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AFATL-TR-67-214

LIQUID VISUAL FLARE

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Ordnance Research Incorporated

TECHNICAL REPORT AFATL-TR-67-214
DECEMBER 1967

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AIR FORCE ARMAMENT LABORATORY
AIR FORCE SYSTEMS COMMAND • UNITED STATES AIR FORCE
EGLIN AIR FORCE BASE, FLORIDA

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FOREWORD

This final report is submitted by Ordnance Research Incorporated, Fort Walton Beach, Florida, in accordance with Contract F08635-67-C-0019 for determination of the feasibility of utilizing a monopropellant fuel-oxidizer liquid slurry for tactical illumination applications.

This effort was conducted in support of Exploratory Development Project Number 2563, "Tactical Illumination", Task 01, Pyrotechnic Illumination.

Work was begun on this contract 16 September 1966 and was completed September 1967.

The United States Air Force Program monitor is Mr. Lawrence W. Moran, AFATL (ATTI), Eglin Air Force Base, Florida.

Very helpful information was obtained from the technical assistance of Messrs. Lawrence W. Moran and William S. Cronk of AFATL (ATTI), Eglin Air Force Base, Florida.

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This technical report has been reviewed and is approved.

AJClipper

A. J. CLIPPER, [illegible]. Colonel, USAF
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ABSTRACT

An exploratory development program was accomplished to analytically and experimentally determine feasibility of developing a liquid visual flare for tactical battlefield illumination. The effort described in this report included literature search, study, analysis, and experiments with various liquid flare compositions, various burner configurations, and extrusion techniques. The liquid visual flare concept was found feasible for both illumination and for target marking since the flexibility afforded by the mechanical flow rate provides a choice of a high powered flare with several million candlepower (intensity) for 2 to 5 minutes or a long burning target marking device up to 30 minutes duration. A monopropellant liquid slurry system was selected for engineering simplicity. The liquid slurry uses atomized magnesium or other metal as a fuel and uses an oxidizer based on the partial solubility of inorganic perchlorates in polyfunctional alcohols and alcohol amines. The liquid flare concept provides approximately the same output power in candle-seconds per gram of composition as solid grain magnesium-sodium nitrate flares and also possesses two distinct advantages over solid flares in that it has been proven relatively insensitive to ballistic impact from small arms fire and its time duration and intensity can be controlled over a period of 2 minutes to 30 minutes by the mechanical flow rate. The next logical step should be the development of a battlefield illumination device and a target marking device utilizing the liquid visual flare concept.

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SECTION I

INTRODUCTION

For twenty years the standard visual illuminating composition has been a pressed grain of atomized magnesium, powdered sodium nitrate, and one of several organic binders. Improvements in the use of this composition have been limited to the investigation of alternate case materials and quality control procedures to achieve reproducibility in burning time and improve long-term storage stability. The inflexibility of this fabrication technique is apparent in the manufacturing difficulty of the present BRITEYE illuminating round. The present program shows the feasibility of an alternate high-efficiency illumination composition based on a liquid pyrotechnic system of great flexibility. The consumption rate of this latter composition, and the candlepower output, are determined by a mechanical pumping rate rather than by grain dimensions. Special configurations for enhanced output are available through design of the liquid slurry burner; the composition is not limited to burning on one or more exposed faces of a solid grain. Most important to rapid tactical use of this new composition, no specialized facilities are required for blending and loading.

The increased development effort and subsequent tactical utilization of large diameter illumination flares has increased the interest in methods to maintain the luminous efficiency of the standard magnesium-sodium nitrate composition with increase in flare diameter. A marked decrease in the luminous efficiency results as the optical density of the plume increases and the plume becomes opaque and a surface radiator. Conduction and convection losses lower the downstream plume temperature and the absorbed energy is not recovered as visual radiation. Plume shaping to decrease optical density and increase radiating area is difficult with a solid grain. High temperature gases containing condensed species are highly erosive to channels and orifices required to direct the exhaust gases into an extended area plume. The liquid system allows the extrusion of a slurry or the pumping of an air-atomized slurry in the desired plume configuration prior to ignition and combustion. Heat transfer to the flare structure is markedly reduced for stable long term operation. An extended flare plume may be formed with maximum presented area to the target for illumination.

The safety of pressed visual flares, both in fabrication and tactical use, is limited by the explosive deflagration of the grain when an extended burning surface area is exposed. This can result from bullet or fragment impact. The liquid slurry cannot suffer gross fracture in this way.

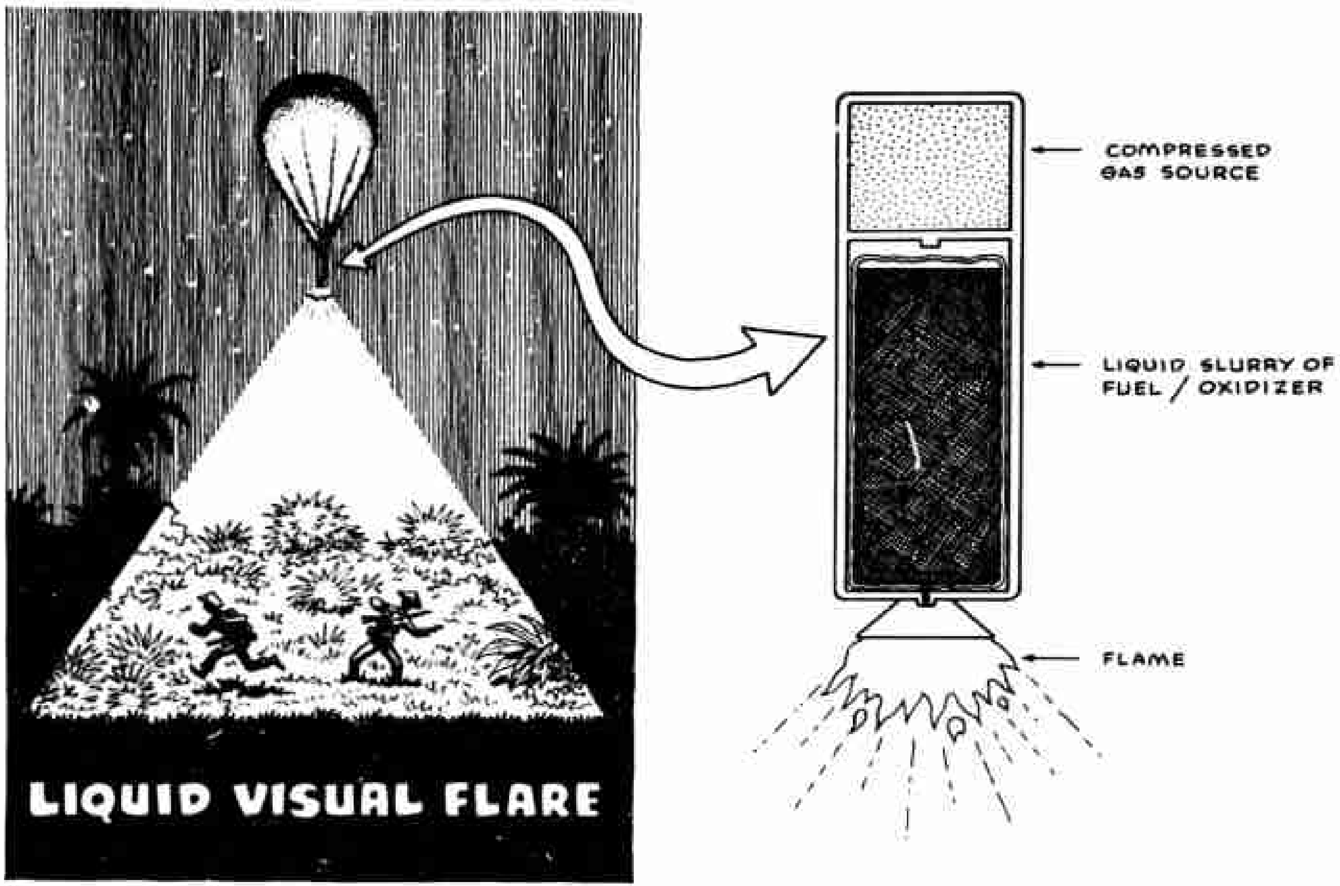


Figure 1. Liquid Visual Flare

The liquid slurry, incorporating both fuel and oxidizer, is held in tankage volume. Gel structure prevents separation of the suspended solids with flow under shear stress by force against a piston or diaphragm. This force, in the range of 40 psi. may be produced by compressed gas, a pyrotechnic gas generator, or mechanical or spring drive. An igniter is required to initiate slurry combustion. The design of the slurry burner is dependent on the item functional requirements. Extrusion through an orifice into a simple chamber which supports the combusting slurry is adequate for low mass flow rate marking round function (up to 30 minutes duration). In order to stabilize the combustion at the high mass flow rates required for illumination applications, and to shape the plume for optimum radiant energy delivery, a more complicated burner assembly is required.

The advantage of engineering simplicity led to the initial development of a monopropellant rather than a bipropellant liquid slurry system, although the latter technique allows the use of more energetic reactants. The oxidizer system is based on the partial solubility of inorganic perchlorates in polyfunctional alcohols and alcohol amines. This decreases the insoluble particle loading and improves wetting of the suspended solids by the liquid carrier for improved slurry rheological behavior. Atomized magnesium or other metal is incorporated as the fuel. Thus, the illumination characteristics approximate those of the familiar pressed magnesium-sodium nitrate.

The combustion rate, and the candle power output, are controlled mechanically by the rate of pumping or extrusion of the liquid. With a suitable alteration of burner configuration, tankage volume may be used for marking round, or low - or high - level illumination applications. Luminous efficiency for marking round operation has been measured at 40,000 candle-seconds per gram. This efficiency may be increased in the radial burner configuration by both plume shaping effects and liquid carrier volatilization prior to combustion.

SECTION II

EXPERIMENTAL EFFORT

In contrast to liquid rocket propellant technology which is based on the bipropellant system of separate liquid fuel and oxidizer mixed and combusted at an injector face in a pressurized chamber, the liquid flare concept utilizes a single fluid monopropellant incorporating both fuel and oxidizer components. The tankage and pumping requirements are thus simplified over the bipropellant case and there is no metering of flow to maintain the desired fuel-oxidizer ratio. The monopropellants are gelled metallized slurries incorporating a soluble oxidizer to reduce solids loading.

Because of lack of published information on liquid or slurry type of pyrotechnic materials applicable to the research study undertaken, initial investigations began with evaluation of basic information on liquid carriers which dissolve partially or entirely perchlorate oxidizers, thereby increasing solids to liquid loading ratios. This will yield slurry systems capable of sustained combustion and also capable of being extruded through various nozzle openings, facilitating the design of nozzles (burners) so that both plume size and plume configuration could be altered by altering slurry viscosities. Atomized magnesium or other light metal is incorporated as the fuel. The illumination characteristics therefore approximate those of the familiar pressed magnesium-sodium nitrate system. Gel structure prevents separation of the suspended solids with flow under shear stress by force against a piston or diaphragm.

The experimental effort consisted of two parts: formulation and luminous efficiency measurement of liquid slurry compositions, and design and fabrication of an extrusion burner. For convenience in radiation measurement, liquid slurry samples were cast in cylindrical cases and functioned as end burning grains, the case wall inhibiting propagation of the flame front into the mass of composition. The effect of the case on the luminous efficiency was minimized by forming thin aluminum foil into cylindrical containers. This technique allowed a determination of the effect of carrier percentage and type, fuel percentage and particle size, and type of oxidizer on the slurry luminous efficiency.

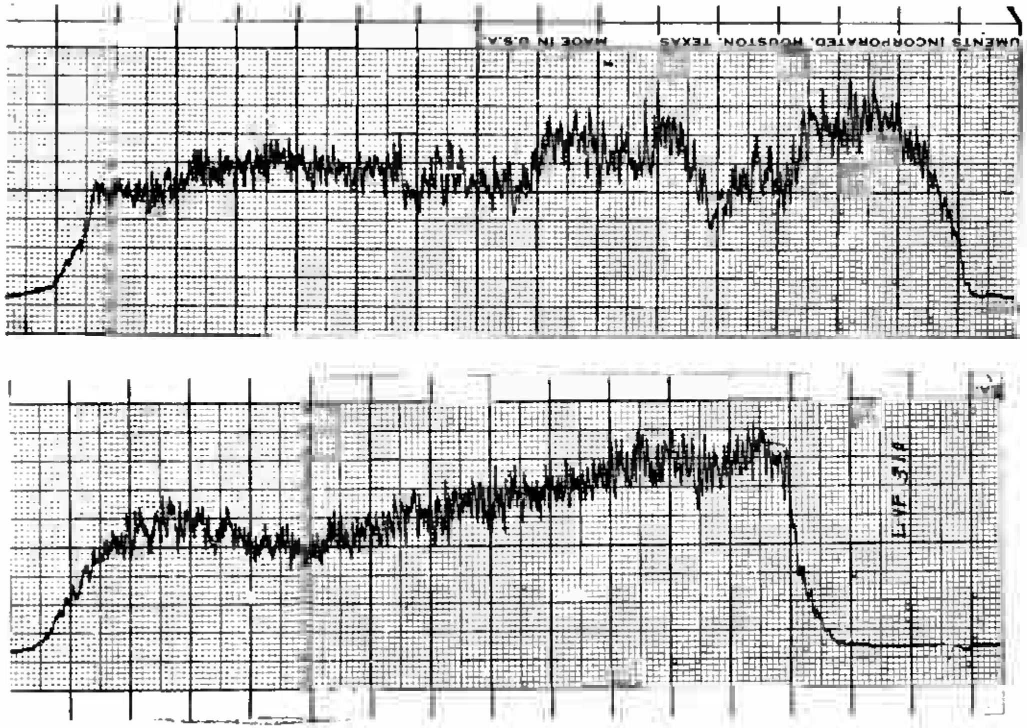
Carriers.- These liquid (carrier) compositions are all based on the use of polyfunctional alcohols or alcohol amines, short chain alcohols or esters. These liquids of dielectric constant intermediate between water and hydrocarbons are excellent solvents for many inorganic perchlorates, a class of oxidizers as useful in pyrotechnics as the nitrates. The use of a soluble oxidizer allows a larger value of the ratio of fuel and oxidizer to liquid carrier, a consideration of importance in the attainment of satisfactory luminous efficiency. Oxidation of this liquid carrier yields carbon monoxide, water and elemental hydrogen, the former two species exhibiting strong emission bands in the infrared which lower the flame temperature and decrease emission in the visible. The polyfunctional solvents burn cleanly to yield combustion products containing little obscuring smoke from unburned carbon.

A number of compositions incorporating polyfunctional alcohol carriers were prepared as follows: ethylene glycol, propylene glycol, polyethylene glycol 200, thiodiethylene glycol, diethylene glycol, butenediol, butynediol, and sodium xylene sulfonate in ethylene glycol. The luminous efficiencies fell in the range of 35,000 to 40,000 candle-seconds per gram when burned in an open cup; differences between compositions varied widely in both slurry viscosity and rheological behavior. Of these only two, ethylene glycol and propylene glycol, demonstrated characteristics that warranted further consideration. The remaining polyfunctional alcohols, because of poor solvent power for sodium perchlorate, produced slurries of undesirable rheological properties, the most important one of which is dilatency where the carrier (liquid) tended to separate from the solids under pressure. Ethylene glycol and propylene glycol on the other hand produced viscous solutions when sodium perchlorate was dissolved in either to significantly reduce dilatent behavior caused by insufficient carrier concentration and particle wetting.

The solubility of sodium perchlorate in propylene glycol was in the same order as in ethylene glycol, i.e., not much difference in in viscosity or gell behavior between ethylene glycol and propylene glycol slurries was noted. Sodium nitrate, on the other hand, did not dissolve in either carrier solution. Several compositions containing 15 to 20 percent propylene glycol were blended and tested (see Appendix I).

Higher carrier percentages, 17 to 20 percent propylene glycol, yielded more consistent results than those slurries containing lower carrier percentages, 15 to 16 percent (LVF-31 and LVF-32). This is probably due to difficulty to producing a homogeneous slurry composition of high viscosity and solids loading. As noted in the traces (figures 2 and 3), compositions of low carrier percentage produced irregular burn characteristics and wide variation in luminous efficiency.

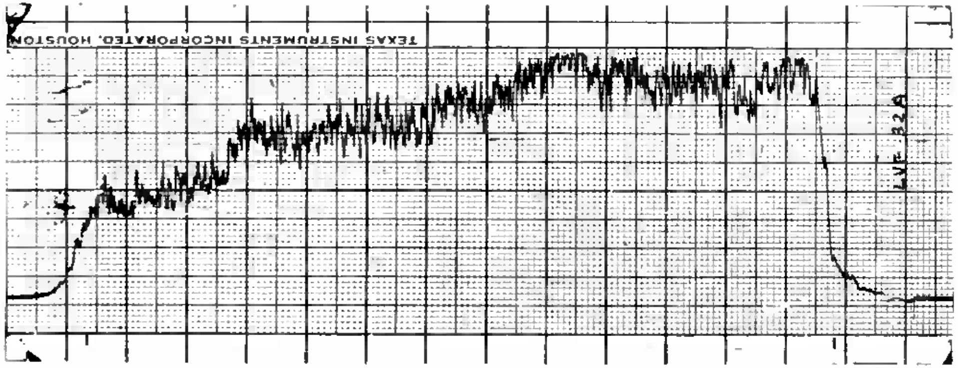
Amine carriers were also investigated for compatibility with sodium perchlorate/magnesium systems. Greatest solvation was noted for monoethanolamine (MEA) and to a lesser extent with diethanolamine (DEA) and triethanolamine (TEA). Dimethylethanolamine compared to diethanolamine in its solvating power for sodium perchlorate. $NaClO_4$ dissolves to a lesser degree in monoethanolamine than in ethylene glycol to produce a more viscous slurry at constant carrier percentage.



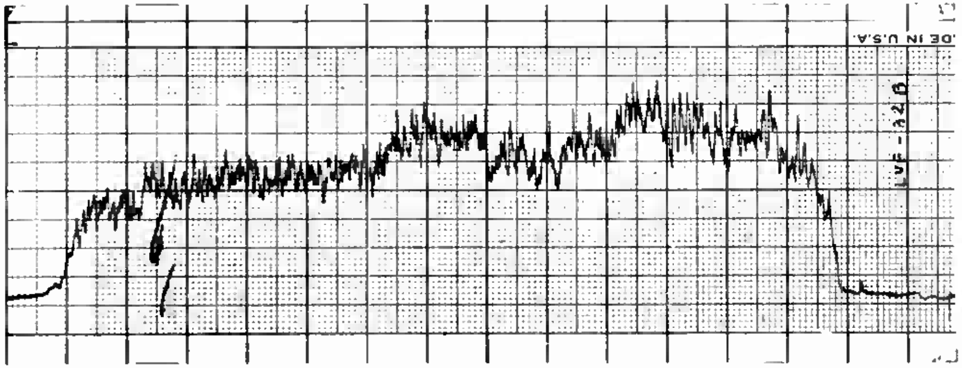
S/N: LVF-31B
January 17, 1967
EG&G Detector Head:
350 to 650 m
Texas Instruments
Oscillo/Riter
Recorder Range: 0.5 V/cm
Weight: 100.0 grams
Distance: 31.6 Feet
Burn Time: 30.8 seconds
Luminous Efficiency:
30,800 can-sec/gram

S/N: LVF-31A
January 17, 1967
EG&G Detector Head:
350 to 650 m
Texas Instruments
OscilloRiter
Recorder Range: 0.5 V/cm
Weight: 100.0 grams
Distance: 31.6 feet
Burn Time: 27.2 seconds
Luminous Efficiency:
27,200 can-sec/gram

Figure 2. Data, LVF-31A and LVF-31B



S/N: LVF-32A
January 17, 1967
EG&G Detector Head: 350-650 m
Texas Instruments Oscillo/Riter
Recorder Range: 0.5 V/cm
Weight: 100.0 grams
Distance: 31.6 feet
Burn Time: 26.0 seconds
Luminous Efficiency: 39,600
can-sec/gram



S/N: LVF-32B
January 17, 1967
EG&G Detector Head: 350-650 m
Texas Instruments Oscillo/Riter
Recorder Range: 0.5 V/cm
Weight: 100.0 grams
Distance: 31.6 feet
Burn Time: 26.0 seconds
Luminous Efficiency: 28,600
can-sec/gram

Figure 3. Data, LVF-32A and LVF-32B

The most suitable carrier found from both a rheological and luminous efficiency standpoint was ethylene glycol which is readily available commercially. Storage stability of the glycol is superior to that of the amine substituted alcohol; an ammoniacal odor develops on standing for 24 hours.

. Fuels and Oxidizers.- The determination of the trade-off between slurry rheological and combustion performance is a complicated and difficult procedure. To reduce the number of variables to be considered during initial carrier and fuel-oxidizer screening a one-to-one oxidizer to fuel weight ratio (O/F) was used. The general practice for battlefield illumination applications is to use from two to four times excess fuel over stoichiometric, depending on afterburning in the air for maximum luminous efficiency. With magnesium as the fuel and sodium perchlorate or sodium nitrate as oxidizer in a one-to-one O/F ratio, the excess fuel is only 25 and 16 percent respectively. Even though the nitrate has more available oxygen per unit weight, the difference between luminous efficiencies was very slight (30,250 candle-seconds/gram for sodium perchlorate versus 29,000 candle-seconds/gram for sodium nitrate) both for 20 percent propylene glycol carrier concentration. The slightly greater perchlorate luminous efficiency is probably due to the higher flame temperature which opens up another area in fuel-oxidizer selection.

In the combustion of magnesium and aluminum fuels in visual flare compositions these metals contribute to the flare luminance through the formation of highly exothermic refractory oxides (heats of formation being -3.57 and -3.92 kcal/gram, respectively) which add to the over-all heat of reaction and flame temperature or latent heat, and a background grey-body continuum radiation. The disadvantage of these fuels is the absorption and scattering of radiation by cooled oxide particles at the periphery of the flare plume; absorption and reradiation at a lower temperature does not add to the luminous emittance. The persistent line radiation from the sodium doublet at 5896 Å is the most efficient source of flare plume radiation in the visible so that magnesium-sodium nitrate compositions with a high color purity show maximum values of luminous efficiency. The use of an endothermic sodium compound as a fuel will increase the color purity of the flare combustion products by an increase in sodium concentration and concomitant decrease in magnesium oxide particle absorption and scattering.

With the inherent flexibility of the mechanically pumped liquid slurry system and in view of standard commercial practice in supplying highly reactive materials in the form of wetted particle slurries it was logical to investigate the feasibility of more reactive fuels and oxidizers.

Candidate fuel compounds include sodium hydride and sodium borohydride with heats of formation of -0.57 and -1.20 kcal/gram respectively. These active reducing agents, the former available as a 55 percent dispersion in mineral oil to inhibit spontaneous oxidation in air, must be handled with a consideration of the possibility of hypergolic ignition in contact with oxidizing agents. The experiments with these materials were therefore conducted on a milligram scale. Trituration of 200 mg of a 55 percent dispersion of sodium hydride in mineral oil with 200 mg of powdered sodium perchlorate gave a hypergolic ignition after a delay of approximately 5 seconds with a rapid transition to explosive deflagration that shattered the mixing container. Ignition was probably initiated by attrition of the mineral oil coating on the sodium hydride particles and direct contact with sodium perchlorate. This effort was discontinued; however, the use of these reactants in a high energy bipropellant liquid system seems promising with no requirement for a separate ignition system.

The addition of powdered sodium borohydride to glycols results in a slight exotherm and the formation of a cakey solid that was not sufficiently wetted for facile mixing with sodium perchlorate, which made the material unsuitable for monopropellant liquid flare systems. Mixtures of the dry powders were stable and a 20 gram sample of a 50/50 mixture was loosely compacted in a plastic sleeve for the observation of combustion stability. The mixture ignited and burned smoothly with less evolution of smoke than magnesium-fuel based compositions.

The evaluation of fuels and oxidizers was then continued with more familiar and less energetic pyrotechnic ingredients. The solubility of the strontium and barium perchlorates in ethylene glycol and monoethanolamine was investigated (the mixed salts in the ratio 1:5 are utilized as oxidizers for illumination compositions). The solubilities were determined to be less than the literature values and the difficulty of handling these more hygroscopic oxidizers eliminated them from further consideration.

A slurry system containing a mixed oxidizer (magnesium perchlorate and sodium perchlorate) was investigated (LVF-33 through LVF-36). The reactivity of magnesium perchlorate with glycol solutions solidified the slurry to a hard cake. Utilizing magnesium perchlorate in slurries was found not feasible; however, one composition (LVF-35) which resembled a cast flare, was tested. The results indicate that magnesium perchlorate as an oxidizer tended to reduce luminous efficiency, as was expected, since the amount of sodium (present in sodium perchlorate) which is the source of increased line radiation was reduced.

Because of rheological problems with the original magnesium-sodium perchlorate composition due to solution and recrystallization of the perchlorate on temperature cycling a mixed oxidizer (30 percent sodium perchlorate and 70 percent sodium nitrate) with magnesium as fuel was investigated. This composition with a one-to-one O/F ratio and 20 percent carrier yielded the highest luminous efficiency (34,900 candle-seconds/gram) of those found suitable for extrusion.

The luminous efficiencies of mixed particle sizes of the magnesium fuel (22 microns and 125 microns) were generally lower than those compositions prepared using only 22 micron magnesium particle size, LVF-22A-I and LVF-22J-O (see Table I-1, Appendix II). When equal parts of 22 micron and 125 micron particle size atomized magnesium were blended and tested, the compositions containing a mixture of larger magnesium particles showed a slightly lower luminous efficiency than those compositions not containing the larger particles; burn times for the compositions were nearly the same, approximately 16 to 17 seconds for a 100-gram sample.

Appendix I contains the leading particulars of the majority of the liquid visual flare compositions tested.

Rheological Factors.- To be suitable for use in a liquid flare concept a slurry should have the following rheological characteristics:

Yield stress substaintial enough for stability in storage but not large enough to interfere with extrusion.

Minimum apparent viscosity over the shear rate range required for the various applications.

Minimum change in viscosity with temperature.

Decrease in viscosity with increase in shear rate (pseudoplastic).

The criteria describe non-Newtonian fluid flow, generally that of a Bingham Plastic, which will hold its structure at a stress less than the yield stress. If this stress is exceeded the mechanical structure of the slurry breaks down into that of a Newtonian fluid or a thixotropic fluid, the latter characterized by further structure breakdown with the duration of the applied shear force. In either case the structure of the slurry at rest avoids separation and settling out of suspended solids, but the material may be pumped or extruded as a fluid by the application of pressure to the gelled fluid column. A simple analogy is the function of a tube of toothpaste.

From a combustion efficiency standpoint, zero percent carrier is desirable but is not compatible with good flow characteristics. On the other hand an excess of carrier not only greatly reduces luminous efficiency but also promotes migration of the particles within the mixture during either storage or extrusion . Too little carrier also promotes separation of suspended solids because the particles in the mixture tend to slip rather than flow. Slip occurs when there is a large velocity gradient between the particles, which ultimately results in the less viscous carrier extruding, forming a compacted cake of solids.

In general the higher the solids loading, the lower the shear rate at which slurries begin to show dilatent properties (viscosity increases with increasing shear rate). To minimize dilatancy effects it is desirable to use small particle sizes and as many soluble components as possible to reduce solids loading. Sodium perchlorate is soluble in ethylene glycol at 43 weight percent at 25°C; the saturation concentration varing with temperature. In mixtures near the saturation limit this would tend to reduce uniformity of the composition when subjected to the wide range of temperature extremes required for USAF world wide operations, thus giving less reproducible results. With an excess of sodium perchlorate crystals form and redissolve to alter both the physical and chemical properties of the slurry. This effect is eliminated by keeping the percent of sodium perchlorate below the solubility limit, the remaining oxidizer being sodium nitrate which is insoluble in ethylene glycol. This combined oxidizer system also appears to increase luminous efficiency slightly.

In some cases gellants have to be added to the compositions to impart the desired non-Newtonian behavior characteristics. These gellants include finely divided carbon and silica, heavy metal soaps and small percentages of in situ formed organic polymers. Cab-O-Sil and other proprietary silicas are useable gellants with ethylene glycol.

The most suitable compositions for the shear rate range used (500 to 1500 sec^{-1}) were those containing about 1 percent gellant, 20 to 22 percent ethylene glycol and the pyrotechnic composition being equal amounts of fuel (magnesium, 22 μ) and oxidizer (30 percent $NaClO_4$, 70 percent $NaNO_3$). The rheological measurements made during this program indicate that the apparent viscosities of the slurries suitable for extrusion ranged from 3,500 to 5,000 centipoise at a shear rate of approximately 1200 reciprocal seconds. (Flow rate of 26 grams/minute through a 0.052 inch diameter orifice). The lower viscosity represents a slurry with about 24 percent carrier and the higher one with about 20 percent carrier, each containing one percent gellant. At a flow rate of lo grams per minute thru the same orifice (500 sec^{-1} shear rate) the viscosity increased to 6000 centipoise for the 24 percent carrier and 18,000 centipoise for the 20 percent carrier composition. The significant effect of shear rate on viscosity is characteristic of pseudoplastic behavior.

Some mention should be made of slurry storage stability. Aging with an increase in viscosity with time has been observed with most of the slurries formulated to date. The major portion of the aging is completed within a few days. There are several possibilities as to the reason for aging. One is that the carrier is absorbed by the porous surface of the metal particles during storage (atomized magnesium is characterized by a high specific surface area) resulting in the formation of additional gel structure. Another possibility is that as the carrier is absorbed on the surface of the sodium nitrate particles it tends to break-up the agglomerates which had previously formed favorable particle size distribution for low slurry viscosity. When the agglomerates are broken-up by the wetting action, the particle distribution becomes less favorable to low viscosity and non-dilatent flow characteristics.

The aging effect was more pronounced for the compositions with higher percentage of carrier. Only slight aging effects were noticed with the 20 percent carrier composition, indicating that this amount of carrier was probably near the quantity required to completely wet the surfaces of the particulates; however, only limited analysis was made of the aging effect. Future work with these monopropellant slurries should investigate the effect of temperature cycling on aging and the relative magnitude of the change in viscosity and illumination efficiency resulting from aging.

Slurry Feed System.- Improvements were incorporated into the slurry feed system throughout the test program. The final configuration shown in Figure 4 was used for extrusion and burner tests. With the instrumentation in this system extrusion force and rate can be continuously monitored. In this configuration compressed air is used as the primary driving force, which acts on the water in the reservior and pushes it through the flow meter to the piston in the slurry cylinder, forcing the slurry out of the nozzle. Since water is essentially incompressible, slurry flow rate and water flow are identical if a rigid control line is used, thus enabling continuous monitoring of slurry extrusion rate even during combustion tests. Figure 5 shows the slurry storage cylinder and piston used during extrusion and combustion tests; during these tests the radial nozzle shown was replaced by a single orifice nozzle.

Burner Tests.- An experimental burner, 12 inches in length and 3 inches in diameter, was charged with 500 grams of slurry LVF-56B and test fired through a single burner nozzle of 0.094 inch diameter. The burner performed satisfactorily at a flow rate of less than 12 grams/minute for six minutes at which time the slurry flow stopped. The liquid carrier had partially exuded from the matrix of solids (dilatancy) in the nozzle orifice stopping flow; the composition burned through the nozzle into the cylinder and produced sufficient internal

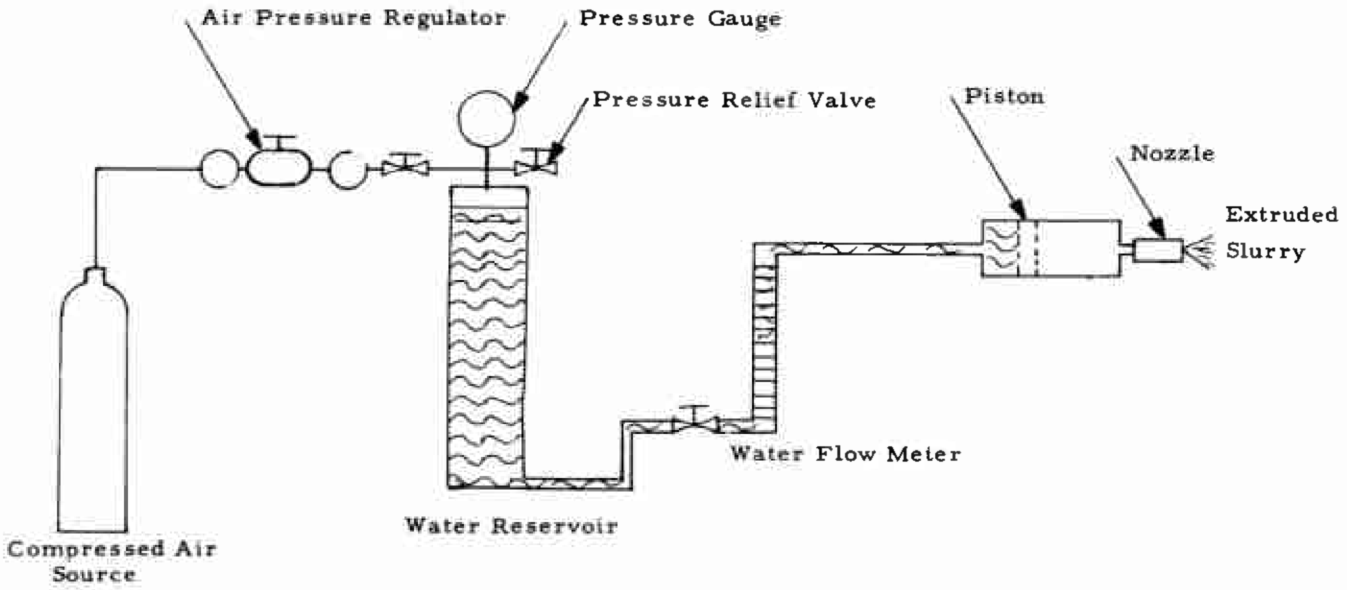


Figure 4. Slurry Feed System

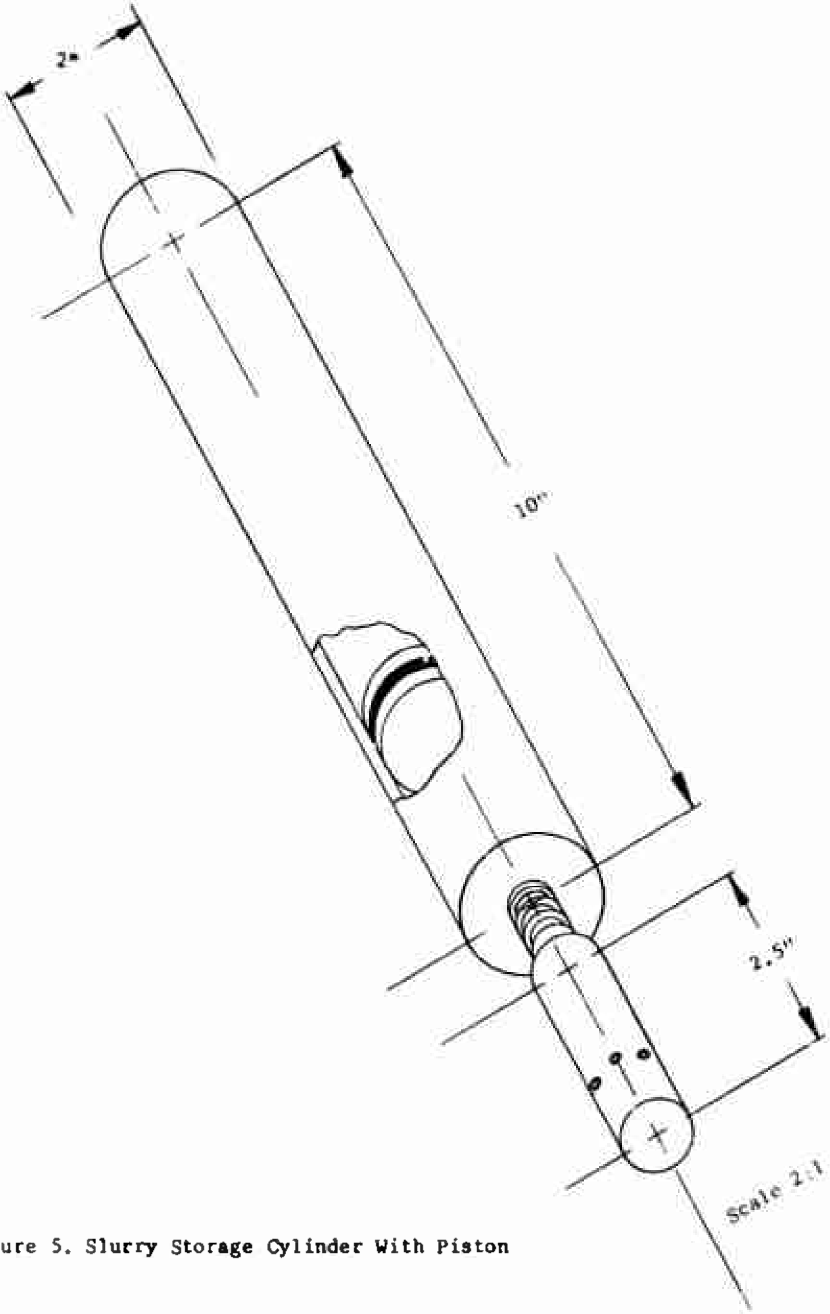


Figure 5. Slurry Storage Cylinder With Piston

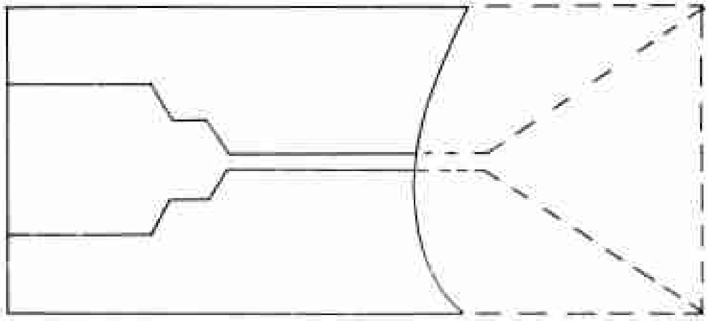
gas pressure to cause the nozzle end of the burner cylinder to rupture. Several burner tests with various nozzle configurations proved unsuccessful because of mechanical problems with the slurry feed system or difficulty in continuously maintaining a combustion zone because the slurry would extrude faster than the flame propagation rate. Once these problems were identified and corrected several burner tests were completed. A ten minute burn test was accomplished with a nozzle having 0.052 x 0.625 inch length orifice. The slurry holder cup burned off after about 5 minutes allowing a portion of the burning slurry to fall to the ground; however, combustion continued at the nozzle exit for another five minutes until slurry flow stopped due to nozzle entrance clogging (again dilatent slurry). The burning surface did not propagate into the 0.052 orifice. Investigation of the nozzle afterwards indicated that all combustion was on the exit side of the orifice; therefore it was felt that the extrusion rate could safely be reduced, thus reducing the possibility of slurry separation. The surface propagation rate of the slurry flame was measured at 6 inches per minute, much slower than the velocity through the orifice during the burn test (about 250 inches per minute).

In an effort to prevent the slurry holder section of the phenolic nozzles (rated at 160^{o}F continuous operating temperature) from burning away a stainless steel sleeve was inserted into the nozzle exit. The first two minutes of burning was stable until the stainless steel insert blocked due to a deposition of magnesium oxide (the high thermal conductivity of the metal promoting this condensation). The pressure of the combusting slurry forced the insert out and combustion continued sporadically until the 0.042 inch orifice exit was blocked with magnesium oxide (six minutes total burn time). A 0.0465 inch orifice nozzle with the stainless steel insert but a shorter slurry holder chamber burned for only two minutes before the orifice exit was blocked with magnesium oxide.

Another nozzle fabricated from Mykroy-750 (tradename for a glass-bonded mica rated at 750 degrees F continuous operation) burned for three minutes before the entire conical shaped slurry cup became blocked with magnesium oxide. This appears to be the result of rapid cooling of the combustion products around the periphery of the nozzle to form a crust of magnesium oxide. The crust continues to build up until the orifice finally becomes completely blocked.

Several other burn tests with phenolic nozzles of various sizes (0.052 to 0.059 inch diameter) resulted in four to six minute burn times; once the oxide crust formed was removed flow would continue without an increase in extrusion force. A larger 0.0625 inch diameter orifice resulted in a seven minute burn followed by propagation of the flame back through the nozzle and into the slurry storage area. The slurry burned very rapidly inside the cylinder, melted the end plate and the thrust developed by the escaping gases turned the burner test stand over.

A twenty minute combustion test was accomplished with the more viscous composition, LVF-86-B. The 280 grams of LVF-86-B began to extrude through the 0.052 inch orifice at 22 psig. No attempt was made to regulate the pressure which gradually increased to 28 psig where it remained throughout the test; the flow meter reading was erratic and ranging from 14 to 20 grams per minute indicated flow (actual average flow rate was 14 grams per minute). Even though all of the slurry was extruded from the cylinder the combustion did not propagate through the 0.052 inch orifice. Approximately one-quarter inch of the 0.90 inch long orifice did burn away during the twenty minutes of burning (see Figure 6). During this test the 0.750 inch long conical shaped slurry cup of the nozzle was pointed upward at a 45 degree angle.



NOTE: Dotted portion was burned away during 20 minute burn test.

Figure 6. Half Section of Typical Nozzle

SECTION III

CONCLUSIONS AND RECOMMENDATIONS

The use of the liquid slurry visual flare concept for application in tactical illumination devices is feasible. Items developed utilizing this concept will be less sensitive to ballistic impact than solid grain magnesium-sodium nitrate flares, a fact demonstrated when Mark 24 flare cases filled with LVF-75 slurry composition failed to ignite when hit by 7.62mm NATO tracer rounds from about 70 yards.

Because of the flexibility of the liquid visual flare concept. The consumption rate and the candlepower output are determined by a mechanical pumping rate, rather than grain dimensions; the tactical illumination applications to which it is suited cover the range from short burning (three to five minutes) high output (1 to 5 million candlepower) illumination devices (see Figure 1) to long burning (15 to 30 minutes) target marking rounds which produce light and/or smoke.

Ten pounds of LVF-87 (35,000 candle-seconds per gram) will produce 159 million candle-seconds of illumination. At a pumping rate of ten pounds per minute the output would be 2.65 million candle for one minute; at a pumping rate of one-third of a pound per minute the ten pounds of slurry would produce 85,000 candles for thirty minutes.

The above results indicate that the next logical step should be the initiation of development of a battlefield illumination device and a target marking round utilizing the liquid visual flare concept.

APPENDIX I

SLURRY FORMULATIONS

During this effort many formulations were investigated, the most important of which are listed in the following pages. The formulations are arranged in ascending order of percent carrier by carrier group. The carrier groupings include ethylene glycol, propylene glycol, other alcohols, propylene carbonate, monoethanol amine and nitroalkanes (Table I-1 through 1-6 respectively).

The serial numbers (LVF--) were assigned for identification purposes only and have no intrinsic meaning. Unless otherwise stated, the numbers below each serial number indicate the weight percent of the chemical listed on the left.

Table I-7 lists the chemicals used in these compositions and their sources. The chemicals marked with an asterisk are experimental compositions whose properties have not been completely determined; they were provided by the manufacturers for evaluation of their potential use in liquid flare slurries.

TABLE I-1. ETHYLENE GLYCOL COMPOSITIONS

	LVF-72	LVF-70	LVF-56	LVF-56A	LVF-568	LVF-75	LVF-76	LVF-74
Ethylene Glycol	13.5	13.0	13.0	15.0	16.0	18.0	18.0	17.0
Sodium Xylene Sulfonate		1.0	2.0	2.0	2.0			1.0
Sodium Perchlorate	43.25	43.0	42.5	41.5	41.0	41.0	41.0	41.0
Magnesium, 22 μ	43.25	43.0	42.5	41.5	41.0	41.0	41.0	41.0
Candle-seconds/gram			39,000 to 33,000				35,700	

[1] 4 percent, 22 μ
37 percent, 125μ

	LVF-63	LVF-55	LVF-1	LVF-87	LVF-79	LVF-80	LVF-85	LVF-88
Ethylene Glycol	17.5	9.25	18.7	19.8	13.3	10.0	20.8	22.0
Sodium Xylene Sulfonate		9.25						
Polyethylene Glycol 200	.5							
NMDP					6.7			
Tris Nitro						10.0		
Sodium Perchlorate	39.8	38.5	40.0	12.0	40.0	40.0		11.9
Sodium Nitrate				27.8			30.5	32.4
Magnesium, 22 μ	39.2	43.0	40.0	39.8	40.0	40.0	39.6	32.9
Cab-O-Sil	2.4			.6				
Carbon, Black			1.3					

TABLE I-1. (Concluded)

	LVF-63	LVF-55	LVF-1	LVF-87	LVF-79	LVF-80	LVF-85	LVF-88
Silica								.8
Candle-seconds/gram				34,490	Slow Burn Difficult to Ignite	Unstable Combustion/ Increase in Size After One Week		23,050

	LVF-82	LVF-81	LVF-86A	LVF-86B	LVF-5
Ethylene Glycol	9.0	7.15	23.6	23.6	31.3
NMPD	7.0	7.15			
Propylene Carbonate	5.0	7.15			
Sodium Perchlorate	39.0	42.8	11.8	11.8	$Ba(ClO_4)_2$ 37.5
Sodium Nitrate			35.5	35.5	$Sr(ClO_4)_2$ 9.4
Magnesium, 22 µ	40.0	35.8	28.4	28.4	21.8
Cab-O-Sil			.7		
Silica			.7	.7	Failed to Ignite
Candle-seconds/gram			18,000	20,300	

TABLE I-2. PROPYLENE GLYCOL COMPOSITIONS

	LVF-32	LVF-24	LVF-36	LVF-35	LVF-31	LVF-30	LVF-34	LVF-29
Propylene Glycol	15	15	15	16	16	17	18	18
Sodium Perchlorate	42.5		21.25	21.0	42	41.5	20.5	41
Sodium Nitrate		42.5						
Magnesium Perchlorate			21.25	21.0			20.5	
Magnesium, 22μ	42.5	42.5	42.5	42.0	42	41.5	41	41
Candle-seconds/gram	39,000 28,600		*	24,300 19,000	30,800 27,200	31,000 31,600	*	23,900 35,700

	LVF-28	LVF-33	LVF-27	LVF-37
Propylene Glycol	19	20	20	20
Sodium Perchlorate	40.5	20	40	
Sodium Nitrate				40
Magnesium Perchlorate		20		
Magnesium, 22μ	40.5	40	40	40
Candle-seconds/gram	23,900 32,200	*	30,000 30,300	27,800 29,400

*Unsatisfactory Physical Properties.

TABLE I-3. OTHER ALCOHOL COMPOSITIONS

LVF-67	
Thiodiethylene Glycol	23.4
Sodium Nitrate	36.8
Barium Nitrate	1.2
Magnesium, 22μ	28.6

LVF-53	
Diethylene Glycol	23.0
Sodium Perchlorate	15.4
Sodium Fluoride	30.8
Magnesium, 22μ	30.8

	LVF-68	LVF-69	LVF-66
Butenediol	21.7	23.0	29.5
Butynediol	2.4	3.4	
Barium Nitrate	1.2	1.8	1.6
Sodium Nitrate	48.2	40.2	65.6
Magnesium, 22μ	26.5	31.6	3.3

TABLE I-4. PROPYLENE CARBONATE COMPOSITIONS

	LVF-25	LVF-26	LVF-40	LVF-39	LVF-62
Propylene Carbonate	15	15	17	18	10
Ethylene Carbonate					8
Sodium Perchlorate	42.5		41.5	41.0	
Sodium Nitrate		42.5			41.0
Magnesium, 22μ	42.5	42.5	41.5	41.0	41
Candle-seconds/gram	30,000	31,600			

	LVF-38	LVF-78	LVF-54	LVF-41
Propylene Carbonate	20	12.5	23.0	25.5
Ethylene Carbonate		10.0		
Sodium Perchlorate	40	52.5	17.0	
Sodium Oxalate				37.2
Sodium Fluoride			20.0	
Magnesium, 22μ	40	25.0	40.0	37.2
Candle-seconds/gram	13,500		Unstable Combustion	29,600 31,500

TABLE 1-5. MONOETHANOL AMINE COMPOSITIONS

	LVF-7	LVF-42	LVF-43	LVF-15	LVF-44	LVF-9	LVF-45	LVF-73
Monoethanol Amine	11.1	12	13	13.3	14	14.7	15	13.0
Sodium Xylene Sulfonate								2.0
Sodium Perchlorate	71.1	44.0	43.5	53.5	43.0	46.3	42.5	42.5
Strontium Perchlorate	1.1							
Magnesium, 22μ	16.7	44	43.5	33.0	43.0	39.0	42.5	42.5
Candle-seconds/gram	13,600 16,200	23,100 30,000	30,900	16,800	21,000 23,000	41,400		

	LVF-13	LVF-46	LVF-47	LVF-48	LVF-49	LVF-12	LVF-50	LVF-4
Monoethanol Amine	15.8	16	17	18	19.0	19.8	20	20
Sodium Perchlorate	42.1	42.0	41.5	41	40.5	39.8	40	45.0
Cab-O-Sil						1.0		
Magnesium, 22μ	42.1	42.0	41.5	41	40.5	39.8	40	35.0
Candle-seconds/gram	32,800	21,000 23,100		25,400 23,100		33,300		

TABLE I-5. (concluded)

	LVF-51	LVF-52	LVF-17	LVF-16	LVF-11	LVF-10
Monoethanol Amine	21	22	21.7	24.7	26.6	32.3
Sodium Perchlorate	39.5	39.0	38.5	36.8	35.5	32.3
Cab-O-Sil			1.7	1.8	2.4	32
Magnesium, 22μ	39.5	39.0	38.5	36.8	35.5	32.3
Candle-seconds/gram			*	*	*	*

*Failed to Ignite

TABLE I-6. NITROALKANE COMPOSITIONS

	LVF-77	LVF-65	LVF-57
2-Nitro-1-Butanol	20	28.5	
Nitromethane			27.4
Sodium Perchlorate	40		36.3
Sodium Nitrate		28.5	
Magnesium, 22μ	40	43.0	36.3
Candle-seconds/gram	31,300 Crucible Eratic Hard Cake After One Week	29,000 Fast Burn Did Not Ignite Readily	too crumbly

TABLE I-7. MATERIALS

1. Barium Perchlorate - C. Frederick Smith Chemical Company, 867 McKinley Avenue, Columbus, Ohio.

2. Butenediol - General Aniline & Film Corporation, 140 West 51 Street, New York, New York 10020.

3. Butynediol - General Aniline & Film Corporation, 140 West 51 Street, New York, New York 10020.

4. Cab-O-Sil, M-5 - Cabot Corporation, 125 High Street, Boston, Mass.

5. Diethanolamine - Allied Chemical Corporation, Nitrogen Division, Orange, Texas.

6. Dimethylethanolamine - Jefferson Chemical Company, Post Office Box 53300, Houston, Texas 77052.

7. Ethylene Carbonate - Jefferson Chemical Company, Inc., Post Office Box 53300, Houston, Texas 77052.

8. Ethylene Glycol - Allied Chemical Corporation, Nitrogen Division, Orange, Texas.

9. Magnesium, Atomized, 22 ±B microns, MIL-U-14067, Type I - Valley Metallurgical Processing, Essex, Connecticut.

10. Magnesium, Atomized, 125 microns, MIL-P-14067, Type I - Valley Metallurgical Processing, Essex, Connecticut.

11. Magnesium Perchlorate - G. Frederick Chemical Company, B67 McKinley Avenue, Columbus, Ohio 43223.

12. Monoethanolamine - Allied Chemical Corporation, Nitrogen Division, Orange, Texas.

13. 2-Nitro-1-butanol - Commercial Solvents Corporation, 260 Madison Avenue, New York, New York 10016.

14. Nitromethane - Commercial Solvents Corporation, 260 Madison Avenue, New York, New York, 10016.

15. NMPD (2-Nitro-2-Methyl-1,3-propanediol) - Commercial Solvents Corporation, 260 Madison Avenue, New York, New York 10016.

16. Polyethylene Glycol 200 - Allied Chemical Corporation, Nitrogen Division, Orange, Texas.

TABLE I-7. (concluded)

17. Propylene Carbonate - Jefferson Chemical Company, Post Office Box 53300, Houston, Texas 77052.

18. Propylene Glycol - Allied Chemical Corporation, Nitrogen Division, Orange, Texas.

19. Silica, Quso G 32 - Philadelphia Quartz Company, Foot of Lamokin Street, Chester, Pennsylvania 19016.

20. Sodium Fluoride - Allied Chemical Corporation, Industrial Chemicals Division, 40 Rector Street, New York, New York 10006.

21. Sodium Nitrate - Allied Chemical Corporation, Industrial Chemicals Division, 40 Rector Street, New York, New York 10006.

22. Sodium Perchlorate - G. Frederick Smith Chemical Company, 867 McKinley Avenue, Columbus, Ohio 43223 and American Potash & Chemical Corporation, 99 Park Avenue, New York, New York 10016.

23. Strontium Perchlorate - G. Frederick Smith Chemical Company, 867 McKinley Avenue, Columbus, Ohio 43223.

24. Sodium Xylene Sulfonate, "SXS" - Retzloff Chemical Company, Post Office Box 45296, Houston, Texas 77045.

25. Triethanolamine - Allied Chemical Corporation, Nitrogen Division, Orange, Texas.

26. "Tris Nitro" (Tris(hydroxymethyl) nitromethane) - Commercial Solvents Corporation, 260 Madison Avenue, New York, New York 10016.

27. Ultra Blend 100 - Witco Chemical Company, Ultra Division, 2 Wood Street, Paterson, New Jersey 07509.

APPENDIX II

LUMINOUS EFFICIENCY MEASUREMENTS

During the course of the program several methods were used to determine luminous efficiency. Since the in ultimate application the slurry will be combusted without a case, it is desirable to reduce extraneous effects.

Case Effect.- During initial tests samples of slurries were poured into paper flare cases approximately three inches long by 1.5 inch I.D. The cases were lined with a polyester material to prevent saturation of the paper with the liquid carrier, thus preventing a reduction of carrier percentage in the slurry. The paper cases burned back with the slurry, maintaining an effective visible plume, simulating a burner effect which is the ultimate application of the liquid slurry.

A wide variation in luminous efficiencies were noted for similar slurry compositions which were prepared and tested in a simple end-burning configuration. In order to determine a test condition for more consistent data, slurries were test fired both in paper cases and 50 ml capacity polyethylene beakers. The samples tested in paper cases showed a wide variation of luminous efficiencies from 18,200 to 38,000 candle-seconds/gram (see Table II-1). More favorable results were obtained from slurries tested in polyethylene beakers, from 29,400 to 32,300 candle-seconds/gram. Also, the figures obtained from those units prepared in polyethylene beakers tended to be higher than those tested in paper cases. It was then concluded that although the paper cases burned away as the slurry burned, some case effect invariably entered into the data probably due to residual case material.

In an effort to reduce the variation in luminous efficiencies small crucibles were tried but the percentage variations were worse. Figure 7 shows the results of three consecutive tests of the same slurry in crucibles.

With the same slurry in a cup made from aluminum foil the results of three tests were reproducible (see Figure II2). The higher luminous efficiencies are expected since the detector head can view the flame directly as the foil burns away; the heat loss to the case wall is also sharply reduced. The combustion of the aluminum foil probably adds a small amount, but this effect should be negligible because of the relative mass of the cup and slurry sample (the cup weighing less than one-half gram holds about 20 grams of slurry). The aluminum foil cup results in more consistant data, which is important when comparing relative performance of compositions. Delivered luminous efficiency will vary with burner configuration, plume size, and plume shape.

Instrumentation and Data Reduction.- The visible radiation was detected by an Edgerton, Germeshausen & Grier Radiometer Model 580. Data on flare intensities obtained for the first fifteen tests were recorded on a non-integrated continuous source range. This, however, posed problems in obtaining an average intensity reading due to fluctuations in intensity during flare combustion. Subsequent tests were run recording data on an integrated scale which yield a total luminous energy for the duration of burn time. The readings so obtained included the time factor, in seconds, which greatly facilitated data reduction. A Weston Model 614 Illumination Meter aided in obtaining additional intensity measurements for purposes of comparison and correlation with the results obtained from the EG&G Radiometer system. A check of calibration of the Weston Meter with a secondary light standard revealed a 2½ percent difference. The EG&G Radiometer, however, showed a 10 percent discrepancy from the secondary standard and was recalibrated.

The difference in intensities reported for earlier tests and subsequent tests (from LVF-16 on) is to be attirbuted to the use of different data recording methods and recalibration of the detection system. Later tests were conducted simultaneously using the EG&G radiometer and the Weston illumination meter. The differences between readings obtained from both can be attributed to the fact that intensity fluctuated and readings on the Weston meter were difficult to average, a condition similar to that which was experienced when the EG&G radiometer was used in the non-integrated position.

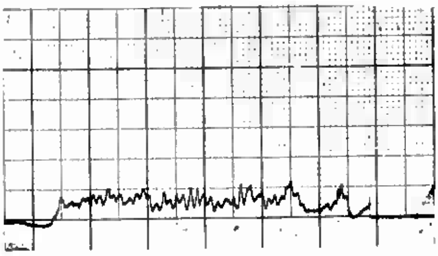
The use of a recorder (Texas Instruments Oscillo/riter) with both instruments greatly facilitated testing and data reduction. The traces obtained showed burn characteristics of slurry compositions which could be visually inspected for uniformity of burn rate. Data reduction (calculation of luminous efficiencies) was accomplished by calibrating the recorder to receive output signals both from the EG&G Radiometer head and from the Weston Illumination Meter. The area under the curve represents total output (candle-seconds). Slurries beginning from composition LVF-23 were tested using the recorder with the Weston or EG&G thus providing a graph of the illumination test.

TABLE II-1. LUMINOUS EFFICIENCY VERSUS SAMPLE HOLDER

SERIAL NUMBER	WEIGHT	BURN TIME SECONDS	E CAN-SEC/G
LVF-22A	100.0 g	15.7	28,000
LVF-22B	100.0 g	16.8	18,200
LVF-22C	100.0 g	16.5	26,700
LVF-22D	100.0 g	17.6	20,200
LVF-22E	100.0 g	17.6	21,000
		Average 16.9	Average 22,800
LVF-22F	70.0 g	15.4	29,400
LVF-22G	70.0 g	15.3	30,500
LVF-22H	70.0 g	14.0	32,200
LVF-22I	70.0 g	13.8	31,300
		*Average 14.4	Average 30,850
LVF-22J	100.0 g	16.7	22,800
LVF-22K	100.0 g	16.1	21,700
LVF-22L	100.0 g	15.9	24,400
LVF-22M	100.0 g	17.3	20,200
LVF-22N	100.0 g	15.2	19,400
LVF-22O	100.0 g	16.1	17,100
		Average 16.2	Average 20,940

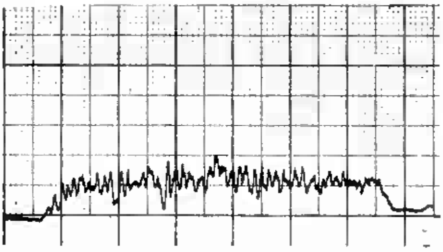
	LVF-22 A-I	LVF-22 J-O
Monoethanolamine	15.0 %	14.0 %
Sodium Perchlorate	42.5	43.0
Magnesium - 22 microns	42.5	21.25
Magnesium - 125 microns		21.25

*Note: 70 grams versus 100 grams for paper cases.



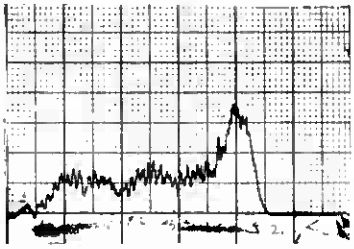
S/N: LVF-86B 20 Sept 1967

EG&G Detector Head: 350 to 650 mμ
Clay Crucible at 15 feet
Weight: 8.7 grams
Burn Time: 10.1 Seconds
Luminous Efficiency: 6,420 candle-seconds/gram



S/N: LVF-86B 20 Sept 1967

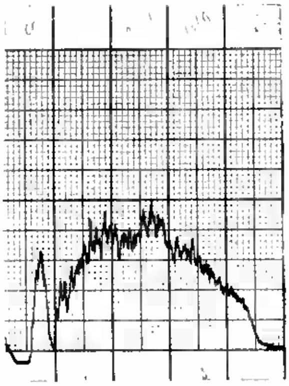
EG&G Detector Head: 350 to 650 mμ
Clay Crucible at 15 feet
Weight: 9.1 grams
Burn Time: 13.3 seconds
Luminous Efficiency: 15,000 candle-seconds/gram



S/N: LVF-86B 20 Sept 1967

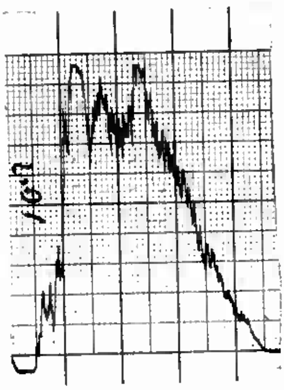
EG&G Detector Head: 350 to 650 mμ
Clay Crucible at 15 feet
Weight: 9.0 gram crucible
Burn Time: 8 seconds
Luminous Efficiency: 12,200 candle-seconds/gram

Figure II-1. Crucible Results



S/N: LVF-86B 25 September 1967

EG&G Detector Head: 350 to 650 mμ
Aluminum Cup: 15 Feet
Weight: 21.0 grams
Burn Time: 8.0 seconds
Luminous Efficiency: 20,250
candle-seconds/gram



S/N: LVF-86B 25 September 1967

EG&G Detector Head: 350 to 650 mμ
Aluminum Cup: 15 feet
Weight: 22.4 grams
Burn Time: B seconds
Luminous Efficiency: 20,640
candle-seconds/gram

Figure II-2. Aluminum Cup Results

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13. ABSTRACT

An exploratory development program was accomplished to analytically and experimentally determine feasibility of developing a liquid visual flare for tactical battlefield illumination. The effort described in this report included literature search, study, analysis, and experiments with various liquid flare compositions, various burner configurations, and extrusion techniques. The liquid visual flare concept was found feasible for both illumination and for target marking since the flexibility afforded by the mechanical flow rate provides a choice of a high powered flare with several million candlepower for 2 to 5 minutes (intensity) or a long burning target marking device up to 30 minutes duration. A monopropellant liquid slurry system was selected for engineering simplicity. The liquid slurry uses atomized magnesium or other metal as a fuel and uses an oxidizer based on the partial solubility of inorganic perchlorates in polyfuntional alcohols and alcohol amines. The liquid flare concept provides approximately the same output power in candle-seconds per gram of composition as solid grain magnesium-sodium nitrate flares and also possesses two distinct advantages over solid flares in that it has been proven relatively insensitive to ballistic impact from small arms fire and its time duration and intensity can be controlled over a period of 2 minutes to 30 minutes by the mechanical flow rate. The next logical step should be the development of a battlefield illumination device and a target marking device utilizing the liquid visual flare concept.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Liquid Visual Flare						
Slurry						
Monopropellant						
Fuel/Oxidizer						
Illumination						
Radiation						
Flare						