidizer would only be considered if major issues with production, lifecycle performance, or availability were to arise.

In June 2006, three LFEP formulations were prepared using M2451, M2453, and M2210D:

KTHW-71 – Al (M2453)/MoO_3(Cl)/BTATz/Kel-F/Carbon,
KTHW-73 – Al (M2451)/MoO_3(Cl)/BTATz/Kel-F/Carbon, and
KTHW-74 – Al (M2210D)/MoO_3(Cl)/BTATz/Kel-F/Carbon.

The sample M2210D was previously used in a primer formulation (KTHU-47) and fired in FY05 at room temperature with a good average all-up round action time of 2.90 $\pm$ 0.06 ms. However, primer testing after aging for 3 months gave very poor results. M2210D initially had an active Al content of 89% but within 2 months, it dropped to about 75% and stabilized at 74% after 1 year in air. All three primer formulations were prepared using the standard composition, 76% MIC, 20% BTATz, 2% Kel-F and 2% carbon. The fuel/oxidizer ratio was set at 2.5 (stoichiometric is 2:1 $2\ Al + MoO_3 \rightarrow Al_2O_3 + Mo$). The 2.5 fuel/oxidizer ratio was also selected to further investigate the impact on action time. Lower action times are expected at low fuel/oxidizer ratios but performance is often more dramatically affected by aging. NAWCWD has developed an alternative approach to mitigate the aging behavior of NovaCentrix's aluminum. A monolayer coating of Palmitic Acid or other hydrophobic coating inhibits the aging behavior. However, the effect of the coating on primer performance has not been determined. The low-temperature performance issue with the NovaCentrix 80-nm Al may be solved by the addition of a small amount (2 wt%) of 2-micron Al (H2) powder or exploded aluminum (ALEX). This will provide the large particles needed to help ignite the propellant bed more efficiently. In conventional primer systems, 25-micron $CaSi_2$ powder is added to enhance reliability, serving the same function. A pan dent-optimization of the MIC was prepared with the Al (80 nm) and Al (H2) to determine the optimal fuel/oxidizer ratio.

The bismuth (III) oxide formulation was tested and evaluated by the NAWCWD Code 4.7 Safety Committee, and the results were given to the NMC, who authorized its use. However, as discussed in the previous sections, bismuth (III) oxide exhibited extremely high sensitivity, and the primers used by the Army have resulted in a number of accidental ignitions. The severe safety hazard precluded its use in the field.

SAFETY CHARACTERIZATION

Since the inception of the program, safety has always been a concern. In 2002, safety tests were performed on BTATz, a gas generant. The MIC formulations containing BTATz indicated that the material continued to be extremely sensitive to ESD and friction. However, the extreme sensitivity of the BTATz-containing materials to ESD led the China Lake researchers to the conclusion that they needed to conduct specific human-body static energy tests to protect the technical personnel who would be working with the materials to fabricate and test primers and test AURs.

Archival static energy sensitivity data was found on the M52A3B1 primer and is shown in Table 8. To supplement this archival information, additional data was collected on standard M52A3B1 primers using the HBSES. This additional data is shown in Table 9. Following the collection of this baseline data on the currently operational electric primer, the BTATz primer configuration was tested to determine its sensitivity to electrostatic discharge. This data is shown

in Table 10. The lowest voltage at which the MIC/BTATz primers reacted was 2.5 kV, compared to 1 kV for the currently used M52A3B1 primer. Some primers from previous test configurations did not fire at even the highest 25-kV setting of the HBSES. Other data from previous tests were of limited value due to the test methodology.

Archival electrostatic data for M52A3B1 primers was plotted alongside comparable data for BTATz primers, as shown in Figure 11, which shows there was a greater safety margin for the BTATz primers at the lower voltages. The larger number of M52A3B1 primers tested and the larger scatter in their data meant that some of the primers were less sensitive than the primers tested with the MIC/BTATz mix. However, the worst case threat associated with normal handling was the sensitivity of primers at the lower voltages. These tests concluded that the BTATz mix was somewhat less electrostatically sensitive than the existing M52A3B1 primer.

Table 8 – Archival M52A3B1 Data

ARCHIVAL DATA AVERAGE RESISTANCE		**VOLTS, kV**							
Ohms		1	1.5	2	2.5	5	10	15	20
1.5K	Tests Fired					10	9	10	9
	% Fires					10%	44%	60%	88%
5.0K	Tests Fired					10	7	9	5
	% Fires					30%	43%	100%	100%
1.0M	Tests Fired	12	6	14	3	8	6		
	% Fires	33%	33%	43%	66%	100%	100%		
Note: Report TSD TS-1-E4-72, M. Skezula and A. Grinoch									

Table 9 – M52A3B1 Data

CASE #	VOLTS, kV						RESISTANCE (K OHMS)
	1	1.5	2	2.5	3	5	
100				Tried		Fired	12.86
101				Fired			18.20
102				Fired			6.00
103				Fired			24.47
104				Fired			17.80
105	Tried		Fired				27.50
106	Tried		Fired				10.60
107	Tried	TRIED	Fired				11.60
108	Tried	Tried	Tried	Tried	Fired		19.90

Notes: All production M52A3B1 primers.
All tests performed with no gap.

Table 10 – Experimental Primer Data

CASE#	VOLTS, kV			RESISTANCE (OHMS)	GAP
	2.5	5	7.5		
90		Fired		131.5	Yes
91		Fired		117.2	Yes
92		Tried	Fired	132.8	Yes
93		Fired		146.3	Yes
94	Tried	Tried	Fired	184.3	No
95	Tried	Fired		132.8	No
96	Tried	Tried	Fired	144.6	No
97	Tried	Tried	Fired	115.3	No
98	Tried	Fired		143.5	No
99	Tried	Fired		147.2	No

Note: KTHS-54 (BTATz) experimental primers.

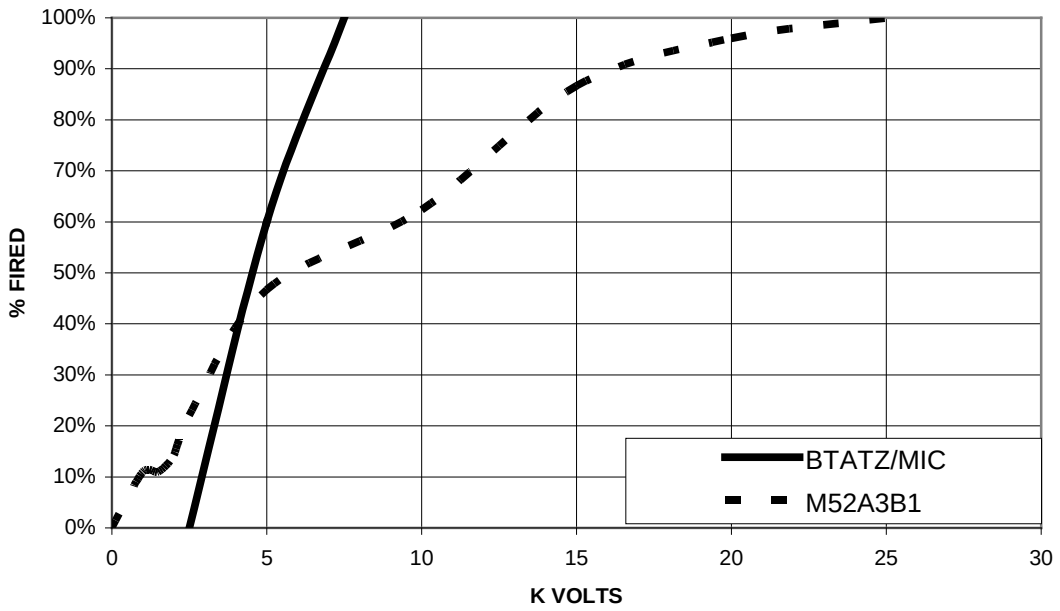


Figure 11 – Percentage of Primers Fired Versus Voltage

Handling Safety

With safety being a very important element of this program, a number of simple handling related evaluations were conducted. One of those evaluations involved the question of how MIC would behave when mixed with hexane and ignited. After applying a torch to a slurry mixture of MIC and hexane on an open surface, the solvent burned with a few sparks and then weakly popped when the solvent was almost completely consumed. A similar test was conducted where the slurry mixture of MIC and hexane was placed in a polyethylene tube. In that case, the hexane and polyethylene tube burned like a candle, and when the tube was almost completely consumed, the MIC primer material produced a weak pop. The burning solvent greatly decreases the reactivity of the MIC.

A series of safety related ignition tests were conducted on MIC alone, BTATz alone, and (3) MIC/BTATz mixtures in varying quantities. These various mixtures were placed in a polyethylene vial and then ignited using an electric match. Table 11 further describes test parameters and observed reactions.

Table 11 – MIC/BTATz Ignition Tests

Material Description	Reaction
MIC (50 milligrams)	Cap pops off – no damage
BTATz (50 milligrams)	Cap pops off – no damage
MIC/BTATz (50 milligrams)	Damages cap
MIC/BTATz (100 milligrams)	Disintegrates cap
MIC/BTATz (200 milligrams)	Disintegrates cap
MIC/BTATz (1000 milligrams)	Cap, bottom and tube fragmented

Ongoing efforts continued to optimize a primer formulation to desensitize the ESD sensitivity without affecting performance. Several incidents have occurred at various testing facilities involving MIC materials, raising safety concerns that could potentially hinder development progress. The sensitivity of the MIC materials was investigated and formulation sensitivity was mitigated through both additives to the formulation and training in handling and processing. ESD tests suggested that the LFEP primer sensitivity would be comparable to or lower than the M52A3B1 primer under proper handling and assembly conditions.

Another approach to mitigate this high ESD sensitivity of the Al/bismuth (III) oxide was to load the MIC in water. To desensitize the Al to water, Puszynski coated the Al with oleic acid. Puszynski claimed that there was no performance degradation even after exposure to water for 72 hours. This was based on ignition studies, not all-up round testing or even pressure-time traces. To evaluate Puszynski's claims, Al (80 nm) powder was coated with 5 wt% palmitic acid (Oleic acid without the unsaturation). This was achieved by sonicating the Al powder and palmitic acid in hexane. Aging and primer performance of this material were evaluated. Results from this effort are described in a subsequent section.

The strategy was to find additives that would mitigate the ESD sensitivity of nano Al powders and then use the best additives for energetic nano composite powders. One such additive was Kel-F (a registered trademark of 3M), which is a fluorocarbon-based polymer (PolyChloroTriFluoroEthylene). No longer produced by 3M, this material is available from sources in Japan. This material contains chemical energy and serves as both a binder and an energetic component of the primer mix. The weight percent of additive ranged from 2 to 67%. All materials were evaluated for high- and low-spark energy using a 5-kV power source and 0.02 µF (0.22 µF for KTHV-12 containing Kel-F) capacitor as shown in Table 12.

Table 12 – ESD Mitigation Experiments For Nano Al Powders

Name	Metal Powder	Composition	Wt% Additive	Energy (mJ)	
				>	<
-	Al (33 nm)	-	-		0.2
-	Al (50 nm)	-	-		0.4
-	Al (80 nm)	-	-		0.4
-	ALEX (~150 nm)	-	-		0.8
-	Al (208 nm)	-	-		0.1
-	Al (H5) – 5 m	-	-		2.5
BASELINE	Al (80 nm) M2210D	-	-	0.20	0.45
KTHU-71	Al (80 nm) M2210D	Sonication	-	0.63	0.901.2
-	Al (80 nm) M2210D	S (Coated)	10		0.9
KTHW-1	Al (80 nm) M2210D	S (Coated)	63.00	9.9	17.6
KTHW-19	Al (80 nm) M2210D	S (Coated using CS_2)	9.80	0.40	0.78
KTHW-7	Al (80 nm) M2210D	Teflon (T7A)	67.00	2	2.5
	Al (80 nm) M2210D	Kel-F (Coated)	10.00		0.6
KTHV-12	Al (80 nm) M2210D	Kel-F (Coated)	44.00	2130	
KTHW-17	Al (80 nm) M2210D	Kel-F	10.00	0.23	0.40
KTHW-16	Al (80 nm) M2210D	C (Dry mixed)	10.00	0.10	0.23
KTHW-15	Al (80 nm) M2210D	Bi	2.00	0.40	0.63
	Al (80 nm) M2210D	Palmitic Acid (Coated)	10		6.4
KTHW-13	Al (80 nm) M2210D	Palmitic Acid	15.00	1.25	1.80
TJF3-83	Al (80 nm) M2210D	$C_{14}F_{27}O_2H$	4.76	6.40	8.10

The 80-nm NTI Al powder exhibited very high ESD sensitivity, well below 1 mJ and could easily be ignited by an ungrounded person. The ESD sensitivity improved slightly when sonicated due to agglomeration of the Al particles. High loading of sulfur, Teflon, or Kel-F powders resulted in lowering the ESD sensitivity. In the case of Kel-F, the material could not be ignited even at > 2 J. The Kel-F is slightly soluble in hexane and coated the Al powder, yielding

better ESD mitigation. However, at 10 wt% Kel-F, the ESD sensitivity of the Al powder was improved but still below 1 mJ. The addition of conductive AB carbon failed to dissipate the charge and left the ESD property virtually unchanged. Coating the Al with Palmitic Acid or Perfluorosebacic acid gave the best results at low loading levels.

Based on the results, an approach of encapsulating the Al/MoO_3 MIC with Kel-F was investigated. This approach was also used to desensitize Al/bismuth (III) oxide MIC. Preliminary ESD testing using the ESD test apparatus set up in the laboratory indicated Kel-F did reduce the ESD sensitivity of the bismuth (III) oxide MIC. These preliminary results showed that the Al (80nm)/bismuth (III) Oxide (320nm)/Kel-F (2.5%) MIC was less sensitive than the Al (80 nm)/Bismuth (III) Oxide (1-micron Skylighter) MIC.

The ESD sensitivity data for MIC formulations is summarized in Table 13. The baseline Al (80 nm)/MoO_3 MIC (KTHW-22) exhibited a very high ESD sensitivity (0.16 μJ). Coating this formulation with palmitic acid did little to reduce the sensitivity. A coating of 10 wt% of Kel-F reduced the sensitivity up to 0.625 μJ, but this formulation could still be easily ignited by human-body ESD (HB = 1 mJ).

Table 13 – ESD Data for Nanocomposites & Coated Nanocomposites

Material	Composition	ESD (mJ)	Comments
Al (80 nm)/MoO_3 (Climax)	39/61	0.016	Baseline-
Al (80 nm)/MoO_3 (Climax)/Palmitic Acid	35/55/10	0.03	-
Al (80 nm) M2210D/MoO_3 (Palmitic Acid)	32.6/50.9/16.5	<0.025	-
Al (80 nm) M2210D/MoO_3 /Palmitic Acid)	26.5/41.5/32	0.10-0.225	-
Al (80 nm)/MoO_3 (Climax)/$C_{12}F_{23}O_2H$	35/55/10	0.36	-
Al (80 nm)/MoO_3 (Climax)/Kel-F	35/55/10	0.625-1.6	-
Al (50 nm)/MoO_3 (Climax)	45/55	0.1	100 W Horn
Al (80 nm)/S	37/63	1.6	Coated Using CS_2
Al (80 nm)/Teflon (1 μ)	33/67	2.5	Dry Mixed
Al (80 nm)/Bi_2O_3 (50 nm)	14/86	< 0.02	Extremely Sensitive
Al (80 nm)/Bi_2O_3 (320 nm)	15/85	< 0.02	-
Al (80 nm)/Bi_2O_3 (2.5 μm)	16/84	< 0.02	-
Al (80 nm)/Bi_2O_3 (<10 μm)	20/80	15.6	-
Al (80 nm)/$AgIO_3$ (235 nm)	79/21	0.22	-
Al (80 nm)/$AgIO_3$ (235 nm)/Kel-F	78/20/2	0.02	-
Al (80 nm)/$Cu(IO_3)_2 \cdot H_2O$ (800 nm)	71/29	2.50	-
Al (80 nm)/$Zn(IO_3)_2 \cdot H_2O$ (15 μm)	72/28	4.90	-
Al (80 nm)/$Bi(IO_3)_3$ (60 nm)	77/23	-	-

The ESD sensitivity of MIC powders is inherently controlled by the ESD sensitivity of the Al powder. While coatings appear to mitigate the ESD sensitivity of Al powders, they do not for the composites. The mitigation observed for the Al powders was probably the result of inhibiting the propagation of Al combustion. Under ESD no-fire testing conditions, some of the nano aluminum probably did ignite but did not provide sufficient energy to propagate combustion to neighboring Al particles. However, in the composites, ignition of nano Al particles resulted in

reaction with available oxidizers and rapid propagation/reaction. The approach of putting a thin protective coating on the MIC did not appear to be feasible for mitigating ESD sensitivity. The addition of conductive carbon particles to dissipate charge also failed.

The Al/MoO_3/BTATz/Kel-F primer formulation did not ignite at 60 mJ (2.5KV @ 0.02 μF). This suggested that the BTATz gas additive may also play an important role in reducing the ESD sensitivity. BTATz may act as an energy dissipation agent, reacting with the burning Al to produce nitrogen gas. BTATz is a high-nitrogen compound that has found applications in pyrotechnic gas generators and fire suppression systems. A composite of nano Al and ZnO (micron) also showed surprising ESD stability. This composite did not ignite at the maximum power level of 5KV @ 0.22 μF (> 2.75 J). This material was ESD safe.

Safety tests performed on the MIC formulations containing the gas generant, BTATz, indicated that the material continued to be extremely sensitive to ESD and friction. Although not necessarily a desirable characteristic, this tendency was not surprising or considered to be an irresolvable flaw, since the proposed MIC formulation is intended to replace an equally sensitive and dangerous lead-styphnate primary explosive.

Although the Al/Bi_2O_3 energetic nano composite outperformed the Al/MoO_3 nano composite, it suffered from extremely high ESD, friction and impact sensitivity. Even consolidation of the Al/Bi_2O_3 composite did not reduce the ESD sensitivity as it did for other nano composites. This made primers based on the Al/Bi_2O_3 treacherous to work with, and has led to a number of incidents. In 2006, two approaches were investigated to mitigate the problem. The first approach was to develop a new Al/oxidizer system that had similar performance as the Al/Bi_2O_3 nano composite. The second approach was to coat the composite with different binders that have different effects on the material properties.

Several new energetic nano composites were prepared based on iodate compounds. Composites of Al (80 nm) powder with different sized powders of $AgIO_3$ were prepared and evaluated. The composites were prepared in a 2:1 Al to $AgIO_3$ molar ratio and expected to react as described by equation 6:

$$4\ Al + 2\ AgIO_3 \rightarrow 2\ Al_2O_3 + 2\ Ag + I_2\ (gas) \qquad (6)$$

The $AgIO_3$ powders were commercially available in 400 and 35 micron sizes. Nano-sized $AgIO_3$ was prepared in-house by ball-milling the 400-μm powder for 4 days and determined to be 895 nm. Smaller $AgIO_3$ (235 nm) was prepared chemically by the reaction of $NaIO_3$ + $AgNO_3$. The Al (80 nm)/$AgIO_3$ (400 μm) composite (KTHW-53) exhibited very poor properties. It could not be ignited with a flame. The Al (80 nm)/$AgIO_3$ (5-37 μm) composite (KTHW-54) ignited with a noiseless flash and emitted a yellow smoke (AgI). The Al (80 nm)/$AgIO_3$ (895 nm) composite (KTHW-56) ignited with a loud pop. The Al (80 nm)/$AgIO_3$ (235 nm) composite (KTHW-46) was outstanding. A pan dent test using 18 mg of composite produced a hole in the aluminum pan slightly larger than that observed for the Al/Bi_2O_3 composite. The pan dent test gave a deflection of 405 mm/g for the non-optimized composite. The ESD testing of the composite exhibited a lower ESD sensitivity than either the Al/Bi_2O_3 or the Al/MoO_3. Two

weight percent of Kel-F was coated onto KTHW-46 to further reduce the ESD sensitivity. The results are reported in Table 13.

Composites of Al (80 nm) with $Cu(IO_3)_2 \cdot (H_2O)$ (800 nm) and $Zn(IO_3)_2 \cdot (H_2O)$ (20 μm) were also prepared based on equations 7 and 8, respectively:

$$14\ Al + 3\ Cu(IO_3)_2 \cdot (H_2O) \rightarrow 7\ Al_2O_3 + 3\ Cu + 3\ I_2\ (gas) \quad (7)$$

$$14\ Al + 3\ Zn(IO_3)_2 \cdot (H_2O) \rightarrow 7\ Al_2O_3 + 3\ Zn\ (gas) + 3\ I_2\ (gas) \quad (8)$$

Although the copper iodate composite made a loud pop when ignited, it made only a small dent in the aluminum pan. However, this composite exhibited relatively good ESD properties (Table 13). The zinc iodate composite only flashed. This may be due to the large oxidizer particle size rather than poor reaction chemistry. The ESD sensitivity was also relatively good (Table 13).

Composites of Al (80 nm) with nano $Bi(IO_3)_3$ (60 nm) were prepared based on equation 9:

$$12\ Al + 2\ Bi(IO_3)_3 \rightarrow 6\ Al_2O_3 + 2\ Bi\ (gas) + 3\ I_2\ (gas) \quad (9)$$

This material created big dents in the aluminum pans indicating poorer performance than the Bi_2O_3 system. However, modification of the mixing conditions gave very good results. The ESD sensitivity was also relatively good (Table 13).

Incidents during compaction of Al/Bi_2O_3 composite resulted in the barring of further work with this composite at NAWCWD until the high sensitivities could be mitigated. Additional incidents using this composite have been reported at NSWC-IH, ARDEC, and LANL, shutting down work at each of these sites. Friction was believed to be the most likely cause of the incidents. Primers based on Al/Bi_2O_3 are the Army's and NSWC-IH's primary lead-free composition and currently NAWCWD's backup formulation. The need for sensitivity mitigation was deemed to be a high priority and a number of coatings were investigated to reduce the Al/Bi_2O_3 composite ESD and friction sensitivity (Table 14). Both ceramic and steel plates were used on the BAM Friction Tester.

Table 14 – BAM Friction and Hammer-Impact Test Results

Coating	**ESD (mJ)**	**BAM Friction (Ceramic)**	**Hammer-Impact Test**
Xeon	> 0.121	Failed	Passed
Fluoroinert FC-40		Failed	
HexaphenylDiphenyl Ether	< 0.009	Failed	
Viton A	< 0.001	Failed	Passed
Baseline Al/Bi_2O_3 uncoated	< 0.001	Failed	Failed

The BAM Friction and Hammer-Impact Tests results were inconsistent. In the hammer test, the hammer was grounded to eliminate the ESD ignition of the samples. The Xeon- and Viton A-coated samples did not ignite under either impact or friction. In the BAM Friction Test, all samples ignited. One potential cause was the use of ceramic materials that can build charge due to the frictional forces. The charge may be responsible for the ignition observed in the BAM test. The ceramic parts were replaced with textured metal components or fine grit metal oxide sand paper with similar surface roughness to the ceramic components. The grounded metal components eliminated ESD ignition from the BAM friction test and gave better friction data for energetic nano composites.

The friction tester was also used for some primer formulations with the M52A3B1 formulation compared as a baseline. The results are shown in Table 15.

Table 15 – ESD and BAM Friction Test Results (MIC)

Mixture	ESD (mJ)	6/6 NF @	Low Fire Point	Conditions	Sample Size	Notes
PETN		42N		25%RH, 67ºF	~35mg	Burn Mark and NOx Generation
M-52 Primer Mix		4N		43%RH, 72ºF	~2 mg	Moderate snap, spark
Al (80 nm)/MoO_3(50 nm) BTATz/Kel-F (KTHW-36)	1.60	9N		43%RH, 72ºF	~2 mg	Moderate snap, spark
Al (80 nm)/MoO_3(50 nm) /Kel-F (KTHW-64)	1.60		0.05N	31%RH, 70ºF	~2 mg	Extremely loud snap, spark
Al (80 nm)/MoO_3(50 nm) (KTHW-63)	0.02		0.05N	31%RH, 70ºF	~2 mg	Extremely loud snap, spark
Al (80 nm)/Bi_2O_3(320 nm) (KTHW-42)	<0.02		0.05N	31%RH, 70ºF	~2 mg	Extremely loud snap, spark
Al (80 nm)/$AgIO_3$(300 nm) /Kel-F (KTHW-50)	0.02		0.05N	34%RH, 70ºF	~2 mg	Extremely loud snap, spark
Al (80 nm)/$AgIO_3$(300 nm) (KTHW-46)	0.22		0.05N	34%RH, 70ºF	~2 mg	Extremely loud snap, spark

PRODUCTION METHODS

Mixing

In 2004 primer-mixing processes were investigated with the objective of reducing the hazards associated with material preparation. In particular, the use of ultrasonic mixing of the BTATz and MIC was investigated. Pre-sonification of the BTATz resulted in two batches of MIC primer material that were dead and would not function. In contrast with that, samples of BTATz and MIC that were sonicated together reacted in a normal way, capable of being initiated by a spark but gave slightly longer action times when tested in AURs. Samples that used hexane mixing of the MIC and BTATz gave the best performance with respect to AUR action times. This work and the laboratory performance of the materials are reported in Table 16.

Table 16 – MIC Formulations and Results of Mixing Experiments

Alternate MIC Formulations	Laboratory Performance Results
BTATz (not grounded or sieved) was dispersed in hexane for 1 minute using the ultrasonic horn. MIC was added and sonicated for 4 minutes. PEG was added and sonicated for 1 minute. Product was dried and carbon added	Material showed very poor performance. Pre-sonification of the BTATz had detrimental effects on the final composite. Testing indicated that this material showed little promise. (Pre-sonification of BTATz was thought to be the cause of a lot of these problems.)
BTATz (not grounded or sieved) was dry mixed with MIC. The mixture of BTATz and MIC was sonicated for 1 minute, filtered and dried.	The material gave a loud response when initiated, again confirming the suspicion that pre-sonification of the BTATz degrades performance.
Mixture of BTATz and MIC were sonicated, filtered, and dried. Added PEG in hexane and dried under nitrogen flow. Added carbon. End-product designated KTHS-92 and used in subsequent AUR test firings.	No laboratory tests to report on this particular mix.
Mixture of BTATz and MIC were sonicated, filtered, and dried. Added Kel-F in hexane and dried under nitrogen flow. Added AB. End-product designated KTHS-93 and used in subsequent AUR test firings	No laboratory tests to report on this particular mix. Samples KTHS-92 and KTHS-93 were made to compare performance using two different binders, Kel-F and PEG.
The MIC (sonicated for 4 minutes) was mixed with BTATz in hexane, coated with Kel-F, and AB was added.	Initiation exhibited good loud, sharp pops.
Scaled up to prepare MIC samples with 2%, 4%, and 6% AB for low-temperature AUR test firings.	No laboratory tests to report on this particular mix.

The China Lake Chemistry Division performed a short series of qualitative laboratory tests. The objective of these tests was to investigate the effects of alternative material preparation (mixing) techniques. These tests were performed using Technanogy MIC that incorporated 50-nm Al as its fuel component. Three mixing methods were evaluated as part of this relatively simple series of tests: (1) dry mixing, (2) ultrasonic bath (one hour), and (3) ultrasonic horn (two minutes). Performance was measured by sound emission when initiated by an electric spark. It was observed that the dry-mix product gave a weak pop and the two ultrasonic mixing methods resulted in a product that produced a loud pop. The results of several different combinations of fuels and oxidizers that were prepared and qualitatively tested are shown in Table 17 below.

Table 17 – Comparison of Mixing Techniques with Alternative Fuels and Oxidizers

Primer Mix Constituents	Performance/Reaction
Dry mix of Al (50 nm) + Kel-F in a stoichiometric ratio	Material burned but did not pop when initiated.
Dry mix of Al (50 nm) + $Zr(WO_4)_2$	Poor performance, possibly due to large oxidizer particle size. Required high temperatures for initiation.
Ultrasonic horn mix of 2 Al (Flake - 0.25 micron X 25.0 micron) + MoO_3	Moderate pop when initiated.
Ultrasonic horn mix of 3 Mg (25-30 micron) + MoO_3	Loud pop when heated in a test tube. Did not ignite using a spark.
Ultrasonic horn mix of 2 Al (50 nm) + 3 ZnO (>5 micron)	Ignite in a flash but did not pop when initiated with spark. Reaction generates Zn in vapor phase, which would contribute to gas pressure. Smaller particle size holds promise.
Ultrasonic horn mix of 8 Al (50 nm) + 3 Ag_2MoO_4	Easily initiated and produced a loud pop comparable to conventional MIC.
Ultrasonic horn mix of 10 Al (50 nm) + 3 Sb_2O_5	Produced a moderate pop.
Ultrasonic horn mix of 10 Al (50 nm) + 3 I_2O_5	Easily ignited but did not pop when initiated.
Ultrasonic horn mix of 3 Al (50 nm) + Bi_2O_3 (10 Micron)	Reaction should be exothermic, but does not react when exposed to spark or flame. High boron-oxygen bond strength creates a large activation barrier.
Ultrasonic horn mix of 3 Mg (>5 micron) + Bi_2O_3 (Sub-Micron)	Ignition and combustion behavior almost identical to Mg alone. Did not appear that B_2O_3 was involved in combustion.
Ultrasonic horn mix of 2 Al (35 nm and 50 nm) + Bi_2O_3 (10 micron) in i-PrOH	Solids mixed for 3 minutes using ultrasonic horn. Material popped when initiated but audio report is not as sharp nor as loud as the conventional MIC material. Solvents used in mixing study were hexane and iso-propanol. In both cases the Al powder separated from the MoO_3. Very poor mixing occurred. LANL used 2 micron material and achieved good results. The critical limit may be somewhere around 2 microns.

Solvents were investigated to improve mix homogeneity. A mixing study was initiated at NAWCWD in 2005 using different size Al (33 nm, 50 nm, 80 nm, 208 nm) and different sizes of oxidizer, bismuth oxide (320 nm, 1 micron, and 10-micron) and molybdenum trioxide (40 nm and 1.6 mm), using two different solvents (hexane and iso-propanol). The results are shown in Table 18.

Table 18 – Al/Oxidizer Material Mixing

	Molybdenum Trioxide MoO_3			
Aluminum Powder	Climax 40 nm		Aldrich 1.6 mm	
	Hexane	i-PrOH	Hexane	i-PrOH
Al (35 nm)	Good			
Al (50 nm)	Good		Good	
Al (80 nm)	Good		Good	

	Bismuth Trioxide Bi_2O_3					
Aluminum Powder	Aldrich 320 nm		Skylighter 1-3 μm		Sigma-Aldrich < 10 μm	
	Hexane	i-PrOH	Hexane	i-PrOH	Hexane	i-PrOH
Al (35 nm)				Poor		
Al (50 nm)	Good	Poor		Poor	Good	Very Poor
Al (80 nm)	Good	Poor	Very Good	Good	Good	
Al (208 nm)					Good	
Al (5 μm)	Good					

Poor – Material Separation (Al from Oxidizer) Was Observed
Good – Little or No Material Separation
Very Good – No Material Separation, Rapid Precipitation, Easy Isolation (Filtration)

The use of iso-propanol produced a good MIC in only one case, Al (80nm) plus bismuth oxide (Skylighter). In all other samples, the dense bismuth oxide separated from the Al in solution and settled at the bottom of the vial. Attempts to filter these samples resulted in isolation of the suspended Al in the filtrate and bismuth oxide on the filter paper.

Ultra-sonication in hexane provided a dramatically different result. In all cases, agglomeration was clearly evident where the MIC homogeneously settled out of the hexane. The product was easily isolated by filtration. The samples prepared in hexane were more homogeneous than that prepared in iso-propanol and showed good performance. Sonication in hexane gave well-mixed products regardless of Al, Molybdenum trioxide or bismuth oxide particle size.

Sonication in iso-propanol gave heterogeneous products except for the Al (80 nm)/ Bi_2O_3(Skylighter) sample. In contrast, all MIC samples prepared in hexane appeared homogeneous and gave good performance. The primer performance is also very dependent on preparation conditions. Primer KTHU-22 was sent to LANL for pressure-time experiments since, LANL had data indicating poor performance for BTATz-based primers. The LANL data was inconsistent with AUR test results obtained at China Lake. KTHU-22 was tested and compared to similar formulations prepared at Los Alamos. Plots of pressure versus percentage of gas additive and pressurization rate versus gas additive are shown in Figures 12 and 13.

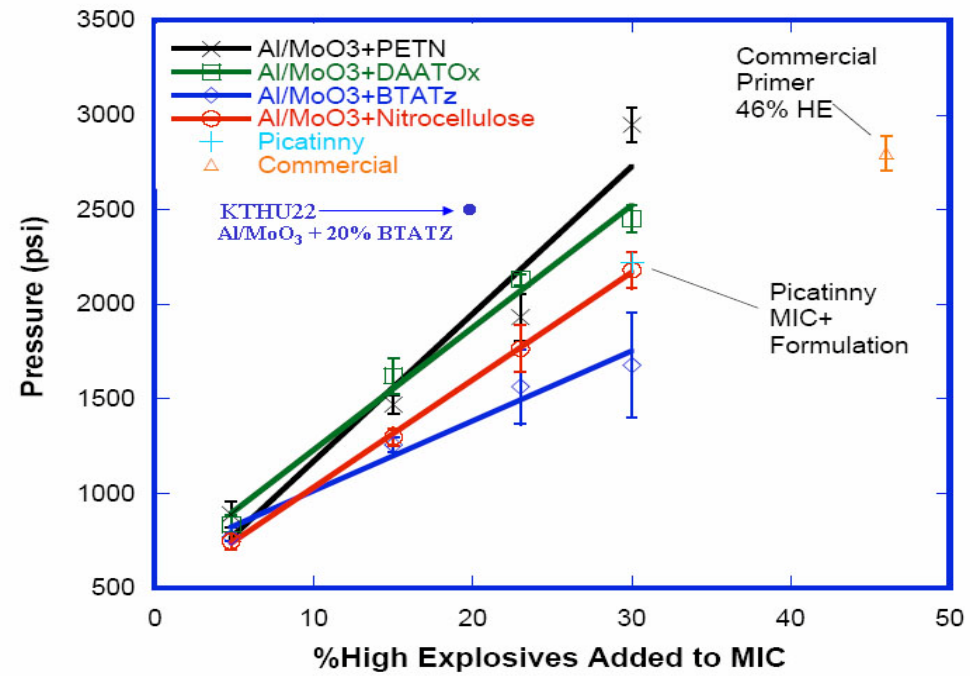


Figure 12 – Maximum Pressure Versus Gas Additive

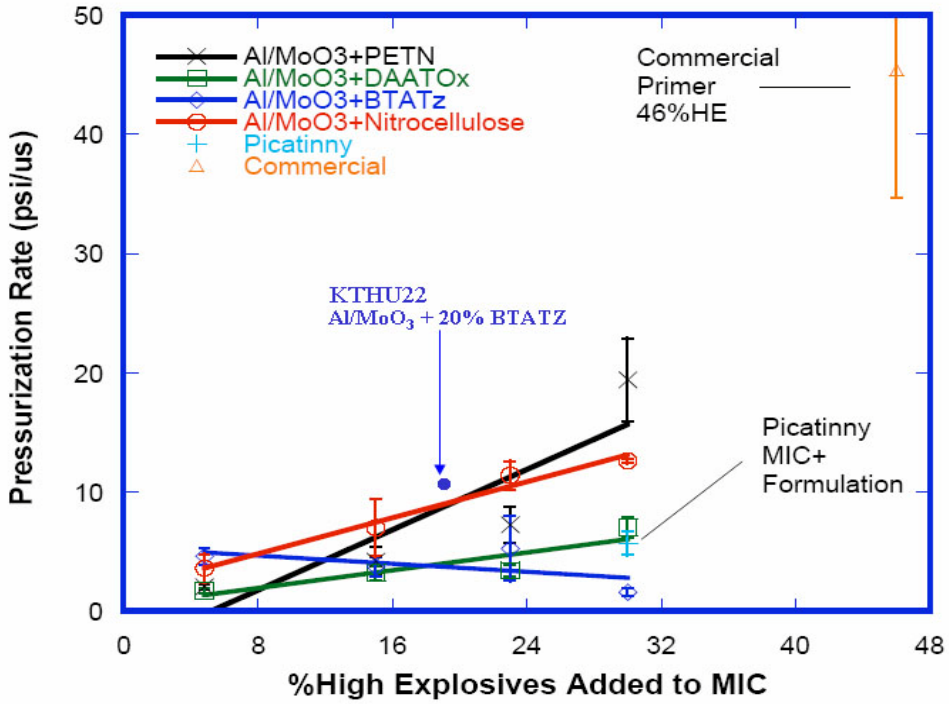


Figure 13 – Pressurization Rate Versus Gas Additive

The blue lines in both plots are the results of the LANL formulations using BTATz. KTHU-22 performed significantly better than analogous materials made by LANL. At the 20% gas additive level, it out performed every other gas additive tested. The difference in performance level was presumably due to slight difference in mixing conditions between NAWCWD and LANL. Additional work is needed to determine if NAWCWD mixing conditions will show further improvements for other gas additives.

The addition of the 400-W Branson 450 Digital Sonifier (horn) dramatically improved formulation mixing and development capability at NAWCWD. Ultrasonic mixing was critical to maintaining homogeneity in the MIC and minute variances can affect the AUR performance.

Improvements have been made in the primer formulation and production process to optimize the performance and reduce handling during hazardous steps of the procedure. The improved formulation process is a progression over the previous process but still involves several hazardous steps. However it does reduce the number of steps as well as eliminate one very hazardous step (grinding dry MIC powders). Both the “old” and “improved” procedures are summarized below:

Old Process
1. Sonicate Al/oxidizer in hexane
2. Filter
3. Vacuum dry MIC
4. Lightly grind MIC (mortar and pestle)
5. Add hexane
6. Add BTATz
7. Magnetic stirring (20 min.)
8. Add Kel-F in hot hexane while stirring
9. Evaporate off hexane
10. Break up product with spatula
11. Vacuum dry
12. Add carbon and dry mix (shaking for 1 min.)

Improved Process
1. Sonicate Al/Oxidizer in hexane
2. Add BTATz
3. Magnetic stirbar mixing
4. Add Kel-F in hot hexane while stirring
5. Evaporate off hexane
6. Break up product with spatula
7. Vacuum dry
8. Add carbon and dry mix (shaking for 1 min.)

A series of MIC samples were prepared under different conditions, with mix time and ultrasonic amplitude being varied. When the different materials reacted, there were discernible differences in audio report. Different MIC samples were prepared for consolidation into primers to support further AUR test and evaluation. Samples KTHU-22, KTHU-47, KTHU-53, KTHU-57, and

KTHU-62 have been prepared using the improved process. Table 19 shows the different mixing schedules and component make-up by weight percent. Evidence showed that slight variances in the formulation process affected the output performance of the mix and thus the action time of the AUR. The impact on the AUR action time gave investigators a better idea as to how critical the material processing conditions are and has helped to define optimal mixing processes. The AUR results are discussed in a later section.

Table 19 – Samples Prepared Using Different Mix Times and Amplitudes

Name	Al Powder	Size (nm)	Oxidizer	Size (nm)	MIC (%)	BTATz (%)	Kel-F (%)	C (%)	Mix Amp (%)	Time (Min)
KTHU-22	Technanogy	50	Moly	50	75.9	20.0	2.0	2.1	75	2
KTHU-47	NTI	80	Moly	40	75.9	20.1	2.0	2.0	75	2
KTHU-53	Technanogy	50	Moly	40	76.0	20.0	2.0	2.0	75	1
KTHU-57	Technanogy	50	Moly	40	75.9	20.1	2.0	2.0	75	0.5
KTHU-62	NTI	80	Bismuth	1K	75.9	19.9	2.1	2.1	75	1

The MIC preparation process was slightly modified to investigate further improvements in the mixing process to enhance LFEP primer performance. The change involved the sonication of the oxidizer powder prior to sonic mixing with the nano aluminum powder. A significant enhancement was observed using the pan dent test. The original Al/MoO_3 MIC powder (50 mg) ignited on an aluminum pan exhibited little effect on the pan (no dent). With the new mixing procedure, 17 mg of MIC produced a significant dent in the pan. This was indicative of a significant increase in reaction rate, probably due to better mixing. This improvement was used on subsequent primer formulations and produced lower AUR action times.

Loading

Since the physical integrity of the primer mix could have an effect on the electrical properties of the primer mix, fabrication/assembly processes were studied as potentially important factors in the production of electric primers. It was noted that consolidation pressure and dwell time, along with the use of an appropriate binder, appeared to be an important factor in achieving a reliably functioning primer. Preliminary studies were conducted to evaluate the effect of consolidation pressure on the performance of the MIC-based primer mix. This investigation led to the conclusion that pressing the mix at a pressure of 8,000 psi produced optimum characteristics and performance.

Efforts were made to find a solvent to facilitate handling of the MIC-based primer mix in a production environment. There were some considerations given to how to consolidate a wet slurry mix into a primer cup. Two potential methodologies were evaluated.

Solvent-Based Loading

The first loading procedure involved the use of a solvent such as hexane, heptane, or fluorocarbon. As part of a simple safety-related experiment, a primer paste was produced using

1:1 ratios of MIC-based primer mix and hexane. The paste was then placed behind a shield in a hood and ignited with a flame. The hexane burned for almost a minute before the MIC produced a single mild popping sound. At the time of the pop, the flame was extinguished, presumably because of the nitrogen gas that was produced from the reaction of the BTATz component of the primer mix.

In the follow-up loading experiment, approximately 130 mg of primer mix and 130 mg of hexane solvent were mixed to produce a thick paste. An electric primer cup was filled to the rim with approximately 200 mg of this paste using a Teflon coated spatula. The primer cup was then set aside for a week to allow the primer mix to dry in air. After this weeklong period, there was no readily discernible shrinkage of the paste in the cup even though the hexane component of the paste had evaporated, leaving only 100 mg of primer material in the cup. It was anticipated that approximately 150 mg, or 50% more primer mix, would be required to reliably initiate the propellant bed in a 20-mm cartridge case. A consolidation by pressing, followed by the addition of more paste and a second consolidation process was required to place a sufficient amount of primer mix into the primer cup. This could not be accomplished in the China Lake Chemistry Division facility due to safety restrictions. The double consolidation process was accomplished in the China Lake Pilot Plant at a later date using custom built hardware and an approved operating procedure described below.

The solvent-based loading procedure for wet-loading primers was written based on previous analytical and empirical work. It was submitted to the local NMC and the NAWCWD Code 47 Safety Committee and approved in January of 2005. This wet-loading procedure involves the incremental pressing of a solvent-based paste using special tooling, which allows a sufficient amount of material to be loaded into the primer in a minimum number of steps. This loading procedure is conducted in an explosives pressing room and is performed in accordance with the Standard Operating Procedure (SOP) entitled: “Pressing and Weighing of Experimental Energetic Materials” (SOP 10090-03). This procedure has facilitated the performance testing of various MIC primer mixes and is expected to help address issues related to large-scale processing and to eventually lead to a method of producing LFEPs in a production environment.

Figure 14 illustrates one of the pressing operations utilized in the assembly process. Due to the potentially hazardous nature of the MIC materials, only a limited number of personnel were authorized to handle and test the primers in specific facilities.

Figure 14 – MIC Pressing Operation

The tooling required to assemble the test primers was designed and built at China Lake. Photographs of the various rams and dies are shown in Figure 15.

Figure 15 – Rams and Dies Used to Load MIC Electric Primers

The following step-by-step description provides more detail on the proposed primer wet-loading procedure.

1. Pour hexane into a vial containing approximately 150 mg of MIC materials and mix until a thick slurry consistency is reached. Each batch is a bit different so it will take trial and error to determine the exact mixture.

2. Transfer the MIC slurry from the vial to the loading die containing the 20 mm primer cup.

3. Install the pressing ram and consolidate at 300 psi.

4. Remove the loaded primer cup from the die and place on a drying tray.

5. Repeat steps 1-4 until the desired number of 20 mm cups is reached.

6. Dry primers in the explosives drying oven at 120ºF for 4 hours.

7. Reinstall primers in the die and press at 8,000 psi.

8. Remove the 20 mm cup from the die.

Puszynski Method

The second loading procedure involved the use of Puszynski's water-loading method. In 2004, NAWCWD collaborated with LANL to evaluate Puszynski's water loading method. It was determined that there were at least three major issues associated with the water loading approach. The first and most obvious factor is the reactivity of the nano aluminum with water. In order to resolve this problem, the nano aluminum was treated with methyltrichlorosilane (CH_3SiCl_3) to place a protective coating on the Al. Samples of untreated and silane coated Al were placed in vials of water to compare their reactivity. The untreated Al began to react with the water almost immediately, evolving hydrogen gas. Within 24 hours, the untreated Al powder had completely reacted. In contrast, after 24 hours there was no evidence of reaction between the silane coated Al and the water. However, after 4 days about 25% of the Al had reacted with the water. Previous surface coating experiments at China Lake have shown that a second silane coating treatment will further enhance the effectiveness of the protective layer.

The second issue associated with the use of water as a solvent involved the necessity to heat treat the MoO_3 to protect it against exposure to liquid water.

The third and final known factor to consider was the overall performance of the LFEP primer. It was possible that the silane-modified Al surface would adversely affect the action time of the primer by creating a barrier between the Al and the oxidizer (MoO_3). To determine the overall impact of this factor, primers were produced using this water-processed primer material and then test-fired to measure the resulting AUR action times. These heat-treating, silane-coating, and AUR firing experiments continued into the following years of the program to determine if the MIC could be safely loaded using water as a solvent to produce high performance LFEPs. After further research, material used in the water loading method, exhibited very low performance. The most likely cause of the low performance was the dissolution of the MoO_3 and subsequent hydrolysis of the nano aluminum. Based on these results, it was strongly believed that hexane, heptane, or a fluorocarbon solvent could be used effectively and safely in the mixing and pressing processes.

In 2006, the water loading method was revisited and was tested on a one-tenth scale and on a full-scale using Puszynski's method. A modified Puszynski method was also evaluated at quarter scale where the material was poured into a petri dish and dried under a flow of air. The results are shown in Table 20.

Table 20 – Modified Water Loading Method Results

Al (80nm)/Bi_2O_3(320nm) (Al/Bi_2O_3 ratio = 2.27)	
Puszynski's Water Loading	Deflection (mm/g)
Y15 1/10th Scale Synthesis	97
W77 Quarter Scale	0
W77A Quarter Scale (Sonicated)	299
Y18 Full Scale	348
Y17 Baseline	513

In both cases using Puszynski's water loading method in one-tenth (0.42g) and full scale (4.2g), MICs did not perform as well as MIC with the same composition prepared by solvent-loading methods. The full-scale run gave a much better deflection result (348 mm/g) than the $1/10^{th}$ scale (97 mm/g). The poorer result for the one-tenth scale was probably due the small size and measuring inaccuracies. The full scale run was also dried for 3 days at 50°C to ensure dryness but still showed a 30% loss in performance (The performance is still significantly better than the best Al (80 nm)/MoO_3 of 144 mm/g). The cause for the loss of performance has not been identified but may be due to phase separation of the ingredients during the drying stage or a small amount of hydrolysis (the MIC powder was harder than usual). In the quarter-scale test, the MIC/water slurry was poured on a 6-inch Petri dish and dried under a flow of nitrogen for 1 hour and vacuum dried for 1 day. The Al and Bi_2O_3 clearly separated as is seen in Figure 16.

Top View (Gray = Al) Bottom View (Yellow = Bi_2O_3)

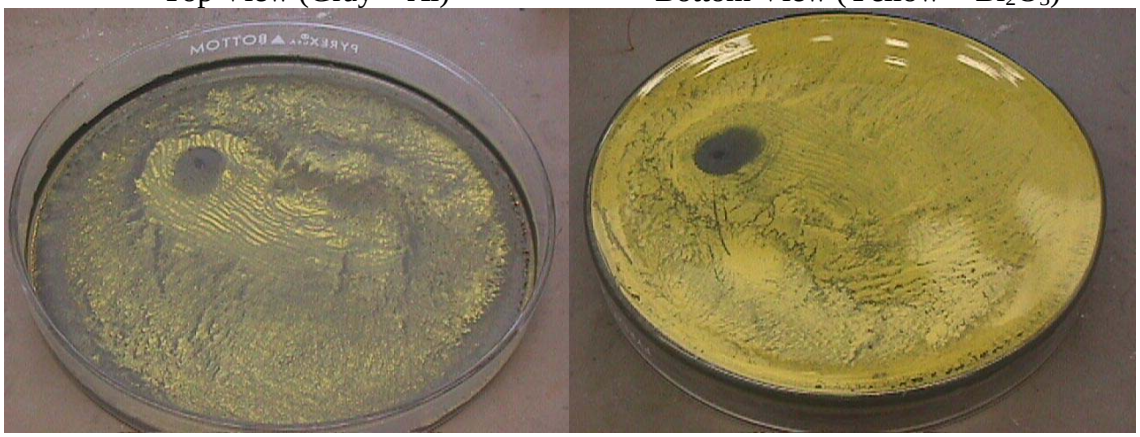


Figure 16 – Separated Al and Bi_2O_3

A well-mixed composite is usually green in color. The separation led to very poorly performing material that gave a very weak audio report on ignition and did not dent the aluminum pan (0 mm/g deflection). The solid was collected and sonicated in 25 ml of hexane at 75% amplitude with a 0.5 second pulse for 2 minutes (standard conditions). The aluminum pan dent test was performed and gave a deflection of 299 mm/g which is similar to the full-scale result of 348 mm/g. This data suggested that hydrolysis of Al may be involved in the water-loading process.

Puszynski's water-loading method was also used with the Al (80 nm)/MoO_3 composite. The composite was also dried for 3 days at 50°C to ensure dryness but appeared to be bluish-gray in appearance instead of gray. The material also weighed 30% higher than expected indicating hydrolysis of the Al. The material exhibited very low performance emitting a few sparks and no audio report. It gave no deflection in the pan test versus 144 mm/g using the hexane-prepared material. Although the cause is not known, the most likely cause is the dissolution of the MoO_3 and subsequent catalytic hydrolysis of the nano Al. The blue color comes from the suboxide of MoO_{3-x} and the higher than expected weight of the product from hydrolysis of the Al forming Al $(OH)_3 \cdot (H_2O)_y$.

Clearly, this method does not work for dilute systems. In the method used by the South Dakota School of Mines, only 0.5 ml of water was used to form a viscous slurry. The use of the ultrasonic horn is not possible for such small volumes. Material segregation probably occurs but at a slower rate.

Water loading of MIC primers will continue to be investigated as it is deemed an important aspect of primer processing. However, it is strongly believed that a heptane or fluorocarbon solvent can be effectively and safely substitute in the mixing and pressing processes with nominal impact to a production line.

Solvent-Based Method Optimized

NAWCWD investigators followed the procedures outlined previously and determined that the proper mix ratios to produce a primer having the correct final height was 160 mg of MIC-based primer mix and 0.4 cc of hexane. The MIC primer mix and hexane were mixed in an aluminum tray instead of a glass vial for ease of mixing. This approach provided an additional safety factor by being able to electrically ground the mixing container and avoided the use of glass containers. The resulting MIC slurry was loaded into the die very quickly and observed to dry rapidly. After allowing the mix to air dry in the primer cup for a few minutes, it was pressed at 300 psi. The cups were removed from the die and then oven dried for 4 hours at 120ºF. The cups were placed back into the die and consolidated at 8,000 psi. Investigators attempted to load a set of primers during some unexpected rain showers when the humidity was above 60% and found that the material would not dry in the cup properly.

Under the new method for loading and pressing, the investigators are able to load approximately 8 primers with 2 grams of material depending on the formulation. The investigators use a single die and mandrel set to press the primers. Approximately 20 primers can be pressed each day with the current loading hardware and drying procedures. Investigators have begun the design and fabrication of a multiple-primer die to load several primers at one time and to dry them in the die to reduce the amount of handling of the primers and the production output. The die will allow for higher production quantity when a formulation has been down selected and being statistically validated.

Production Processes

In conjunction with the improvements to loading hardware, extensive research is being done into the manufacturing processes involved in M52A3B1 primers including discussions with the Army's Lake City Army Ammunition Plant (LCAAP). The goal is to incorporate loading techniques and processes that are currently being used in industry and develop a strategy for mass production of Lead-Free Electric Primers.

Navy investigators traveled to Lake City during 2002. The LCAAP is a government owned/contractor operated facility that is currently being operated by the Alliant Techsystems Corporation (ATK). The purpose of this trip was to meet with LCAAP/ATK representatives and describe the objectives/status of the LFEP program as well as tour the 20-mm M52A3B1 electric-primer production line. The production-line tour was considered to be especially important, since future production of medium caliber electric primers could remain in place at LCAAP and inspection of the current production methods of electric primers was expected to be helpful in addressing suitable methods for the production of LFEPs using MIC-based materials. Contact with LCAAP/ATK personnel has continued, with discussions focused on possible approaches for the production of LFEPs based on MIC formulations.

A Canadian company, SNC (now General Dynamic Ordnance and Tactical Systems (GDOTS), Canada), was granted a license in 2002 to use the Navy's patented MIC technology in the production of lead-free primers for 20-mm ammunition. SNC also expressed an interest in the establishment of a cooperative research and development agreement (CRADA) with the Navy. Although closure on this CRADA was never achieved, the company has since been engaged as a potential partner for the LFEP Program.

AUR RESULTS

There have been opportunities during past years to test fire LFEPs of several different configurations, the principal differences being the composition of the mix and how the primer material was processed prior to and during consolidation into the primer cup. The following paragraphs describe the most significant AUR test efforts.

China Lake Chemistry Division personnel produced a number of different primer mix formulations during the 2004 calendar year. The compositions of these formulations, along with some of the key primer fabrication details, are shown in Table 21. Additional formulation data can be found in Appendix B.

Table 21 – Alternate MIC Formulations Investigated During FY04

Formulation Identity	MIC Source	MIC (Wt %)	BTATz (Wt %)	Kel-F (Wt %)	AB Carbon (Wt %)	Primer Mix Weight (mg)	Consol Pressure (psi)	Dwell Time (Sec)
KTHS-43	Technanogy	75.88	19.91	2.09	2.12	130	8,000	5
KTHS-44	Technanogy	84.80	10.34	2.11	2.75	130	8,000	5
KTHS-56	Technanogy	63.46	29.98	2.05	4.51	130	8,000	5
KTHS-74	NSWC - IH	80.56	15.48	1.93	2.03	150	8,000	5
KTHS-75	NSWC - IH	70.79	25.38	1.85	1.98	150	8,000	5
KTHS-90	Technanogy	67.26	29.2	1.93	1.60	130	8,000	5
KTHU-3	CL – 100W	75.86	20.10	2.02	2.02	130	8,000	5
KTHU-4	CL – 100W	74.41	19.72	1.97	3.91	130	8,000	5
KTHU-5	CL – 100W	72.62	19.24	1.92	6.21	130	8,000	5

Additional details on the parameters used in the fabrication of the AURs are included in Appendix C.

A number of AURs were fabricated using LFEPs that were produced from the formulations noted above. Theses AURs were test-fired to evaluate the performance of the different compositions, including the ones containing BTATz and Kel-F. Table 22 summarizes the data collected from these tests. (Note: Test values are averages of more than one test firing. Individual primer firing test results can be found in Appendix D.)

Table 22 – Phase III AUR Firing Test Data Summary

Formulation Identity	Initial Temperature (ºF) of Primer	Estimated Primer Test Temperature (ºF)	Action Time (msec)	Chamber Pressure (ksi)	Velocity (fps)
KTHS-43	Ambient	Ambient	2.98	Unk	3322
KTHS-44	Ambient	Ambient	3.64	Unk	3320
KTHS-56	Ambient	Ambient	4.15	Unk	3321
KTHS-74	Ambient	Ambient	3.04	Unk	3283
KTHS-75	Ambient	Ambient	2.96	62.7	3276
KTHS-90	Ambient	Ambient	4.14	61.9	3380
KTHS-90	-65	Unk	25.12	Unk	3339
KTHU-3	-65	-10.0 to -14.5	3.65	51.6	3295
KTHU-4	-65	-9.0 to -20.0	3.63	53.3	3272
KTHU-5	-65	-10.0 to -14.5	5.91	51.1	3279

As noted above, low-temperature firing tests were performed to evaluate the performance of the LFEP design concept at temperatures as low as -65ºF, the temperature requirement that is

called out in the typical medium caliber ammunition specification. The initial approach was to pre-condition the ammunition in an environmental chamber to get its temperature down to the requisite -65ºF. However, it was first speculated and then confirmed that the cartridge case and the primer were gaining an appreciable amount of heat through contact with the relatively ambient breech of the Mann barrel prior to its being fired. It was estimated that the temperature of the primer at the time of firing could have increased to as much as 0ºF. This observation was shared with others in the DOD gun/ammunition community and it was learned that this same effect had been observed at other test facilities. Although there was no documented method to overcome this situation in a normal test environment, the Navy investigators were led to the decision that they would attempt to overcome the effects of this heat load and keep the primer closer to its pre-conditioned -65ºF temperature prior to its being fired.

The selected approach was to reduce the pre-fired temperature of the Mann barrel and breech cap to prevent a high rate of heat transfer from them to the round of ammunition being tested. The first step was to wrap half-inch diameter copper tubing around the most rearward 12 inches of the breech end of the Mann barrel. Insulation was placed around the copper tubing to reduce the influx of heat from the surrounding environment. A conventional ice chest was used to hold a supply of dry ice. An Al reservoir with inlet and outlet fittings and an overflow relief valve was placed in the ice chest along with an abundant amount of dry ice. Appropriate plastic hoses were attached to the inlet and outlet fittings of the reservoir. The other ends of these hoses were attached to the copper tubing that had been wrapped around the breech end of the Mann barrel. Again, foam type insulation was placed around the plastic hoses to reduce the influx of heat from the surrounding environment. A mixture of environmentally friendly anti-freeze and isopropyl alcohol was pumped into this chilling system via a simple electrically driven fluid pump. The output of this pump was determined to be on the order of 2.8 gallons per minute. These actions resulted in bringing the temperature of the breech end of the barrel down to the 0ºF to +20ºF range. The temperature of the muzzle end of the Mann barrel was measured to be over 100 ºF. Subsequent thermal analysis estimated the temperature of the primer to be in the range of -13ºF at the time that they would be fired. Further efforts have been made to refine this barrel cooling approach by improving the thermal coupling between the copper tubing and the barrel as well as replacing the dry ice bath with a liquid nitrogen tank (LN2) and insulating the cooling coil arrangement with a shroud fixture.

The following general observations were made based on a review of these test results. MIC-based primer mixes utilizing BTATz and Kel-F were formulated, processed, produced, and consolidated into primers, which were then installed in cartridges and test fired in AURs that met fundamental cartridge action-time requirements. MIC-based primer mix produced from micron-size materials was not shown to be capable of meeting AUR action time requirements. Successful MIC formulations will likely use BTATz in ratios ranging from 15% to 25% and approximately 2% Kel-F and 2 % AB (carbon). Additional formulation work and AUR testing will be required to optimize the ratios and processing of these MIC constituents. The current formulations show a great deal of promise for meeting low-temperature functional requirements. Based on the positive results of preliminary work by the Army, as well as the Navy, alternative oxidizers, such as Bi_2O_3, will be further investigated during the ESTCP funded program.

Recent testing of several of the LFEP MIC primer mixes was completed using the new MIC formulation processes (different sonication times for the MIC) and the primer wet-loading procedure. The results of the testing are shown in Table 23. (Note: Test values are averages of more than one test firing. Individual primer firing test results can be found in appendix D.) Cold testing on two formulations was also conducted to evaluate primer performance under extreme environmental conditions specified by the medium caliber ammunition requirement.

Table 23 – AUR Firing Test Data Summary

Formulation Identity	Firing Test Date	Initial Temperature (ºF) of Primer	Primer Test Temperature (ºF)	Action Time (msec)	Chamber Pressure (ksi)	Velocity (fps)
KTHU-22	2 Mar 05	Ambient	Ambient	3.03	53.63	3370.38
KTHU-22	4 Apr 05	Ambient	Ambient	2.98	52.88	3325.06
KTHU-47	23 Jun 05	Ambient	Ambient	2.90	54.74	3452.08
KTHU-53	23 Jun 05	Ambient	Ambient	2.87	54.35	3437.58
KTHU-57	27 Sept 05	Ambient	Ambient	3.10	54.65	3387.09
KTHU-22	20 Oct 05	-65	-60 (-34 to-73)	3.21	52.72	3232.55
KTHW-24	24 Oct 05	-65	-65 (-47 to-75)	4.46	52.10	3278.25

The baseline LFEP primer formulation is KTHU-22, which is the standard primer mix consisting of 76% MIC (Technanogy 50-nm Al powder), 20% BTATz, 2% Kel-F and 2% carbon. The KTHU-47, KTHU-53, and KTHU-57 have the same composition, but differ only in the length of sonication time, 2, 1, 0.5 minutes, respectively. The KTHU-47 was the standard primer mix consisting of Al/MoO_3/BTATz/Kel-F with the exception of 80-nm Al powder from NTI being used instead of the 50-nm Al powder from Technanogy. The KTHU-53 consisted of the standard Al/ MoO_3/BTATz/Kel-F (using the 50-nm Al powder from Technanogy), but used a shorter sonication time (1 minute versus 2 minutes) for the mixing of the MIC (Al powder and MoO_3). The KTHU-57 consisted of the standard Al/ MoO_3/BTATz/Kel-F (using the 50-nm Al powder from Technanogy), but used a shorter sonication time (0.5 minute versus 2 minutes) for the mixing of the MIC (Al powder and molybdenum trioxide).

It appeared that sonication of the MIC for only 1 minute did not affect the action time, however, sonication of the MIC for only 0.5 minute did have some affect on the action time but is not very significant. Also replacing the Al (50 nm) with the Al (80 nm) did not affect the action time at ambient temperature. It was also noted for the KTHU-47 and KTHU-53 mixes that the formulations were prepared approximately 4 months prior to testing, and the KTHU-57 primer mix formulation had been prepared approximately 7 months prior to testing and apparently had little effect on the action times.

The initial results indicated that the cold-temperature testing process was very effective at insuring the primers are close to -65 ºF at the time they were fired. The action time for the KTHU-22 at cold temperature fell within the requirement and suggests that the molybdenum primer formulation had good potential as the down-selected candidate. The results from the KTHW-24 were unexpected. The lower particle size dispersion in the NTI 80-nm Al powder

should have given comparable results. However, the decreased performance may be due to the severe aging behavior of the aluminum material. Follow-up of the results is being conducted through further AUR testing with iterations of the KTHW-24 formulation using 80-nm Al stored under argon.

Samples KTHW-24, KTHW-32, and KTHW-36 were pressed into primers and tested in AURs. The formulations were prepared using the same standard formulation used in KTHU-22 but using 80-nm Al instead of 50-nm. The AURs were tested at cold temperatures and exhibited a wide dispersion of action times and a high average action time. The average results are shown in Table 24 below and the full results are given in Appendix D.

The KTHW-71, KTHW-73, and KTHW-74 performance test results were even less favorable. These results in AUR testing have generated several questions regarding the NovaCentrix Al and the effect of any changes in recent formulations. The formulations tested were made very similar to formulations that have shown promising results in the past, but using Al with a thicker passivation layer to protect against aging. Action times were very high, and shot-to-shot repeatability was dubious. Another issue was that the KTHW-73 formulation was identical to KTHU-22 except using NovaCentrix Al instead of Technanogy, and the results were very different. Based on the varying results and the transition of this program into an Environmental Security Technical Certification Program (ESTCP), the first step was to isolate the failure and reproduce the KTHU-22 formulation using the NovaCentrix Al.

Table 24 – AUR Test Results

Sample	**Conditioned Temperature**	**Primer Test Temperature**	**Action Time**	**Standard Deviation**
	(ºF) of Primer	**(ºF)**	**(msec)**	
KTHU-22	-65	-60 (-34 to-73)	3.21	0.12
KTHW-24	-65	-65 (-47 to-75)	4.46	1.02
KTHW-32	-65	-59 (-52 to-65)	8.45	4.57
KTHW-36	-65	-62 (-57 to-69)	4.23	1.21
KTHW-71	-65	-65 (-47 to-75)	6.93	7.06
KTHW-73	-65	-59 (-52 to-65)	53.3	27.38
KTHW-74	-65	-62 (-57 to-69)	63.28	18.2

The all-up primers prepared were designed to evaluate the use of NovaCentrix's 80-nm Al in place of the Technanogy's 50-nm Al powder in the MIC. As Technanogy is no longer in operation, NovaCentrix's 80-nm Al appears to be the best source of nano aluminum. Excellent AUR action times were obtained at both ambient and at low temperature using the legacy (Technanogy) 50-nm Al powder. The objective was to demonstrate that the NovaCentrix's 80-nm Al could give similar or better AUR action times.

Preliminary results using the NovaCentrix Al (80 nm) gave very promising results using the composition of the standard (KTHU-22). The fuel-oxidizer ratio in our KTHU-22 was 3.15,

which was also the optimal ratio found in our Pan Dent Optimization of the Al (50 nm)/MoO_3 composite. KTHU-47, using a fuel ratio of 2.0, gave an ambient temperature AUR action time of 2.90 $\pm$ 0.06 ms. Formulations, KTHW-24, KTHW-32, and KTHW-36 were all prepared using the standard formulation to investigate the low-temperature performance. The difference between the samples was variance of the fuel/oxidizer ratio, 2.37 for KTHW-24, 2.0 for KTHW-32, 2.5 for KTHW-34, and 3.0 for W36. Formulation KTHW-24 exhibited a wide range of action times and a high average action time of 4.46 + 1.02 ms.

Five factors that would likely affect the primer performance include:

1. The 80-nm Al does not work as well as the 50-nm at -65°F. This is unlikely since the 80-nm Al powder has a better particle size distribution and more active Al. However, the large particles in the Technanogy 50-nm Al powder may enhance propellant bed ignition.

2. The 80-nm Al was stored in air to determine the aging characteristics of the NovaCentrix powder. This material showed severe aging and after talking to the manufacturer, it was discovered that the oxide coating was only about 1.1-nm thick instead of 2.5 nm requested. The active Al content dropped from 86% active down to 74% over a period of 6 weeks. The primer was made using this Al and not fired for 2 months. The additional aging could have lowered the active Al content further thus degrading the MIC performance.

3. A new batch of BTATz was used in the formulation. Nothing abnormal was noted about the material. The quality of the BTATz is unlikely a contributor.

4. A fuel/oxidizer ratio of 2.37 was used in the preparation of the Al/Mo_2O_3 composite. This composition was recommended by LANL. This may or may not be the optimum composition for this aging Al.

5. Another final possibility is due to storage in hexane over a prolonged period that promoted phase separation of the primer powder.

PARTNERING

For this project, the China Lake team formally partnered with the Picatinny Arsenal and the LANL in order to take advantage of the specialized skills, knowledge, and capabilities of these organizations. The Navy supplied funding to these two facilities to support their efforts. One of the advantages of these ongoing partnerships was the financial benefit that accrues as a result of leverage. This leverage was available since these organizations have similar efforts that overlap China Lake's areas of interest and the Navy effectively got more for its investment than NAWCWD would normally realize. All institutions benefited from this union, having shared information about processing, handling/testing techniques, and measurements of performance parameters. It is expected that these joint efforts will continue through the duration of the program.

Again, as already noted, NAWCWD also established important working relationships with SNC (now GDOTS, Canada, Inc.) and LCAAP. Since GDOTS, Canada, Inc was expecting to use MIC materials in production of primers, it was believed that their insights into production techniques would be of great value to our efforts. Our working relationship with LCAAP was likewise beneficial; allowing NAWCWD to obtain, at no cost to the program, the necessary primer metal parts needed during the test program.

The Navy, both "Air" and "Sea" arms, and the Army were able to coordinate their efforts. NAWCWD hosted a Multi-Agency Medium Caliber Ammunition Environmental Team meeting where each of the agencies described their ongoing efforts, including problems and areas of concern. This meeting resulted in a number of action items focused on resolving issues of interest to all of the representatives. The meetings were beneficial to all concerned.

The MIC Joint (Service) Working Group (JWG) also assisted SERDP efforts. Each of the services sent their representatives to the meetings and allowed them to remain in close contact with peers from other organizations that looked at MIC as a technology to be exploited for a number of different applications. The plan was for future MIC JWG meetings to be held in conjunction with DOE/DOD Technical Coordinating Group (TCG) Lethality Enhancement (TCG II) meetings.

In return, the Army requested that NAWCWD provide some critical test support in their evaluation of various primer mixes for their medium-caliber percussion primer program. The Navy used their Ballistics Test Lab to perform these tests during the first quarter of calendar year 2004.

COLLABORATION/ON-SITE TRAINING

One final aspect of the partnering situation was that NAWCWD provided a Navy employee to work at the LANL facility over a 12-month period. This individual, Dr. Tim Foley, worked closely with Dr. Steve Son, a LANL employee that was significantly involved in the study of MIC materials, including the LFEP Program. Dr. Foley learned and shared knowledge and techniques with Dr. Son and other LANL personnel during his period of temporary duty at LANL. As another example of leverage, the cost of Dr. Foley's efforts while working at LANL was covered by the DOD/DOE memorandum of understanding (MOU).

Initial Army work was conducted in looking at a nano aluminum-based MIC compound plus gas-producing additives as a substitute for the lead styphnate-based initiating compound in electric primers. The base compound was referred to as MIC +10, nano aluminum/molybdenum trioxide MIC plus the additives PETN, ethyl cellulose, and calcium resinate.

MIC +10 had been investigated as the initiation compound in small-caliber percussion primers and laser ignition developmental work. While basic MIC showed promise, it exhibited relatively large variation with respect to ignition time (defined here as time to peak pressure). Work recently completed on small-caliber percussion primers showed that this material provided excellent progress in significantly reducing this variability.

With the success of the BTATz formulation as discussed earlier, the Army intended to expand their efforts to include BTATz as an alternative gas additive in primer mix. The Army's work with the BTATz formulation was hampered by some initial confusion on mixing procedures. These issues were overcome and the Army was expecting to begin their processing efforts in the near future. The Army purchased primer metal parts and was prepared to perform primer assembly processes once they had appropriate MIC materials to work with.

Experiments were conducted using three lots of nano aluminum provided by the Technanogy company. This evaluation took the form of producing basic MIC and MIC +10 from the same nano aluminum lot, charging the resultant compositions into percussion primers, and firing these primers in an ignition test fixture. The time to peak pressure, which indicated the ignition efficiency of the MIC composition, was measured and is listed in Table 25. In addition, MIC +10 samples made from these lots were also evaluated.

The lots provided by Technanogy were labeled:

A: 31.1 nm; 63.0 m^2/g; 63.5 wt% Al; 1.6 nm oxide
B: 31.5 nm; 62.6 m^2/g; 64.6 wt% Al; 1.6 nm oxide
C: 32.3 nm; 60.9 m^2/g; 63.8 wt% Al; 1.7 nm oxide

Each lot of nano aluminum was mixed with molybdenum trioxide (Climax lot 1131). The standard mixing procedure was followed in all cases. The unused MIC materials were then re-blended with the MIC + 10 additives that are intended to increase the output of the primer to produce ignition efficiency and ballistic performance comparable to the lead styphnate primers currently in use.

Table 25 – Time to Peak Pressure (milliseconds)

	LOT A	LOT A+	LOT B	LOT B+	LOT C	LOT C+
	0.0192	0.0024	0.0122	0.0022	0.0100	0.0024
	0.0113	0.0024	0.0140	0.0023	0.0080	0.0023
	0.0156	0.0024	0.0138	0.0024	0.0050	0.0022
	0.0270	0.0023	0.0175	0.0024	0.0070	0.0023
	0.0112	0.0024	0.0125	0.0023	0.0104	
	0.0093	0.0025	0.0156	0.0024		
	0.0210	0.0022	0.0148	0.0022		
	0.0188	0.0022	0.0092	0.0020		
	0.0102	0.0024	0.0114	0.0021		
	0.0300	0.0023		0.0021		
Average	0.0169	0.00235	0.0123	0.0022	0.0081	0.0023
Std Dev	0.0060	9.2 E-5	0.0025	1.4 E-4	0.0022	8.17E-5
High	0.0300	0.0025	0.0156	0.0024	0.0100	0.0024
Low	0.0093	0.0022	0.0092	0.0020	0.0050	0.0022

Note: "+" indicates MIC with additives.

The ignition performance, in terms of time to peak pressure, of the basic MIC material varies by lot and is four to eight times longer than the desired level seen in a lead styphnate based primer, or approximately 2.2 milliseconds. While the standard deviation for the basic MIC material is small, it is still in the same order of magnitude as the mean, which gives an undesirable spread in the potential ignition times. However, when the additives are added to the basic MIC, a high degree of uniformity and decreased the delay of propellant ignition is achieved. The time to peak pressures for the standard lead styphnate (FA 956) primer mix and the modified MIC is virtually identical as shown by the graphs in Figures 17 and 18.

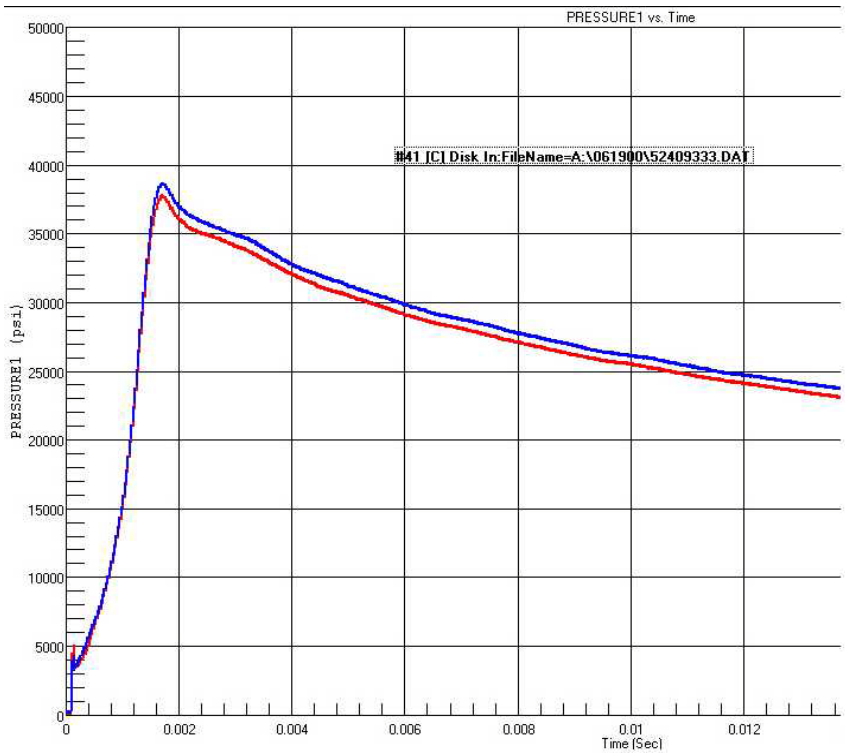


Figure 17 – Pressure Versus Time Curve for Typical FA 956 Primer Energetics

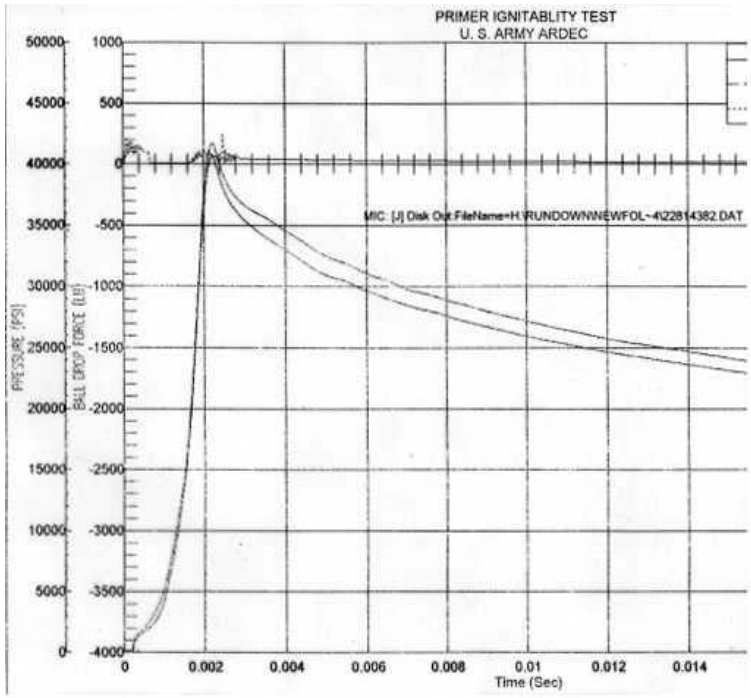


Figure 18 – Pressure Versus Time Curve for MIC +10 Primer Energetics

Initial characterization of the MIC +10 material as a conductive mix was evaluated. MIC +10 was loaded into existing 20-mm M52 primer hardware and the electrical resistance was measured. One sample had a resistance value of 18 M ohms, while the other sample showed an open circuit. To enhance conductivity, carbon black was added to the mix at 1% and 5% concentrations. The sample with the 1% carbon black concentration again indicated an open circuit while the 5% concentration measured as a shorted circuit (zero ohms). Further attempts to replicate a conductive mix similar to the standard lead styphnate materials were made with substitution of 10% calcium silicide as an additive instead of the calcium resinate currently used in the MIC +10. Resistance values for this sample measured 2.2 M ohms. All of the samples were set aside in an open environment to determine if any changes in the MIC properties would occur over time, as was observed in the percussion primer work. Table 26 shows the initial resistance of the initial as well as the subsequent measurement taken approximately 6 weeks after assembly. As shown in the table, the resistance values for all of the samples with materials added to the MIC +10 have changed with time, while the resistance of the basic MIC +10 material has remained basically the same.

Table 26 – MIC +10 Conductive Mix Results Data

MIC+10	**Additive**	**Resistance/Date**	**Resistance 6-Sep 02**
100%	0	18 Mohm/ 18 Jul 02	open
100%	0	Open/18 Jul 02	open
99	1% carbon black	Open/12 Aug 02	5.9 Mohm
95	5% carbon black	0 (short)/13 Aug 02	155 ohm
*	10% CaSi2	2.2 Mohm/23 Aug 02	5.6 Mohms

*Calcium Resinate Additive Replaced with Calcium Silicide

Collaboration continued with Picatinny Arsenal on their work with the lead-free substitute for the 25-mm M115 percussion primer. China Lake agreed to perform fundamental primer-cup loading operations for the Army, but due to some confusion over the availability of tooling required to load the MIC-based primer material into the primer metal parts, this effort was not successfully completed.

Cooperative efforts with the Army on the 30-mm PA520 electric primer were also conducted. NAWCWD shared the results of the Navy's most recent and effective MIC mixture formulations, as well as the results of the Navy-sponsored oxidizer study performed by LANL. China Lake created a matrix for the testing of MIC-based compounds for the PA520 electric primer.

The Army reported that they fabricated 12 PA520 electric primers using the Al/BiO_3/PETN formulation. The electrical resistance of these primers was verified before loading them into 30-mm cartridge cases. All of these cartridges had the flashtube assembly in place in accordance with the standard end-item drawings. Six of the flashtubes were loaded with black powder and six were loaded with IB52 pellets. The cartridge cases were test-fired and demonstrated acceptable action times.

The Army fired the first iteration of AURs with the PA520 consisting of 10 rounds with the same MIC primer mix that the Army is currently using for the 5.56 mm primers. Of the 10 rounds tested, eight AURs had action times under 3 milliseconds (msec). The primer configuration was a carbon bridge on the taper button and all MIC primer mix filled the remainder of the primer cup. The demonstration of the 30-mm AURs was conducted at Picatinny Arsenal. Two of the AURs had action times greater than 3 msec—one 4.1 msec and one 5.0 msec.

Cooperative efforts with the Army, LANL, LCAAP and GDOTS, Canada, Inc. are expected to continue into the transition to ESTCP.

SUMMARY AND CONCLUSIONS

The past 5 years have been very productive in terms of gaining additional knowledge about the materials used in the preparation of primer mixes and the processes that can be used to treat them prior to and during the primer fabrication process. NAWCWD overcame some major obstacles, meeting both the low-temperature firing requirement (-65°F) and AUR action time (less than 4ms). However, there have also been some setbacks. Tests have failed to reproduce the AUR action times due to either the constituents, including the nano aluminum and BTATz. To date there has not been a single incident with the current aluminum/molybdenum formulation. This demonstrates that the LFEP can be used with suitable safety guidelines. Other areas where significant progress has been made or is in the process of being made include the following:

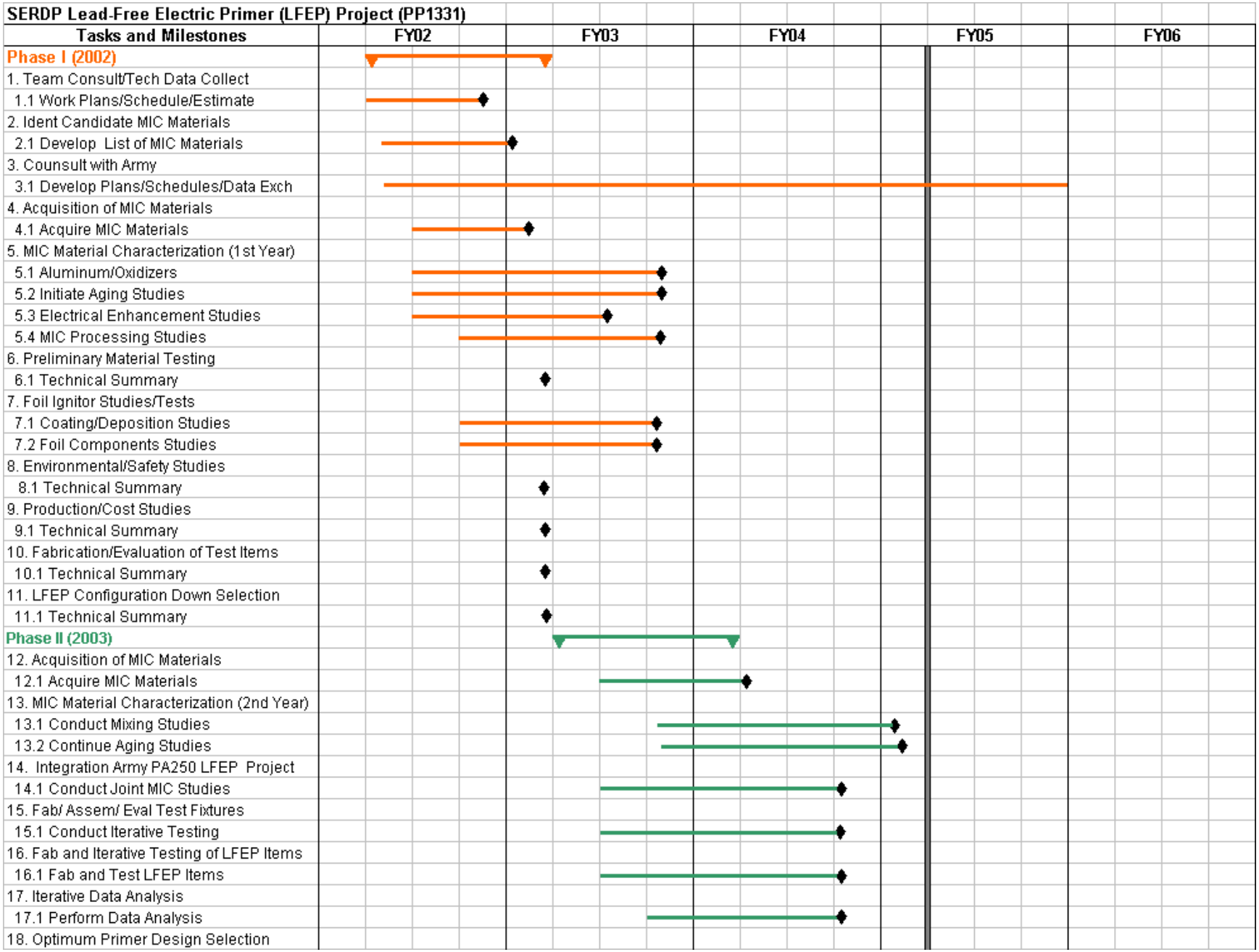
- The lack of larger particles in NovaCentrix 80 nm Al powders can be mitigated with the intentional addition of 1-2% 2-micron Al. This should solve the poor performance at low temperature. The micron Al particles are expected to act as large burning particles to ignite the propellant bed. $CaSi_2$ is added to conventional primers for this reason.
- Bismuth trioxide composites, out perform molybdenum trioxide, but exhibits extremely high ESD sensitivity, and the severe safety hazard precludes its use in the field.
- Micron-sized particles have been shown to offer improved handling safety, but do not produce sufficiently rapid reaction rates.
- The sequence for adding the ingredients into the primer mix has been found to be critically important to the overall performance of the primer.
- Final ratios of BTATz, Kel-F, and AB have been established and are used in the current production of primer mix.
- Aging of nano aluminum in air is partially controlled by the passivation oxide thickness. A thickness of at least 2.7 nm is needed for DOD applications. NAWCWD is working with NovaCentrix to produce nano aluminum powders with consistent active Al content and with passivation oxide layers of 2.7- to 3.0-nm thick.

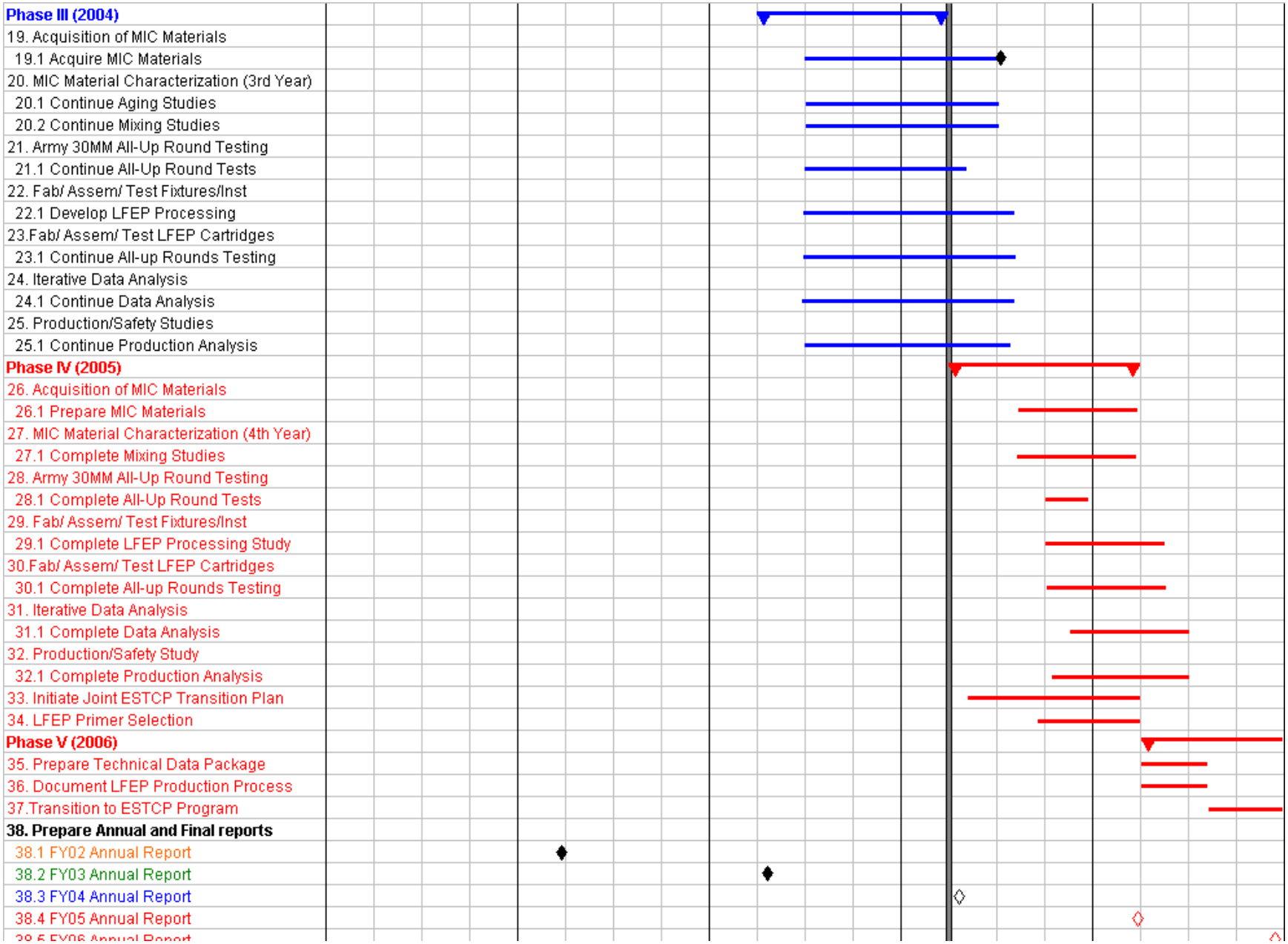
- Aging of nano MoO_3 has been eliminated by an appropriate heat treatment that converts the monoclinic to the desirable orthorhombic crystalline phase. The orthorhombic phase is neither light nor water sensitive. It has been observed that the heat treatment of the MoO_3 produces larger particle sizes that could adversely affect reaction rates and AUR action times.
- Mixing technique has been shown to have a major effect on the performance of the primer mix.
- A number of different solvents have been investigated as alternatives to hexane decrease AUR action time. Enhanced performance was obtained using either FC-77 or heptane instead of hexane.
- Wet consolidation of the primer mix into the primer cup holds promise of increased safety and potentially more compatibility with high rate of production techniques.
- Water loading of the Al/MoO_3 composite exhibited low performance most likely due to the dissolutions of the MoO_3 and subsequent catalytic hydrolysis of the nano Al.

The transition process to ESTCP will include verifying mixing and pressing procedures. The solvent used will also be further examined an attempt to find an alternative may be found to decrease AUR action times. The reasoning behind this is that a solvent with a higher boiling point produces higher performance MIC and may produce shorter AUR action times. NAWCWD will implement the use of solvent recycling hardware. Lastly, despite confidence in the optimization of the current LFEP formulation, there are still concerns about the commercial availability of some constituents, including BTATz. Alternative gas generants and methods to optimize gas generation will be explored during the earliest phase of the ESTCP transition.

Appendix A

UPDATED SCHEDULE/MILESTONES


SERDP Lead-Free Electric Primer (LFEP) Project (PP1331)
Tasks and Milestones
FY02
FY03
FY04
FY05
FY06
Phase I (2002)
1. Team Consult/Tech Data Collect
1.1 Work Plans/Schedule/Estimate
2. Ident Candidate MIC Materials
2.1 Develop List of MIC Materials
3. Counsult with Army
3.1 Develop Plans/Schedules/Data Exch
4. Acquisition of MIC Materials
4.1 Acquire MIC Materials
5. MIC Material Characterization (1st Year)
5.1 Aluminum/Oxidizers
5.2 Initiate Aging Studies
5.3 Electrical Enhancement Studies
5.4 MIC Processing Studies
6. Preliminary Material Testing
6.1 Technical Summary
7. Foil Ignitor Studies/Tests
7.1 Coating/Deposition Studies
7.2 Foil Components Studies
8. Environmental/Safety Studies
8.1 Technical Summary
9. Production/Cost Studies
9.1 Technical Summary
10. Fabrication/Evaluation of Test Items
10.1 Technical Summary
11. LFEP Configuration Down Selection
11.1 Technical Summary
Phase II (2003)
12. Acquisition of MIC Materials
12.1 Acquire MIC Materials
13. MIC Material Characterization (2nd Year)
13.1 Conduct Mixing Studies
13.2 Continue Aging Studies
14. Integration Army PA250 LFEP Project
14.1 Conduct Joint MIC Studies
15. Fab/ Assem/ Eval Test Fixtures
15.1 Conduct Iterative Testing
16. Fab and Iterative Testing of LFEP Items
16.1 Fab and Test LFEP Items
17. Iterative Data Analysis
17.1 Perform Data Analysis
18. Optimum Primer Design Selection


Phase III (2004)
19. Acquisition of MIC Materials
19.1 Acquire MIC Materials
20. MIC Material Characterization (3rd Year)
20.1 Continue Aging Studies
20.2 Continue Mixing Studies
21. Army 30MM All-Up Round Testing
21.1 Continue All-Up Round Tests
22. Fab/ Assem/ Test Fixtures/Inst
22.1 Develop LFEP Processing
23.Fab/ Assem/ Test LFEP Cartridges
23.1 Continue All-up Rounds Testing
24. Iterative Data Analysis
24.1 Continue Data Analysis
25. Production/Safety Studies
25.1 Continue Production Analysis
Phase IV (2005)
26. Acquisition of MIC Materials
26.1 Prepare MIC Materials
27. MIC Material Characterization (4th Year)
27.1 Complete Mixing Studies
28. Army 30MM All-Up Round Testing
28.1 Complete All-Up Round Tests
29. Fab/ Assem/ Test Fixtures/Inst
29.1 Complete LFEP Processing Study
30.Fab/ Assem/ Test LFEP Cartridges
30.1 Complete All-up Rounds Testing
31. Iterative Data Analysis
31.1 Complete Data Analysis
32. Production/Safety Study
32.1 Complete Production Analysis
33. Initiate Joint ESTCP Transition Plan
34. LFEP Primer Selection
Phase V (2006)
35. Prepare Technical Data Package
36. Document LFEP Production Process
37.Transition to ESTCP Program
38. Prepare Annual and Final reports
38.1 FY02 Annual Report
38.2 FY03 Annual Report
38.3 FY04 Annual Report
38.4 FY05 Annual Report

Appendix B

PRIMER MIX FORMULATION DATA

Formula Name	MIC						Fuel/Oxi	Binder	Carbon	Wet/Dry	Date Formulated mm/dd/yy	MIC Wt%	BTATz Wt%	Binder Wt%	Carbon Wt%	Sonication			
	Fuel	Source	Size nm	Oxidizer	Source	Size nm										Horn	Amplitude	Pulse (s)	Time (min)
KTHS42	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		06/02/03	64.8	31.0	2.1	2.1	100W			
KTHS43	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		06/03/03	75.9	19.9	2.1	2.1	100W			
KTHS44	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		06/05/03	85.8	10.3	2.1	1.7	100W			
KTHS52	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		07/14/03	67.3	28.9	1.9	1.9	100W			
KTHS53	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		07/15/03	68.6	29.4	2.0	0.0	100W			
KTHS54	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips	Wet Hexane	08/21/03	75.0	22.0	1.5	1.5	100W			
KTHS56	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		11/01/03	66.0	30.0	2.0	2.0	100W			
KTHS57	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		11/01/03	66.7	29.4	2.0	2.0	100W			
KTHS69	Aluminum	Technanogy	50	MoO_3	Climax	40	3.12	Kel-F	Chevron-Phillips	Wet Hexane	01/12/04	55.8	39.8	2.4	2.0	100W	100	0.5	2
KTHS70	Aluminum	Technanogy	50	MoO_3	Climax	40	3.12	Kel-F	Chevron-Phillips		01/12/04	55.8	39.7	2.4	2.1	100W	100	0.5	2
KTHS73	Aluminum	Indian Head	50	MoO_3	Climax	40	2.81	Kel-F	Chevron-Phillips		02/06/04	66.7	29.2	2.1	2.0	US Bath	100		60
KTHS74	Aluminum	Indian Head	50	MoO_3	Climax	40	2.81	Kel-F	Chevron-Phillips		02/18/04	80.6	15.5	1.9	2.0	US Bath	100		60
KTHS75	Aluminum	Indian Head	50	MoO_3	Climax	40	2.81	Kel-F	Chevron-Phillips		02/18/04	70.9	25.4	1.8	1.8	US Bath	100		60
KTHS90	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		04/05/04	67.0	29.1	1.9	2.0	400W			

Formula Name	MIC						Fuel/Oxi	Binder	Carbon	Wet/Dry	Date Formulated mm/dd/yy	MIC Wt%	BTATz Wt%	Binder Wt%	Carbon Wt%	Sonication			
	Fuel	Source	Size nm	Oxidizer	Source	Size nm										Horn	Amplitude	Pulse (s)	Time (min)
KTHS92	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		04/05/04	67.1	28.7	2.2	1.9	400W			
KTHS93	Aluminum	Technanogy	50	MoO_3	Climax	40		Kel-F	Chevron-Phillips		04/05/04	67.3	28.8	2.1	1.8	400W			
KTHU3	Aluminum	Technanogy	50	MoO_3	Climax	40	3.12	Kel-F	Chevron-Phillips	Wet Hexane	06/07/04	75.9	20.1	2.0	2.0	100W	100	0.5	2
KTHU4	Aluminum	Technanogy	50	MoO_3	Climax	40	3.12	Kel-F	Chevron-Phillips	Wet Hexane	06/07/04	74.4	19.7	2.0	3.9	100W	100	0.5	2
KTHU5	Aluminum	Technanogy	50	MoO_3	Climax	40	3.12	Kel-F	Chevron-Phillips	Wet Hexane	06/07/04	72.6	19.2	1.9	6.2	100W	100	0.5	2
KTHU6	Aluminum	Technanogy	50	MoO_3	Climax	40	3.12	Kel-F	Chevron-Phillips	Wet Hexane	06/07/04	75.9	20.1	2.0	2.0	100W	100	0.5	2
KTHU22	Aluminum	Technanogy	50	MoO_3	Climax	40	3.02	Kel-F	Chevron-Phillips	Wet Hexane	01/26/05	75.9	20.0	2.0	2.1	400W	75	0.5	2
KTHU47	Aluminum	NanoTechnology	80	MoO_3	Climax	40	2.04	Kel-F	Chevron-Phillips	Wet Hexane	02/16/05	75.9	20.1	2.0	2.0	400W	75	0.5	2
KTHU53	Aluminum	Technanogy	50	MoO_3	Climax	40	2.87	Kel-F	Chevron-Phillips	Wet Hexane	02/23/05	76.0	20.0	2.0	2.0	400W	75	0.5	1
KTHU57	Aluminum	Technanogy	50	MoO_3	Climax	40	2.84	Kel-F	Chevron-Phillips	Wet Hexane	02/25/05	75.9	20.1	2.0	2.0	400W	75	0.5	0.5
KTHU62	Aluminum	NanoTechnology	80	Bi_2O_3	Aldrich	320	2.03	Kel-F	Chevron-Phillips	Wet Hexane	03/03/05	75.9	19.9	2.1	2.1	400W	75	0.5	1
KTHU78	Aluminum	NanoTechnology	80	Bi_2O_3	Skylighter	2.5 µm		Kel-F	Chevron-Phillips	Wet Hexane	05/16/05	76.0	20.1	1.9	2.0	400W	75	0.5	1
KTHW24	Aluminum	NanoTechnology	80	MoO_3	Climax	40	2.37	Kel-F	Chevron-Phillips	Wet Hexane	10/11/05	76.0	20.0	2.0	2.0	400W	75	0.5	2

Appendix C

PRIMER FABRICATION INFORMATION

Primer	Formulation	Date Received mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Wet/Dry	Humidity %	Weight Dry Assembly gm	Weight Powder mg	Weight Loaded gm	Height in	Consolidation Pressure Kpsi	Dwell Time Sec	Primer Resistance Ohms	Notes/Comments All initial consolidation at 300 psi
6	KTHU-22		02/14/05	3/2/05	Wet Loaded Oven Dried		1.329	150		0.244	8			
7	KTHU-22		02/14/05	3/2/05	Wet Loaded Oven Dried		1.314	160		0.24	8			
8	KTHU-22		02/14/05	3/2/05	Wet Loaded Oven Dried		1.349	160		0.256	8			
9	KTHU-22		02/14/05	3/2/05	Wet Loaded Oven Dried		1.32	160		0.241	8			
10	KTHU-22		02/16/05	3/2/05	Wet Loaded Oven Dried		1.339	160		0.256	8			
11	KTHU-22		02/16/05	3/2/05	Dry Pressed		1.357	160		0.26	8			
12	KTHU-22		02/16/05	3/2/05	Wet Loaded Oven Dried		1.145	160		0.251	8			
13	KTHU-22		02/16/05	3/2/05	Wet Loaded Oven Dried		1.78	160		0.256	8			
14	KTHU-22		02/16/05	3/2/05	Wet Loaded Oven Dried		1.205	160		0.266	8			
15	KTHU-22		02/16/05		Wet Loaded Oven Dried		1.232	160		0.254	8			
16	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.2	160		0.254	8			
17	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.82	160		0.253	8			
18	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.42	160		0.258	8			
19	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.4	160		0.258	8			

Primer	Formulation	Date Received mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Wet/Dry	Humidity %	Weight Dry Assembly gm	Weight Powder mg	Weight Loaded gm	Height in	Consolidation Pressure Kpsi	Dwell Time Sec	Primer Resistance Ohms	Notes/Comments All initial consolidation at 300 psi
20	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.62	160		0.254	8			
21	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.55	160		0.259	8			
22	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.65	160		0.248	8			
23	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.55	160		0.265	8			
24	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.22	160		0.263	8			
25	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.65	160		0.262	8			
26	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.54	160		0.257	8			
27	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.52	160		0.261	8			
28	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.57	160		0.261	8			
29	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.55	160		0.254	8			
30	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.54	160		0.257	8			
31	KTHU-22		03/31/05		Wet Loaded Oven Dried		1.62	160		0.256	8			
1	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.52	160		0.248	8			Clumpier and harder to work with
2	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.56	160		0.252	8			
3	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.56	160		0.248	8			

Primer	Formulation	Date Received mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Wet/Dry	Humidity %	Weight Dry Assembly gm	Weight Powder mg	Weight Loaded gm	Height in	Consolidation Pressure Kpsi	Dwell Time Sec	Primer Resistance Ohms	Notes/Comments All initial consolidation at 300 psi
4	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.54	160		0.246	8			
5	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.61	160		0.248	8			
6	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.48	160		0.245	8			
7	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.56	160		0.253	8			
8	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.38	160		0.236	8			
9	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.5	160		0.246	8			
10	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.38	160		0.236	8			
11	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.4	160		0.238	8			
12	KTHU-47		05/12/05		Wet Loaded Oven Dried		1.28	160		0.23	8			
1	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.5	160		0.25	8			
2	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.5	160		0.245	8			
3	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.56	160		0.254	8			
4	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.56	160		0.254	8			
5	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.57	160		0.265	8			
6	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.54	160		0.256	8			

Primer	Formulation	Date Received mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Wet/Dry	Humidity %	Weight Dry Assembly gm	Weight Powder mg	Weight Loaded gm	Height in	Consolidation Pressure Kpsi	Dwell Time Sec	Primer Resistance Ohms	Notes/Comments All initial consolidation at 300 psi
7	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.55	160		0.256	8			
8	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.48	160		0.251	8			
9	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.57	160		0.264	8			
10	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.51	160		0.252	8			
11	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.47	160		0.248	8			
12	KTHU-53		05/12/05		Wet Loaded Oven Dried		1.59	160		0.268	8			
1	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.255	8			
2	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.256	8			
3	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.258	8			
4	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.256	8			
5	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.26	8			
6	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.258	8			
7	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.259	8			
8	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.259	8			
9	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.256	8			

Primer	Formulation	Date Received mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Wet/Dry	Humidity %	Weight Dry Assembly gm	Weight Powder mg	Weight Loaded gm	Height in	Consolidation Pressure Kpsi	Dwell Time Sec	Primer Resistance Ohms	Notes/Comments All initial consolidation at 300 psi
10	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.259	8			
11	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.256	8			
12	KTHU-57		09/19/05		Wet Loaded Oven Dried			160		0.256	8			

Appendix D

FUNCTIONAL/FIRING TEST DATA

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (° F)	Conditioned Temperature (° F)	Estimated Primer Temp (° F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	KTHS-43			2/12/04	-	-	-	2.97	-	3322
2	KTHS-43			2/12/04	-	-	-	3.05	-	3317
3	KTHS-43			2/12/04	-	-	-	3.03	-	3322
4	KTHS-43			2/12/04	-	-	-	3.04	-	3328
5	KTHS-43			2/12/04	-	-	-	2.93	-	3311
6	KTHS-43			2/12/04	-	-	-	2.88	-	3333
							Average	2.98	-	3322.17
							High	3.05	-	3333.00
							Low	2.88	-	3311.00
							SD	0.07	-	7.78

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (° F)	Conditioned Temperature (° F)	Estimated Primer Temp (° F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	KTHS-44			2/12/04	-	-	-	3.93	-	3322
2	KTHS-44			2/12/04	-	-	-	3.58	-	3328
3	KTHS-44			2/12/04	-	-	-	3.75	-	3317
4	KTHS-44			2/12/04	-	-	-	3.37	-	3317
5	KTHS-44			2/12/04	-	-	-	3.79	-	3322
6	KTHS-44			2/12/04	-	-	-	3.41	-	3311
							Average	3.64	-	3319.50
							High	3.93	-	3328.00
							Low	3.37	-	3311.00
							SD	0.22	-	5.82

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (° F)	Conditioned Temperature (° F)	Estimated Primer Temp (° F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	KTHS-56			2/12/04	-	-	-	4.72	-	3322
2	KTHS-56			2/12/04	-	-	-	3.76	-	3317
3	KTHS-56			2/12/04	-	-	-	5.09	-	3322
4	KTHS-56			2/12/04	-	-	-	3.52	-	3322
5	KTHS-56			2/12/04	-	-	-	3.49	-	3322
6	KTHS-56			2/12/04	-	-	-	4.31	-	3322
							Average	4.15	-	3321.17
							High	5.09	-	3322.00
							Low	3.49	-	3317.00
							SD	0.67	-	2.04

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (° F)	Conditioned Temperature (° F)	Estimated Primer Temp (° F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	KTHS-74			3/25/04	-	-	-	3.04	62.3	3273
2	KTHS-74			3/25/04	-	-	-	3.13	61.1	3268
3	KTHS-74			3/25/04	-	-	-	2.91	61.3	-
4	KTHS-74			3/25/04	-	-	-	3.08	63.0	3284
5	KTHS-74			3/25/04	-	-	-	3.03	61.9	3273
6	KTHS-74			3/25/04	-	-	-	3.11	63.0	3279
7	KTHS-74			3/25/04	-	-	-	2.89	61.5	3328
8	KTHS-74			3/25/04	-	-	-	2.94	61.9	-
9	KTHS-74			3/25/04	-	-	-	3.09	62.3	3273
10	KTHS-74			3/25/04	-	-	-	3.13	62.5	3284
							Average	3.04	62.08	3282.75
							High	3.13	63.00	3328.00
							Low	2.89	61.10	3268.00
							SD	0.09	0.66	19.15

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (° F)	Conditioned Temperature (° F)	Estimated Primer Temp (° F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	KTHS-75			3/25/04	-	-	-	3.01	61.7	3273
2	KTHS-75			3/25/04	-	-	-	2.91	61.7	3273
3	KTHS-75			3/25/04	-	-	-	2.89	61.7	3268
4	KTHS-75			3/25/04	-	-	-	2.92	61.8	3273
5	KTHS-75			3/25/04	-	-	-	2.89	63.9	3289
6	KTHS-75			3/25/04	-	-	-	2.91	63.1	3273
7	KTHS-75			3/25/04	-	-	-	2.89	61.9	3273
8	KTHS-75			3/25/04	-	-	-	2.98	63.6	3279
9	KTHS-75			3/25/04	-	-	-	2.96	63.2	3273
10	KTHS-75			3/25/04	-	-	-	3.20	64.1	3289
							Average	2.96	62.67	3276.30
							High	3.20	64.10	3289.00
							Low	2.89	61.70	3268.00
							SD	0.10	1.00	7.18

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (° F)	Conditioned Temperature (° F)	Estimated Primer Temp (° F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	KTHS-90			4/29/04	-	ambient	-	4.83	62.1	3377
2	KTHS-90			4/29/04	-	ambient	-	3.82	61.6	3377
3	KTHS-90			4/29/04	-	ambient	-	3.67	62.1	3373
4	KTHS-90			4/29/04	-	ambient	-	4.23	61.9	3392
5	PGU-27/B			4/29/04	-	ambient	-	2.60	55.0	3435
6	PGU-27/B			4/29/04	-	ambient	-	-	-	3539
7	PGU-27/B			4/29/04	-	ambient	-	-	-	-
8	PGU-27/B			4/29/04	-	ambient	-	2.62	54.0	3430
							Average	4.14	61.93	3379.75
							High	4.83	62.10	3392.00
							Low	3.67	61.60	3373.00
							SD	0.52	0.24	8.38

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (^{o} F)	Conditioned Temperature (^{o} F)	Estimated Primer Temp (^{o} F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	KTHS-90			4/29/04	-	-65	-	>35	-	-
2	KTHS-90			4/29/04	-	-65	-	40.80	-	3341
3	KTHS-90			4/29/04	-	-65	-	23.00	-	3321
4	KTHS-90			4/29/04	-	-65	-	50.30	-	3285
5	KTHS-90			4/29/04	-	-65	-	45.80	-	3378
6	KTHS-90			4/29/04	-	-65	-	4.84	-	3337
7	KTHS-90			4/29/04	-	-65	-	5.60	-	3355
8	KTHS-90			4/29/04	-	-65	-	5.47	-	3355
9	PGU-27/B			4/29/04	-	-65	-	2.74	54.7	3292
10	PGU-27/B			4/29/04	-	-65	-	2.68	58.0	3392
							Average	25.12	-	3338.86
							High	50.30	-	3378.00
							Low	4.84	-	3285.00
							SD	20.37	-	29.67

1	PGU-27/B			7/29/04	104	NA	NA	2.6	48.9	3290
2	PGU-27/B			7/29/04	104		-10	2.6	46.3	3257
3	KTHU-3			7/29/04	104		-13	3.5	52.6	3333
4	KTHU-3			7/29/04	104		-14	5.7	55.5	3367
5	KTHU-3			7/29/04	104		-14.5	3.6	51.6	3279
6	KTHU-3			7/29/04	104		-13	3.6	52.4	3322
7	KTHU-3			7/29/04	104		-10	3.2	30.6	3269
8	KTHU-3			7/29/04	104		-10	3.7	51.2	3278
9	KTHU-3			7/29/04	104		-10	3.3	51.5	3278
10	KTHU-3			7/29/04	104		-13	3.8	53.1	3301
11	KTHU-3			7/29/04	104		-10	3.4	53.2	3289
12	KTHU-3			7/29/04	104		-13.5	3.8	53.0	3279
13	KTHU-3			7/29/04	104		-13	3.6	53.6	3300
14	KTHU-3			7/29/04	104		-13.5	3.4	51.4	3279
15	KTHU-3			7/29/04	104		-13	3.6	52.5	3290
16	KTHU-3			7/29/04	104		-13	3.5	53.1	3300
17	KTHU-3			7/29/04	104		-14	3.5	52.4	3279
18	KTHU-3			7/29/04	104		-14	3.5	53.4	3290
19	KTHU-3			7/29/04	104		-12.5	3.8	51.6	3279
20	KTHU-3			7/29/04	104		-13	3.4	54.3	3300
21	KTHU-3			7/29/04	104		-13	3.3	52.9	3278
22	KTHU-3			7/29/04	104		-10	3.8	52.8	3311
							Average	3.65	51.64	3295.05
							High	5.70	55.50	3367.00
							Low	3.20	30.60	3269.00
							SD	0.51	5.06	23.66

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (^{o} F)	Conditioned Temperature (^{o} F)	Estimated Primer Temp (^{o} F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	PGU-27/B			9/16/04	94		-20	2.40	50.1	3379
2	PGU-27/B			9/16/04	94		-13.5	2.50	50.7	3322
3	PGU-27/B			9/16/04	94		-13	2.50	52.7	3648
4	PGU-27/B			9/16/04	94		-9.5	2.48	57.4	3677
5	PGU-27/B			9/16/04	94		-10	2.60	47.2	3610
6	PGU-27/B			9/16/04	94		-13	2.56	48.8	-
7	PGU-27/B			9/16/04	94		-13	2.57	48.3	3449
8	PGU-27/B			9/16/04	94		-9	2.60	50.8	3268
9	KTHU-4			9/16/04	94		-13	3.40	53.1	2976
10	KTHU-4			9/16/04	94		-13	3.83	52.4	3279
11	KTHU-4			9/16/04	94		-13	3.88	52.7	3290
12	KTHU-4			9/16/04	94		-13	3.47	52.8	3290
13	KTHU-4			9/16/04	94		-12.5	3.96	53.6	3299
14	KTHU-4			9/16/04	94		-13	3.61	53.1	3279
15	KTHU-4			9/16/04	94		-13	3.43	54.5	3289
16	KTHU-4			9/16/04	94		-13	3.88	53.2	3290
17	KTHU-4			9/16/04	94		-13.5	3.45	54.4	3290
18	KTHU-4			9/16/04	94		-13	3.68	52.0	3290
19	KTHU-4			9/16/04	94		-18	-	-	-
20	KTHU-4			9/16/04	94		-10	3.58	52.6	3279
21	KTHU-4			9/16/04	94		-9	3.47	53.1	3289
22	KTHU-4			9/16/04	94		-13	3.67	54.2	3290
23	KTHU-4			9/16/04	94		-13	3.69	53.7	3290
24	KTHU-4			9/16/04	94		-13	3.48	52.9	3289
25	KTHU-4			9/16/04	94		-13	3.63	52.8	3279
26	KTHU-4			9/16/04	94		-13	3.49	54.5	3289
27	KTHU-4			9/16/04	94		-13	3.91	52.9	3290
28	KTHU-4			9/16/04	94		-17	3.45	54.3	3299
							Average	3.63	53.31	3271.89
							High	3.96	54.50	3299.00
							Low	3.40	52.00	2976.00
							SD	0.18	0.76	71.89

Test No.	Formulation	Date Rec'd (mm/dd/yy)	Date Loaded (mm/dd/yy)	Date Tested (mm/dd/yy)	Ambient Temperature (° F)	Conditioned Temperature (° F)	Estimated Primer Temp (° F)	Action Time (ms)	Pressure (ksi)	Velocity (fps)
1	PGU-27/B			10/7/04	85	NA	NA	2.49	50.2	3344
2	PGU-27/B			10/7/04	85		-10	2.49	49.5	3289
3	KTHU-5			10/7/04	85		-13	3.94	51.7	3289
4	KTHU-5			10/7/04	85		-14	4.07	51.7	3278
5	KTHU-5			10/7/04	85		-14.5	5.47	49.4	3278
6	KTHU-5			10/7/04	85		-13	4.37	52.1	3289
7	KTHU-5			10/7/04	85		-10	3.87	52.8	3300
8	KTHU-5			10/7/04	85		-10	6.24	49.9	3268
9	KTHU-5			10/7/04	85		-10	4.40	50.7	3278
10	KTHU-5			10/7/04	85		-13	4.60	52.2	3289
11	KTHU-5			10/7/04	85		-10	-	-	3257
12	KTHU-5			10/7/04	85		-13.5	4.77	49.2	3267
13	KTHU-5			10/7/04	85		-13	4.29	52.1	3289
14	KTHU-5			10/7/04	85		-13.5	4.44	46.8	3225
15	KTHU-5			10/7/04	85		-13	4.81	53.3	3300
16	KTHU-5			10/7/04	85		-13	29.72	52.9	3300
17	KTHU-5			10/7/04	85		-14	3.71	50.5	3278
18	KTHU-5			10/7/04	85		-14	5.73	49.9	3278
19	KTHU-5			10/7/04	85		-12.5	3.95	51.2	3289
20	KTHU-5			10/7/04	85		-13	5.28	48.7	3246
21	KTHU-5			10/7/04	85		-13	4.28	51.6	3289
22	KTHU-5			10/7/04	85		-10	4.32	53.6	3300
							Average	5.91	51.07	3279.35
							High	29.72	53.60	3300.00
							Low	3.71	46.80	3225.00
							SD	5.81	1.76	19.36

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M53			3/2/05	65	NA	NA	2.44	45.8	3313
2	M54			3/2/05	65	NA	NA	2.41	46.9	3337
3	M55			3/2/05	65	NA	NA	2.46	45.7	3324
4	KTHU-22			3/2/05	65	NA	NA	3.39	54.3	3371
5	KTHU-22			3/2/05	65	NA	NA	3.09	54.3	3375
6	KTHU-22			3/2/05	65	NA	NA	2.94	53.7	3362
7	KTHU-22			3/2/05	65	NA	NA	3.12	53.3	3372
8	KTHU-22			3/2/05	65	NA	NA	2.75	54.2	3369
9	KTHU-22			3/2/05	65	NA	NA	2.90	52.6	3369
10	KTHU-22			3/2/05	65	NA	NA	2.86	54.3	3383
11	KTHU-22			3/2/05	65	NA	NA	3.20	52.3	3362
							Average	3.03	53.63	3370.38
							High	3.39	54.30	3383.00
							Low	2.75	52.30	3362.00
							SD	0.21	0.81	6.84

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52			4/14/05	65	NA	NA	2.69	49.4	3340
2	M52			4/14/05	65	NA	NA	2.66	41.6	3283
3	KTHU-22			4/14/05	65	NA	NA	3.07	53.3	3209
4	KTHU-22			4/14/05	65	NA	NA	2.93	51.6	3362
5	KTHU-22			4/14/05	65	NA	NA	3.02	51.8	3189
6	KTHU-22			4/14/05	65	NA	NA	3.09	52.7	3362
7	KTHU-22			4/14/05	65	NA	NA	2.90	52.2	3195
8	KTHU-22			4/14/05	65	NA	NA	3.07	52.9	3213
9	KTHU-22			4/14/05	65	NA	NA	3.00	52.7	3362
10	KTHU-22			4/14/05	65	NA	NA	3.41	54.2	3380
11	KTHU-22			4/14/05	65	NA	NA	2.84	52.0	3355
12	KTHU-22			4/14/05	65	NA	NA	2.77	52.5	3360
13	KTHU-22			4/14/05	65	NA	NA	3.00	52.7	3372
14	KTHU-22			4/14/05	65	NA	NA	2.79	54.0	3374
15	KTHU-22			4/14/05	65	NA	NA	2.82	53.6	3372
16	KTHU-22			4/14/05	65	NA	NA	2.80	54.2	3369
17	KTHU-22			4/14/05	65	NA	NA	3.13	52.6	3358
18	KTHU-22			4/14/05	65	NA	NA	3.01	53.0	3369

Average	2.98	52.88	3325.06
High	3.41	54.20	3380.00
Low	2.77	51.60	3189.00
SD	0.16	0.81	74.13

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52A1B3			6/23/05	90	NA	NA			3450
2	M52A1B3			6/23/05	90	NA	NA	2.64	46.0	3429
3	M52A1B3			6/23/05	90	NA	NA	2.50	46.4	3366
4	KTHU-47			6/23/05	90	NA	NA	2.83	54.2	3474
5	KTHU-47			6/23/05	90	NA	NA	2.85	55.0	3463
6	KTHU-47			6/23/05	90	NA	NA	2.81	57.0	3473
7	KTHU-47			6/23/05	90	NA	NA	2.88	54.9	3444
8	KTHU-47			6/23/05	90	NA	NA	2.88	55.8	3453
9	KTHU-47			6/23/05	90	NA	NA	2.87	54.2	3433
10	KTHU-47			6/23/05	90	NA	NA	2.94	54.4	3445
11	KTHU-47			6/23/05	90	NA	NA	3.00	54.6	3455
12	KTHU-47			6/23/05	90	NA	NA	2.91	55.5	3460
13	KTHU-47			6/23/05	90	NA	NA	2.93	52.5	3434
14	KTHU-47			6/23/05	90	NA	NA	2.94	54.6	3445
15	KTHU-47			6/23/05	90	NA	NA	3.00	54.2	3446

Average	2.90	54.74	3452.08
High	3.00	57.00	3474.00
Low	2.81	52.50	3433.00
SD	0.06	1.08	13.49

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52A1B3			6/23/05	96	NA	NA			3450
2	M52A1B3			6/23/05	96	NA	NA	2.64	46.0	3429
3	M52A1B3			6/23/05	96	NA	NA	2.50	46.4	3366
4	KTHU-53			6/23/05	96	NA	NA	2.85	54.5	3436
5	KTHU-53			6/23/05	96	NA	NA	2.87	53.6	3439
6	KTHU-53			6/23/05	96	NA	NA	2.79	54.4	3455
7	KTHU-53			6/23/05	96	NA	NA	2.78	54.4	3444
8	KTHU-53			6/23/05	96	NA	NA	2.75	53.6	3446
9	KTHU-53			6/23/05	96	NA	NA	2.89	54.9	3458
10	KTHU-53			6/23/05	96	NA	NA	2.98	54.8	3429
11	KTHU-53			6/23/05	96	NA	NA	2.79	54.1	3434
12	KTHU-53			6/23/05	96	NA	NA	2.85	54.8	3437
13	KTHU-53			6/23/05	96	NA	NA	2.77	55.3	3444
14	KTHU-53			6/23/05	96	NA	NA	2.83	55.5	3419
15	KTHU-53			6/23/05	96	NA	NA	3.23	52.3	3410

Average	2.87	54.35	3437.58
High	3.23	55.50	3458.00
Low	2.75	52.30	3410.00
SD	0.13	0.87	13.72

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52A1B3			9/27/05	85	NA	NA	2.45	49.3	3396
2	M52A1B3			9/27/05	85	NA	NA	2.61	48.1	3360
3	M52A1B3			9/27/05	85	NA	NA	2.56	44.6	3383
4	KTHU-57			9/27/05	85	NA	NA	3.12	54.7	3387
5	KTHU-57			9/27/05	85	NA	NA	3.11	55.1	3390
6	KTHU-57			9/27/05	85	NA	NA	3.00	56.2	3387
7	KTHU-57			9/27/05	85	NA	NA	3.23	52.6	3385
8	KTHU-57			9/27/05	85	NA	NA	3.00	53.9	3376
9	KTHU-57			9/27/05	85	NA	NA	2.94	53.2	3385
10	KTHU-57			9/27/05	85	NA	NA	3.15	54.7	3378
11	KTHU-57			9/27/05	85	NA	NA	3.01	52.8	3390
12	KTHU-57			9/27/05	85	NA	NA	3.11	56.3	3397
13	KTHU-57			9/27/05	85	NA	NA	3.11	55.0	3385
14	KTHU-57			9/27/05	85	NA	NA	3.33	56.9	3398
							Average	3.10	54.65	3387.09
							High	3.33	56.87	3398.00
							Low	2.94	52.60	3376.00
							SD	0.11	1.43	6.73

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52A1B3			10/20/05	73	Ambient	-70	3.03	37.5	n/a
2	M52A1B3			10/20/05	73	Ambient	-70	2.92	41.0	n/a
3	M52A1B3			10/20/05	73	Ambient	-70	2.74	45.9	3329
4	KTHU-22			10/20/05	73	-65	-63	3.36	52.6	2800
5	KTHU-22			10/20/05	73	-65	-52	3.36	50.8	3276
6	KTHU-22			10/20/05	73	-65	-60	3.20	51.9	3274
7	KTHU-22			10/20/05	73	-65	-60	3.27	51.9	2815
8	KTHU-22			10/20/05	73	-65	-52	3.13	55.3	3320
9	KTHU-22			10/20/05	73	-65	-55	3.14	51.9	3268
10	KTHU-22			10/20/05	73	-65	-62	3.24	51.1	3270
11	KTHU-22			10/20/05	73	-65	-56	3.18	49.6	3259
12	KTHU-22			10/20/05	73	-65	-73	3.23	52.6	3282
13	KTHU-22			10/20/05	73	-65	-72	3.36	59.5	3283
14	KTHU-22			10/20/05	73	-65	-57	3.23	51.9	3286
15	KTHU-22			10/20/05	73	-65	-34	3.27	53.0	3304
16	KTHU-22			10/20/05	73	-65	-54	2.96	55.2	3307
17	KTHU-22			10/20/05	73	-65	-48	2.95	57.1	3298
18	KTHU-22			10/20/05	73	-65	-62	3.23	52.2	3267
19	KTHU-22			10/20/05	73	-65	-64	3.15	54.1	3275
20	KTHU-22			10/20/05	73	-65	-56	3.25	50.3	3280
21	KTHU-22			10/20/05	73	-65	-58	3.35	51.5	3281
22	KTHU-22			10/20/05	73	-65	-60	3.06	51.1	3255
23	KTHU-22			10/20/05	73	-65	-60	3.18	50.7	3251
						Average	-57.90	3.21	52.72	3232.55
						High	-34.00	3.36	59.50	3320.00
						Low	-73.00	2.95	49.60	2800.00
						SD	8.31	0.12	2.44	146.41

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52A1B3			10/24/05	73	-65	-65.1	2.70	49.2	3336
2	M52A1B3			10/24/05	73	-65	-62.2	2.79	46.1	3299
3	M52A1B3			10/24/05	73	-65	-60.2	2.78	44.7	3260
4	M52A1B3			10/24/05	73	-65	-71.1	2.80	47.3	3308
5	M52A1B3			10/24/05	73	-65	-70.2	2.81	47.7	3269
6	KTHW-24			10/24/05	73	-65	-68.5	3.24	53.0	3269
7	KTHW-24			10/24/05	73	-65	-57.7	4.66	52.6	3290
8	KTHW-24			10/24/05	73	-65	-63.4	3.81	55.6	3309
9	KTHW-24			10/24/05	73	-65	-64.6	5.38	53.8	3291
10	KTHW-24			10/24/05	73	-65	-65.9	4.55	53.8	3302
11	KTHW-24			10/24/05	73	-65	-64.4	3.22	53.0	3267
12	KTHW-24			10/24/05	73	-65	-67.5	3.92	53.0	3300
13	KTHW-24			10/24/05	73	-65	-65.7	5.70	53.4	3296
14	KTHW-24			10/24/05	73	-65	-70.6	4.31	53.8	3287
15	KTHW-24			10/24/05	73	-65	-65.3	3.29	53.0	3270
16	KTHW-24			10/24/05	73	-65	-70.1	6.63	51.5	3272
17	KTHW-24			10/24/05	73	-65	-62.9	4.40	51.1	3264
18	KTHW-24			10/24/05	73	-65	-65.6	3.31	52.3	3244
19	KTHW-24			10/24/05	73	-65	-47	4.59	35.8	3283
20	KTHW-24			10/24/05	73	-65	-66.9	3.63	51.5	3271
21	KTHW-24			10/24/05	73	-65	-61	6.54	54.1	3289
22	KTHW-24			10/24/05	73	-65	-62.9	4.03	53.0	3287
23	KTHW-24			10/24/05	73	-65	-69.6	4.02	52.6	3263
24	KTHW-24			10/24/05	73	-65	-75.6	5.23	52.3	3221
25	KTHW-24			10/24/05	73	-65	-60.3	4.80	52.8	3290
						Average	-64.78	4.46	52.10	3278.25
						High	-47.00	6.63	55.60	3309.00
						Low	-75.60	3.22	35.80	3221.00
						SD	5.81	1.02	3.97	20.88

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52A1B3			1/24/06	73	Amb	-65	n/a	n/a	3212
1	M52A1B3			1/24/06	73	Amb	-19	2.93	51.3	3203
6	KTHW-32			1/24/06	73	-65	-57	3.87	50.8	3263
7	KTHW-32			1/24/06	73	-65	-52	3.90	49.8	3252
8	KTHW-32			1/24/06	73	-65	-60	13.60	53.8	3288
9	KTHW-32			1/24/06	73	-65	-61	4.73	57.0	3253
10	KTHW-32			1/24/06	73	-65	-60	4.40	48.8	3236
11	KTHW-32			1/24/06	73	-65	-63	12.20	54.1	3290
12	KTHW-32			1/24/06	73	-65	-55	13.40	52.1	3277
13	KTHW-32			1/24/06	73	-65	-65	n/a	n/a	3276
14	KTHW-32			1/24/06	73	-65	-58	11.50	52.8	3279
15	KTHW-32			1/24/06	73	-65	-55	223.90	61.0	3229
16	KTHW-32			1/24/06	73	-65	57	4.70	55.5	3378
17	KTHW-32			1/24/06	73	-65	58	6.67	63.9	3358
18	KTHW-32			1/24/06	73	-65	61	3.49	66.9	3389
19	KTHW-32			1/24/06	73	-65	63	3.84	56.1	3373
20	KTHW-32			1/24/06	73	-65	64	3.42	58.2	3394
21	KTHW-32			1/24/06	73	-65	66	3.58	58.4	3361
22	KTHW-32			1/24/06	73	-65	67	3.79	57.0	3341
23	KTHW-32			1/24/06	73	-65	68	8.56	57.7	3290
24	KTHW-32			1/24/06	73	-65	69	3.30	58.1	3367
25	KTHW-32			1/24/06	73	-65	70	3.41	59.8	3389
					COLD	Average	-58.60	32.39	53.36	3264.30
						High	-52.00	223.90	61.00	3290.00
						Low	-65.00	3.87	48.80	3229.00
						SD	3.98	71.94	3.78	21.25
					Ambient	Average	64.30	4.48	59.16	3364.00
						High	70.00	8.56	66.90	3394.00
						Low	57.00	3.30	55.50	3290.00
						SD	4.52	1.76	3.57	30.71

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52A1B3			3/6/06	73	Amb	73	2.57	44.0	3321
2	M52A1B3			3/6/06	73	Amb	73	2.62	n/a	3316
6	KTHW-36			3/6/06	73	-65	-58	3.35	56.4	3317
7	KTHW-36			3/6/06	73	-65	-58	3.38	56.9	3300
8	KTHW-36			3/6/06	73	-65	-64	3.97	57.9	3306
9	KTHW-36			3/6/06	73	-65	-59	4.66	57.2	3301
10	KTHW-36			3/6/06	73	-65	-65	6.06	58.1	3321
11	KTHW-36			3/6/06	73	-65	-62	3.92	59.4	3331
12	KTHW-36			3/6/06	73	-65	-60	3.86	58.1	3301
13	KTHW-36			3/6/06	73	-65	-62	7.46	60.7	3321
14	KTHW-36			3/6/06	73	-65	-57	4.08	58.1	3305
15	KTHW-36			3/6/06	73	-65	-69	3.66	58.3	3302
16	KTHW-36			3/6/06	73	-65	-69	3.52	60.4	3330
17	KTHW-36			3/6/06	73	-65	-59	3.52	58.8	3319
18	KTHW-36			3/6/06	73	-65	-67	3.59	58.8	3317
						Average	-62.23	4.23	58.39	3313.15
						High	-57.00	7.46	60.70	3331.00
						Low	-69.00	3.35	56.40	3300.00
						SD	4.23	1.21	1.25	11.18

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	KTHW-71			7/1/06	95	-65	-53.2	5.36	57.4	3199
2	KTHW-71			7/1/06	95	-65	-76.6	4.95	49.5	3263
3	KTHW-71			7/1/06	95	-65	-64	4.99	50.5	3166
4	KTHW-71			7/1/06	95	-65	-64.6	3.81	50.6	3245
5	KTHW-71			7/1/06	95	-65	-44	4.47	51.4	3194
6	KTHW-71			7/1/06	95	-65	-85	4.85	55.5	3238
8	KTHW-71			7/1/06	95	-65	-53.4	28.18	54.0	3248
9	KTHW-71			7/1/06	95	-65	-92	4.85	56.0	3270
12	KTHW-71			7/1/06	95	-65	-67.2	5.07	52.4	3160
13	KTHW-71			7/1/06	95	-65	-50.8	4.58	50.0	3180
15	KTHW-71			7/1/06	95	-65	-65	5.10	54.0	3249
						Average	-65.07	6.93	52.85	3219.27
						High	-44.00	28.18	57.40	3270.00
						Low	-92.00	3.81	49.50	3160.00
						SD	14.78	7.06	2.69	40.19

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
3	KTHW-73			7/1/06	95	-65	-57.8	55.70	41.0	3315
4	KTHW-73			7/1/06	95	-65	-75	74.84	40.6	3176
5	KTHW-73			7/1/06	95	-65	-53.4	6.19	36.1	3247
9	KTHW-73			7/1/06	95	-65	-60.7	60.64	46.7	3165
10	KTHW-73			7/1/06	95	-65	-65.5	69.27	49.1	3154
						Average	-62.48	53.33	42.69	3211.40
						High	-53.40	74.84	49.05	3315.00
						Low	-75.00	6.19	36.11	3154.00
						SD	8.27	27.38	5.17	68.38

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
2	KTHW-74			6/28/06	95	-65	-62	44.60	53.7	3329
3	KTHW-74			6/28/06	95	-65	-49	44.60	56.0	3263
4	KTHW-74			6/28/06	95	-65	-77.4	51.40	55.0	3218
5	KTHW-74			6/28/06	95	-65	-45	5.40	63.9	3226
8	KTHW-74			6/28/06	95	-65	-61.8	45.68	52.0	3214
9	KTHW-74			6/28/06	95	-65	-51.2	60.52	53.0	3192
11	KTHW-74			6/28/06	95	-65	-70.6	32.73	56.6	3273
12	KTHW-74			6/28/06	95	-65	-65	63.28	52.0	3283
						Average	-60.25	43.53	55.28	3249.75
						High	-45.00	63.28	63.90	3329.00
						Low	-77.40	5.40	52.00	3192.00
						SD	11.15	18.20	3.89	45.18