

Full Paper

Applications of High-Nitrogen Energetics in Pyrotechnics: Development of Perchlorate-Free Red Star M126A1 Hand-Held Signal Formulations with Superior Luminous Intensities and Burn Times

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Abstract

Perchlorate-free hand-held signal illuminant formulations for the M126A1 red star parachute have been developed. The formulations exhibited longer burn times and higher luminous intensities compared to the US Army in-service M126A1 formulation. The perchlorate-free formulations derive their enhanced performance from the inclusion of strontium *bis*-(1-methyl-5-nitriminotetrazolate) monohydrate, the choice of magnesium used, and replacing a polyester binder system with an epoxy binder system.

Keywords: Energetic Materials, Environmentally Friendly, High-Nitrogen, Illuminant Compositions, Pyrotechnics

1 Introduction

Hand-held signals (HHS) are used in signaling troop movements and aircraft. They are meant to attract attention day or night, and serve as a beacon for rescuers to identify the positions of military personnel. HHS technologies find use in both training exercises and combat situations, and improvements are being sought to advance existing HHS technology.

Although perchlorates are superior oxidizers with large positive oxygen balances, environmental and human health concerns are associated with these oxidizers. Potassium perchlorate, a widely used oxidizer in pyrotechnics, has a high solubility in water, thus contaminating ground-water and endangering public drinking supplies. Although

once believed to rank amongst the ideal oxidizers with respect to cost, low hygroscopicity, good stability, and low toxicity, perchlorates have recently been found to be teratogenic, and the anion has been found to compete with iodide anion in binding with the thyroid gland, interfering with the production and regulation of thyroid hormones [1]. According to Conkling and Mocella [2], this binding effect appears to be preventable and reversible, as the intake of iodide-containing food and beverages appears to lead to a replacement of the perchlorate anion with iodide anion. Nonetheless, the US Environmental Protection Agency has recently set the permissible perchlorate level to 15 parts per billion (ppb) [3], while the states of California and Massachusetts have further reduced their respective permissible perchlorate levels to 6 and 2 ppb [4]. As a result of these regulations, the US Department of Defense spends billions of dollars annually in cleanup and legal fees associated with perchlorate decontamination, and the use of perchlorate-containing munitions on continental United States (CONUS) and outside the continental United States (OCONUS) training ranges has been limited and/or prohibited.

To address the aforementioned perchlorate issues, an effort was initiated by Armament Research, Development, and Engineering Center (ARDEC) to eliminate KClO_4 from the M126A1 red star parachute. To maximize the performance of the new formulations, KClO_4 was replaced with strontium *bis*-(1-methyl-5-nitriminotetrazolate) monohydrate (**1**). Preliminary toxicity studies

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have been performed on 5-aminotetrazole, a precursor to *bis*-tetrazolate **1** [5]. The toxicity of 5-aminotetrazole was compared to that of 3-aminotriazole, a thyroid carcinogen in rats, and a structural analog of 5-aminotetrazole. Although 5-aminotetrazole is structurally similar to 3-aminotriazole, it did not possess the primary undesirable effects observed with short-term administration of 3-aminotriazole. The US Army Public Health Command has stated that quantitative structure activity relationship (QSAR) modeling is not a useful tool in determining the toxic nature of high nitrogen salts such as *bis*-tetrazolate **1**. Therefore, no data on the toxicology of *bis*-tetrazolate **1** exists at this time. However, the US Army Public Health Command is working to acquire such data on this compound and other high-nitrogen compounds by performing Ames mutagenicity tests and *in vitro* tests to measure acute toxicity.

The use of high-nitrogen compounds for energetic purposes has gained interest in recent years [6], and the remarkable stabilities of these compounds have been demonstrated by Klapötke and co-workers. In addition to developing perchlorate-free HHS formulations, the decision was made to simultaneously increase the burn time and luminous intensity of the formulations in an effort to develop performance-enhanced HHS illuminants to benefit the warfighter. Although burn time and luminous intensity values are known to have an inverse relationship, it was believed that simultaneous increase in burn time and luminous intensity could be obtained using a multi-dimensional approach.

2 Experimental Part

2.1 Materials

Mg 30/50 and Mg 50/100 were purchased from Reade. KClO_4 , $\text{Sr}(\text{NO}_3)_2$, and PVC were purchased from Hummel Croton. Laminac 4116 was purchased from Ashland Chemical, Inc. Lupersol was purchased from Norac. Epon 813 was purchased from Hexion Specialty Chemicals. Versamid 140 was purchased from Cognis. Strontium *bis*-(1-methyl-5-nitriminotetrazolate) monohydrate (**1**) was synthesized in-house (Picatinny Arsenal, NJ) by a proprietary procedure. Formulations were pressed into non-coated Kraft fiberboard tubes, obtained from Security Signals, Inc.

2.2 Preparation of M126A1 Formulations

Batches of 20 g mixes were prepared by weighing out the chemicals according to their weight percentages in the formulations. After drying the chemicals in the oven overnight at 60 °C, they were introduced to a binder system (95 % Laminac 4116/5 % Lupersol or 80 % Epon 813/20 % Versamid 140), and the mixture was hand-blended for 20 min. Laminac 4116/Lupersol-based formulations were dried in the oven overnight, and Epon813/Versamid 140-based formulations were dried in air for 2–3 h at ambient temperature before consolidation.

Formulations were weighed out in two 3-g increments, and were pressed into non-coated Kraft fiberboard tubes (length of 4.93 cm, inner diameter of 0.84 cm) with the aid of a tooling die (inner diameter of 1.27 cm, height of 5.08 cm), and a manual hand press at a consolidation dead load of 287 kg. Between 5.91 and 6.04 g of energetic material was used per pellet, and 6–7 pellets were tested for each formulation. The top of each pellet was coated with a thin layer of proprietary first-fire composition. The pellets were fired in a light tunnel by means of an electric match with an energy source of 2 V.

2.3 Characterization

For each iteration, the performances of the formulations were compared to a perchlorate-containing baseline. Optical emissive properties of these formulations were characterized using both a single element photopic light detector and a 2048 element optical spectrometer. The light detector used was manufactured by International Light and is composed of an SED 033 silicon detector (33 mm² area silicon detector with quartz window) coupled to a photopic filter (Y-filter) and a field of view limited hood (H-hood). The current output of the detector was converted to voltage using a DL Instruments 1211 transimpedance amplifier. Voltage output was collected and analyzed from the amplifier using a NI-6115 National Instruments data card and in-house developed Labview™ based data acquisition and analysis software.

The optical emissive spectrum of each sample was measured using a 2048 element Ocean Optics HR2000 spectrometer coupled to a 400 µm core optical fiber. The dominant wavelength and spectral purity was measured based on the 1931 CIE method using illuminant C as the white reference point. The spectrometer was calibrated using both an Hg–Ar light source (Ocean Optics HG-1 wavelength standard) and a calibrated tungsten light source (Ocean Optics LS-1-Cal). The average dominant wavelength and spectral purity based on the full burn of the sample was calculated.

3 Results and Discussion

The formulation components and weight percentages of the US Army's perchlorate-containing in-service M126A1 illuminant composition is summarized in Table 1.

Table 1. Composition of the US Army in-service M126A1 formulation.

Component	Weight %
Strontium Nitrate	39.3
Magnesium 50/100	14.7
Magnesium 30/50	14.7
PVC	14.7
Potassium Perchlorate	9.8
Laminac 4116/Lupersol	6.8

$\text{Sr}(\text{NO}_3)_2$ and KClO_4 served as the oxidizers, while Mg 30/50 and Mg 50/100 were the main fuel sources in the formulation. Magnesium engaged in redox reactions with the oxidizers to form MgO in a highly exothermic process. MgO is a gray body emitter and was largely responsible for the thermal energy needed to stimulate the system (i.e. promote valence electrons to the excited state) to produce red light.

Polyvinyl chloride (PVC) is a low-energy fuel, and its primary function was to serve as a chlorine donor/color enhancer through the liberation of chlorine during the combustion process. Another reason for the utilization of PVC was the volatilization of incandescent MgO by transforming it to the more volatile MgCl [6a]. When strontium and chlorine reacted in the excited state, metastable strontium chloride (SrCl) was produced. SrCl was responsible for the brilliant red flame exhibited during combustion of the pyrotechnic material, though formation of metastable strontium hydroxide (SrDH) was also a minor contributor to red flame illumination. Laminac 4116/Lupersol served as a binder system in the formulation, which provided mechanical strength, prevented segregation amongst oxidizers and fuels during powder consolidation, reduced the impact, friction, and ESD sensitivities of formulations, thus mitigating potential safety hazards.

Studies toward the development of improved HHS illuminants commenced with a “drop-in” experiment of the baseline formulation in which KClO_4 was replaced with strontium *bis*-(1-methyl-5-nitriminotetrazolate) monohydrate (**1**) (Figure 1). *Bis*-tetrazolate **1** was previously evaluated in perchlorate-free illuminant compositions and was determined to be a suitable perchlorate replacement due to its colorant properties, its high thermal stability, and its low sensitivities to impact, friction, and electrostatic discharge [7]. The performance of “drop-in” formulation **A** against the baseline formulation is summarized in Table 2. Formulation **A** exhibited a faster burn time, likely due to the presence of *bis*-tetrazolate **1** which increased the overall energy of the formulation. Although

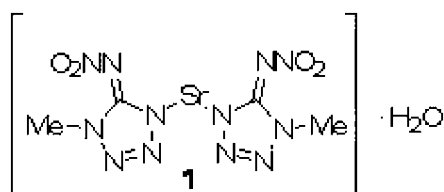


Figure 1. Strontium *bis*-(1-methyl-5-nitriminotetrazolate) monohydrate.

formulation **A** had a larger luminous intensity than the baseline, this was expected since pyrotechnics with shorter burn times typically burn brighter. Both formulations exceeded the minimum spectral purity requirement of 76% by a large margin.

To increase the burn time of the perchlorate-free illuminants, the weight percentages of Laminac 4116/Lupersol binder system were increased incrementally from 6.8 to 12%. Although higher binder percentages resulted in marginal increases in burn time, all formulations failed to meet the burn time of the baseline, and propagation was inconsistent at higher binder levels. Therefore, increasing the weight percentages of Laminac 4116/Lupersol binder system was not a viable way to increase the burn time.

To extend burn times, further modifications were made to perchlorate-free formulation **A**. The baseline formulation and formulation **A** used a 1:1 ratio of two types of magnesium in its formulation; Mg 30/50 and Mg 50/100. Since Mg 30/50 is composed of larger particle sizes than Mg 50/100, it was believed that doubling the amount of Mg 30/50, while eliminating Mg 50/100 from the formulation would be beneficial (Table 3).

The result of using Mg 30/50 as the sole magnesium fuel source (formulation **B**) is summarized in Table 4. Although still short in meeting the burn time of the baseline formulation, the coarser particle size of Mg 30/50 resulted in a significant increase in the burn time. Although the burn time needed further improvement, using Mg 30/50 as the only magnesium source in future formulation development made the most sense in achieving this goal.

Since longer burn times were needed in meeting baseline requirements, the binder system in the formulation again came into focus. Although increasing the percentage of Laminac 4116/Lupersol binder system was unsuccessful in prolonging the burn time to the baseline value, recent studies at ARDEC demonstrated that replacing Laminac 4116/Lupersol binder system with Epon 813/Versamid 140 binder system resulted in prolonged burn times for a variety of high-nitrogen containing formulations [8]. Therefore, changing the binder system was believed to be a sensible approach in prolonging the burn time to desirable levels. Formulation **C**, with Epon 813/Versamid 140 as the binder system, is outlined in Table 5.

Table 3. Composition of formulation **B**.

Component	Weight %
Strontium Nitrate	39.3
Magnesium 30/50	29.4
PVC	14.7
<i>Bis</i> -Tetrazolate 1	9.8
Laminac 4116/Lupersol	6.8

Table 2. “Drop-in” experiment of the M126A1 HHS.

Formulation	Burn Time (s)	Luminous Intensity (cd)	Dominant Wavelength (nm)	Spectral Purity (%)
Baseline	29.3	574.0	617.0	94.0
A	22.3	849.4	616.1	95.7

Table 4. Effect of using Mg 30/50 as the sole metallic fuel source.

Formulation	Burn Time (s)	Luminous Intensity (cd)	Dominant Wavelength (nm)	Spectral Purity (%)
Baseline	29.3	574.0	617.0	94.0
B	26.2	728.0	617.4	95.2

Table 5. Composition of formulation C.

Component	Weight %
Strontium Nitrate	39.3
Magnesium 30/50	29.4
PVC	14.7
Bis-Tetrazolate 1	9.8
Epon 813/Versamid 140	6.8

As summarized in Table 6, there was a substantial increase in burn time when Epon 813/Versamid 140 binder system was used. Formulation C burned 50 % longer than formulation B (and burned 23 % longer than the baseline), and while the luminous intensity of formulation C was lower than formulation B, it exceeded the luminous intensity of the baseline. Despite replacing Laminac 4116/Lupersol binder system with Epon 813/Versamid 140 binder system, the required dominant wavelength (military specification of 620 ± 20 nm) and spectral purity values (military specification of 76 % minimum) of formulation C were achieved. Therefore, formulation C was deemed to be a suitable perchlorate-free formulation in replacing the US Army's in-service M126A1 formulation.

Given the success in exceeding burn time and luminous intensity values of the baseline formulation, a series of other formulations were developed to optimize these values. Of interest was determining how altering the

$\text{Sr}(\text{NO}_3)_2$ /bis-tetrazolate 1 ratio and the Mg 30/50/bis-tetrazolate 1 ratio would affect these values in the formulations. For formulations C–E, $\text{Sr}(\text{NO}_3)_2$ and bis-tetrazolate 1 weight percentages were altered in 3 % increments (Table 7).

The performances of formulations C–E are summarized in Table 8. Formulation C afforded the longest burn time and the lowest luminous intensity, while formulation E provided the shortest burn time and the highest luminous intensity value. Formulations C–E exceeded the performance of the perchlorate-containing baseline.

As Table 8 indicates, burn times decreased and luminous intensities increased with larger weight percentages of $\text{Sr}(\text{NO}_3)_2$ and smaller weight percentages of bis-tetrazolate 1. Although a high-energy fuel, bis-tetrazolate 1 is oxygen negative, with an oxygen balance of approximately –34 % [6]. Presumably, bis-tetrazolate 1 competed with Mg 30/50 for available oxygen during the combustion process. At lower weight percentages of bis-tetrazolate 1 and higher weight percentages of $\text{Sr}(\text{NO}_3)_2$, more oxygen was available to react with magnesium to produce MgO , thus raising observed visible light output and reducing the burn time. Conversely, at higher weight percentages of bis-tetrazolate 1 and lower weight percentages of $\text{Sr}(\text{NO}_3)_2$, less oxygen was available to react with magnesium to produce MgO , thus leading to an extended burn time and compromising the observed visible light output.

Table 6. Effect of using Epon 813/Versamid 140 as the binder.

Formulation	Burn Time (s)	Luminous Intensity (cd)	Dominant Wavelength (nm)	Spectral Purity (%)
Baseline	32.2	423.5	623.0	86.4
C	39.4	544.3	620.6	89.1

Table 7. Composition of formulations C–E.

Formulation C		Formulation D		Formulation E	
Component	Weight %	Component	Weight %	Component	Weight %
$\text{Sr}(\text{NO}_3)_2$	39.3	$\text{Sr}(\text{NO}_3)_2$	42.3	$\text{Sr}(\text{NO}_3)_2$	45.3
Mg 30/50	29.4	Mg 30/50	29.4	Mg 30/50	29.4
PVC	14.7	PVC	14.7	PVC	14.7
Bis-Tetrazolate 1	9.8	Bis-Tetrazolate 1	6.8	Bis-Tetrazolate 1	3.8
Epon 813/Versamid 140	6.8	Epon 813/Versamid 140	6.8	Epon 813/Versamid 140	6.8

Table 8. Effect of altering the $\text{Sr}(\text{NO}_3)_2$ oxidizer/bistetrazole fuel ratio.

Formulation	Burn Time (s)	Luminous Intensity (cd)	Dominant Wavelength (nm)	Spectral Purity (%)
Baseline	32.2	423.5	623.0	86.4
C	39.4	544.3	620.6	89.1
D	37.9	568.8	620.0	89.0
E	35.3	664.7	619.6	89.4

Table 9. Formulations **C**, **F**, and **G**.

Formulation C		Formulation F		Formulation G	
Component	Weight %	Component	Weight %	Component	Weight %
Sr(NO ₃) ₂	39.3	Sr(NO ₃) ₂	39.3	Sr(NO ₃) ₂	39.3
Mg 30/50	29.4	Mg 30/50	32.4	Mg 30/50	35.4
PVC	14.7	PVC	14.7	PVC	14.7
<i>Bis</i> -Tetrazolate 1	9.8	<i>Bis</i> -Tetrazolate 1	6.8	<i>Bis</i> -Tetrazolate 1	3.8
Epon 813/Versamid 140	6.8	Epon 813/Versamid 140	6.8	Epon 813/Versamid 140	6.8

Table 10. Effect of altering Mg 30/50 fuel/high-nitrogen fuel ratio.

Formulation	Burn Time (s)	Luminous Intensity (cd)	Dominant Wavelength (nm)	Spectral Purity (%)
Baseline	32.2	423.5	623.0	86.4
C	39.4	544.3	620.6	89.1
F	36.4	666.5	618.7	90.0
G	35.6	812.8	618.2	90.6

For formulations **C**, **F**, and **G**, Mg 30/50 and *bis*-tetrazolate **1** weight percentages were altered in 3% increments (Table 9) and the performance of these formulations is summarized in Table 10. All formulations exceeded the performance of the perchlorate-containing baseline. A decrease in burn times and an increase in luminous intensities were observed with higher percentages of Mg 30/50 and lower percentages of *bis*-tetrazolate **1**. Again, it appeared that lower weight percentages of oxygen negative *bis*-tetrazolate **1** fuel allowed more oxygen to react with magnesium to produce greater quantities of MgO, leading to shorter burn times and larger luminous intensity values. Furthermore, it has been well established that magnesium-rich illuminating candles are typically associated with shorter burn times and larger visible light outputs [9]. The increase in thermal conductivity of the metal-rich system leads to an increased rate of propagation, and excess magnesium is known to undergo air oxidation in its vapor state, leading to increases in reaction rate and visible light output.

Formulation **G** was of particular interest. Compared to the perchlorate-containing baseline, formulation **G** burned 10% longer with a 92% increase in luminous intensity. In examining formulations that exceeded the performance of the baseline, formulation **C** had the longest burn time and the lowest luminous intensity, while formulation **G** had the fastest burn time and the highest luminous intensity. These two formulations represent both extremes of the burn time/luminous intensity spectrum, and they will be subjected to full-up prototype testing (90 g candles).

The military specifications of the 90 g US Army in-service M126A1 illuminant are a minimum burn time of 50 s, a minimum luminous intensity of 10,000 candela, a dominant wavelength of 620 ± 20 nm and a minimum spectral purity of 76%. On small-scale (6 g candles), formulations **C** and **G** had reasonable dominant wavelengths and exceeded the burn time, luminous intensity, and spectral purity of the in-service illuminant. The 6 g candles

had a length of 4.93 cm and a spherical surface area of 2.21 cm², while the 90 g candles will have a length of 8.15 cm and a spherical surface area of 30.50 cm². Due to the longer length and larger spherical surface area of the 90 g illuminant tube, the full-up formulations are expected to burn significantly longer and brighter than the 6 g illuminating candles. Compared to small-scale formulation development, the burning behavior (top-down), the material of the tube encasing the consolidated pyrotechnic formulation (Kraft non-coated fiberboard), and the loading pressure and dwell time used to consolidate the pyrotechnic formulations relative to the diameter of the illuminant tube will remain the same at the full-up level. During small-scale formulation development, comparing the performances of all new formulations to the in-service baseline formulation was essential to reliably extrapolate their performances at the full-up level. Because the performance of formulations **C** and **G** comfortably exceeded the baseline formulation on small-scale, it is relatively safe to assume that the performance of these formulations will also exceed the baseline formulation at the full-up level. The results of this prototyping study will be reported in due course.

4 Conclusion

Perchlorate-free formulations with enhanced luminous intensities and burn times have been developed for the M126A1 red star parachute HHS by tuning the optical properties of pyrotechnic formulations. The use of *bis*-tetrazolate **1** to replace KClO₄ oxidizer was beneficial because while serving as a high-energy fuel, it aided in prolonging burn times, while contributing to the overall energy of the pyrotechnic formulations. The discovery of longer and brighter burning M126A1 formulations is significant because it may aid the future development of smaller HHS munitions without compromising the required values for burn time and luminous intensity. Small-

er HHS munitions would provide significant tactical advantages to the warfighter such as reducing their bulk load to be carried while enhancing survivability and maneuverability during combat operations.

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