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Richard G. Gann
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ADVANCED PROPELLANT/ADDITIVE DEVELOPMENT FOR FIRE SUPPRESSING GAS GENERATORS: DEVELOPMENT + TEST

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The "Advanced Propellant/Additive Development for Gas Generator" project is a collaborative effort between General Dynamics Ordnance and Tactical Systems (GD-OTS) and the Naval Air Warfare Center-Weapons Division (NAWCWD). The objectives of this NGP project are to develop new highly efficient, environmentally acceptable, chemically active fire suppressant capabilities based upon solid propellant gas generators; and improve understanding of propellant and additive effectiveness in fire suppression.

New propellant formulations, based upon novel high-energy, high-nitrogen fuels, were refined in order to reduce overall combustion temperatures while maintaining ballistic robustness. These fuels included 5-aminotetrazole and the new high nitrogen compound BTATZ ($C_4H_4N_{14}$). The fuels were formulated with oxidizers and chemical coolants to reduce combustion temperatures, and modified with chemical additives to enhance fire suppression effectiveness. Cooler exhausts were also generated using Hybrid Fire Extinguisher systems, which contain combinations of solid propellant gas generators and fire suppressing fluids.

Suppression effectiveness of several propellant and hybrid configurations was tested in the GD fire test fixture against a controlled JP-8 fire. These tests compared HFE to SPGG performance, evaluated the effect of discharge temperature and the presence of chemically active additives.

This presentation will summarize results to date of propellant formulations, hybrid configurations and fire suppression effectiveness testing, and attempt to correlate effectiveness with additives and exhaust temperature.

Introduction

The utility of solid propellant gas generator (SPGG) technology for fire suppression has been amply demonstrated on a variety of platforms, from subscale[1]-, to midscale[2]-, to full-scale[3] and production platforms. Many of the benefits of SPGG-based fire protection systems, such as their ability to deliver large quantities of suppression agents in a rapid, mass- and volume-efficient manner, are a direct result of the solid propellant combustion process.

Conceptually, both SPGGs and pressure-bottle (blowdown) systems deliver agent according to the same analytical expression, whereby the rate of agent delivery $\dot{m}_d$ is related to the discharge coefficient C_d of the agent, the throat area A_t and the pressure inside the delivery device P_c, i.e.

$$\dot{m}_d = C_d * A_t * P_c . \tag{1}$$

In a blowdown system, the initial bottle pressure is given by the sum of agent vapor pressure P_{vap} plus the pressure of a pressurant gas (typically nitrogen) $P_{pressurant}$:

$$P_c = P_{vap}(agent) + P_{pressurant} . \tag{2}$$

Here, P_c is at its maximum in the pre-discharge condition, and both P_c and $\dot{m}_d$ decrease rapidly upon initiation of the discharge process. Where the SPGG differs from the blowdown bottle is the ability to store agent at zero internal pressure, and then to generate high pressures internally by the combustion of the solid propellant to form a blend of inert gases.

For solid propellants, P_c is directly related to the product of the burning surface area $A_{surface}$ and the rate at which gas is generated from the propellant, typically referred to as the "burn rate," (BR, units in/s or cm/s)[4]. The BR itself is a function of the system pressure, with log(BR) typically increasing linearly with log(pressure) with slope referred to as the pressure exponent n. The expression relating P_c and BR is given below:

$$P_c \propto A_{surface} * BR^{1/n} . \tag{3}$$

According to expression (1) above, this high ballistically-induced internal pressure can translate into sustained high rates of agent discharge.

Inherent to the SPGG process is the fact that the gases generated by propellant combustion are hot. Reducing the temperature of the exhaust gases would increase their fire suppression effectiveness, since they would then be capable of absorbing more heat from the fire. Additionally, cooler exhaust gases would have less impact on the structures and materials with which they contact during the agent discharge process.

Maintaining high discharge rates for different propellants, then, requires maintaining a sufficiently high P_c. According to the above discussion, then, for the desired cooled propellant compositions to meet desired rapid discharge requirements, some minimum P_c (and/or BR) must be maintained.

This NGP project has developed several means for reducing the exhaust temperatures of SPGG devices. Both approaches mix the hot combustion gases with materials that can absorb heat and cool the actual exhaust. In one case the coolant is incorporated directly into the solid propellant composition, and in the other the propellant gases are mixed with a fluid characterized by a high

heat capacity. This latter fluid is referred to as a hybrid fluid and the device is referred to as a hybrid fire extinguisher (HFE).

In addition to cooler propellant approaches, the GD-NAWC effort has explored the effect of different approaches to incorporating chemical activity into SPGG and HFE devices. Earlier work by GD/NAWC[2] has demonstrated the improvements possible by incorporating chemically active precursors into the SPGG event. Work on the subject of Advanced Propellant/Additive Development for Fire Suppressing Gas Generators is now in its third phase. Phase I and initial Phase II results are presented in previous works[2,5]. This present work describes the results from the latter part of Phase II and the initial part of Phase III. Phase II and Phase III focus on the benefits achievable by incorporating the chemically active additive directly into the propellant and/or hybrid fluid.

Experimental Techniques

Propellant Development

The developmental propellants described here were made up in relatively small batches (50g – 1 kg). In a typical process, individual ingredients were pulverized and then wet-mixed in an inert fluorocarbon medium, where polymeric binder was deposited upon the powder and the mixture precipitated. The solid was then collected, dried and compression molded into pellets for burn rate analysis. Larger batches of propellant were manufactured and pressed on an automated rotary press.

Test Fixture Description

The mid-scale Fire Test Fixture (FTF), developed by General Dynamics, was used to test the effectiveness of various agents (Figure 1). The agents were delivered to the fire using the following technology:

- Solid Propellant Gas Generator (SPGG)
- Hybrid Fire Extinguisher (HFE)

This same test fixture was used during previous test phases[2,5] and its purpose is to provide a platform where fire suppressants can be evaluated under repeatable test conditions. The mass flow rate of air through the fixture was set at 450 g/s (1 lb_m/s). JP-8 fuel was used with an air-fuel ratio of 31 $\dot{m}_{air}/\dot{m}_{fuel}$ and an equivalence ratio of 0.5. This created a fire with a flame temperature of ~1000 K and a ~700 kW intensity. The residence time through the fire zone of the fixture was calculated to be 1.2 seconds and the discharge time of the SPGG and HFE units was ~100-200 milliseconds. Additional details about the FTF, including a facility schematic and a list of the major subsystems, have been published previously[2].

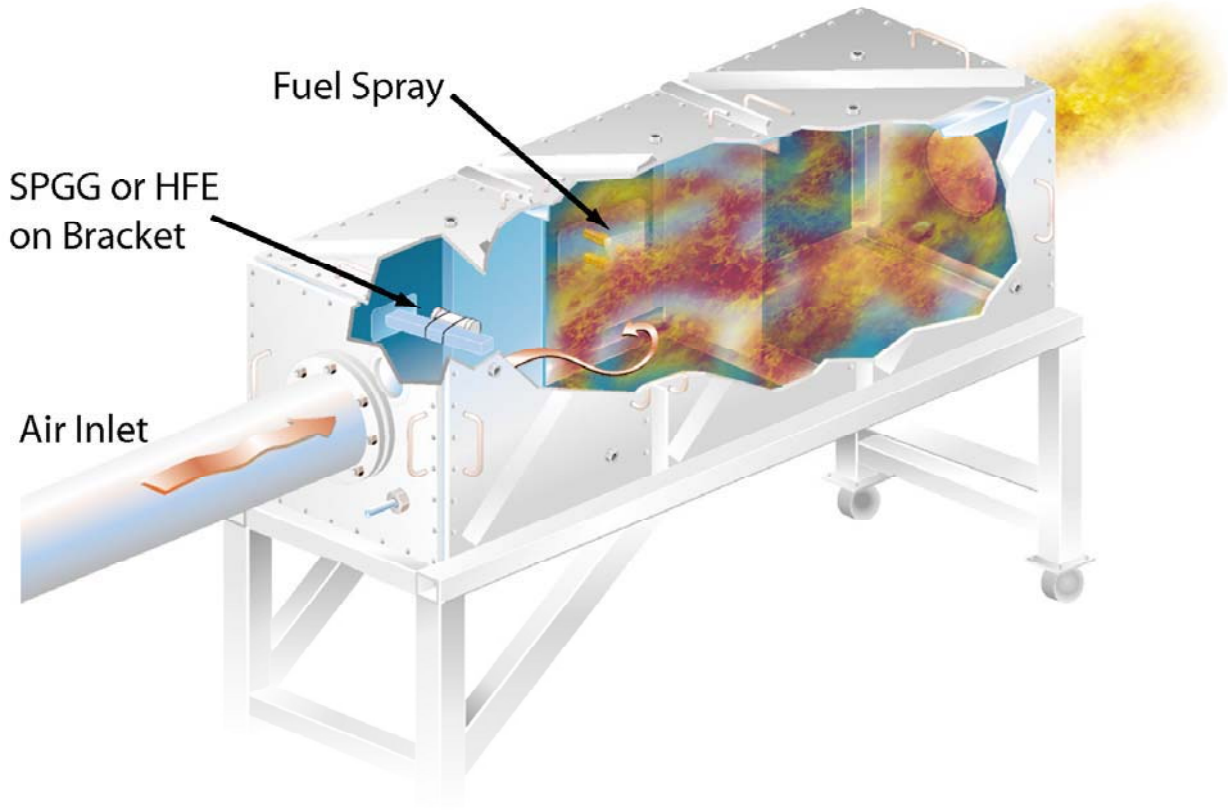


Figure 1: GD Fire Test Fixture (FTF)

Results

Propellant Development: Cooler Formulations

Cooler solid propellant compositions were examined by direct incorporation of coolant into the propellant blend. In examining a number of solid propellant compositions, we have found that the benefits of decreased exhaust temperatures are often offset by a decrease in the burn rate of the propellant, which in turn relates to a decrease in the rate of suppressant delivery. This is illustrated in Figure 2, where ln(burn rate) is plotted as a function of 1/T. The linear relationship indicates that modeling this relationship as an Arrhenius-type activated process is appropriate.

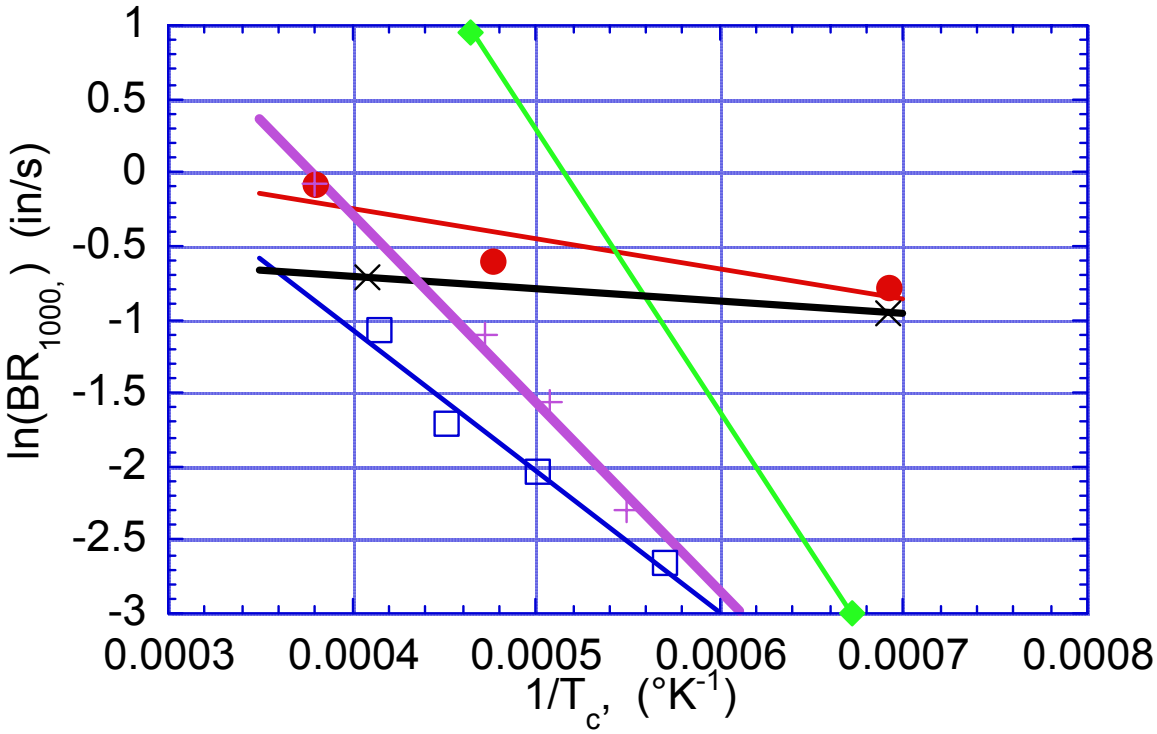
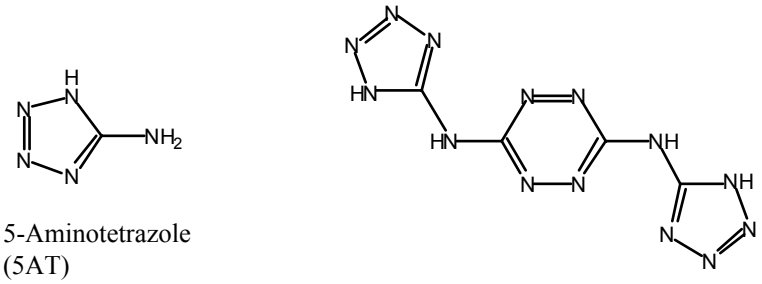


Figure 2: Plot of ln(burn rate) vs. 1/T

The GD-NAWC NGP program has utilized the model above for maintaining adequate agent delivery rates while reducing propellant exhaust temperatures. We have created some novel formulations using compositions that burn at sufficiently rapid rates so they can be modified by incorporation of chemical coolants to reduce exhaust temperatures, while maintaining agent delivery rates at levels sufficient for rapid flame extinction. The effect of coolant level upon calculated adiabatic agent temperature for one family of compositions is shown in Figure 3.

Figure 3: Plot of Adiabatic Agent Temperature and Burn Rate vs. Coolant Level

Propellant formulations incorporating the new high nitrogen compound BTATZ ($C_4H_4N_{14}$), provide increased means for reducing propellant combustion temperatures. BTATZ is structurally similar to 5-aminotetrazole (5AT), the fuel used in GD's FS01-40 propellant (Figure 4). The preparation of BTATZ has progressed to the 1 lb scale, with purity ~ 97–99%.



5-Aminotetrazole
(5AT)

Bis(aminotetrazolyl)tetrazine
(BTATZ)

Figure 4: High-nitrogen fuels used in GD-NAWC propellant development

Scale-up work on the 3 gal. scale has proven successful. The recent acquisition of a 50 gal. reactor will allow us to scale up to an extent that the synthesis of intermediates need only be carried out periodically. Further safety data on BTATZ itself has been obtained. BTATZ shows acceptable friction and impact sensitivity but is somewhat sensitive to electrostatic initiation. When formulated into a molding powder with poly(ethylacrylate), electrostatic sensitivity is still

a concern, even when 0.5% carbon black is added, however when pressed into pellets or deposited as a thin layer the material meets the criteria set for routine handling of energetics.

A number of formulations have been evaluated for their suitability in agent generation devices; several formulations are based on BTATZ, although a number of others involve other energetic fuels such as 5-aminotetrazole (5AT). The results are presented in Table 1 below. These data again illustrate the falloff in burn (agent generation) rate with decreased adiabatic combustion temperature.

Table 1. Propellant Physical Properties

Descriptors	Major Constituents[(a)]	T_c, K	Gas, mol/100g	Theoretical Density, g/cc	BR_{1000}, in/s
BTATZ KP	BTATZ-5AT KP	1961	4.12	1.72	1.31
BTSN-00	BTATZ $Sr(NO_3)_2$	2774	2.27	2.38	1.09
BTSN-10	BTATZ $Sr(NO_3)_2$	2562	2.12	2.43	TBD
BTSN-20	BTATZ $Sr(NO_3)_2$	2258	2.00	2.49	0.75[(b)]
BTSN-30	BTATZ $Sr(NO_3)_2$	189	1.89	2.55	TBD
BTSN-40	BTATZ $Sr(NO_3)_2$	1503	1.79	2.61	0.35[(c)]
BTSN-50	BTATZ $Sr(NO_3)_2$	1444	1.54	2.67	0.15

(a) The balance remaining in each formulation is made up of coolant, binder, opacifier and process aid.
(b) Reported burn rate corresponds to 17% coolant composition.
(c) Reported burn rate corresponds to 34% coolant composition.

Measurements of exhaust temperatures for these compositions were measured at several points along the exit streamline. These measurements indicate gas temperatures can vary widely, from below 200 °C at points ~ 30 cm from the gas generator to nearer 600 °C directly outside the generator.

Additive Development: Active Compositions

Chemical additives were incorporated into formulations wherein the chemically active agent is liberated upon combustion of the solid propellant, the exhaust consisting of inert gases plus entrained additives. Chemically active additives tested in the course of this project include various alkali metal salts such as acetates, carbonates and bicarbonates, halides, and polyhalogenated aromatics (pentabromophenyl ether). These additives (or their precursor) were blended directly into the propellant for SPGG (or HFE) delivery, or the additive (or precursor)

was blended directly into the hybrid fluid for HFE delivery. Several compositions were developed such that a common composition "family" evolved having different levels of additive.

We examined a series of compositions that produced potassium carbonate in the exhaust. Potassium carbonate was incorporated into the propellant blend either as the additive itself, or as the precursor potassium nitrate. In the latter case, the partial pressure of carbon dioxide is sufficient to convert the potassium oxide (gas generation reaction product) to the carbonate:

$$KNO_3 + Fuel \rightarrow K_2O \xrightarrow{+CO_2} K_2CO_3 .$$

Processing of these different compositions greatly influenced their reactivity. High levels of K_2CO_3 itself tended to slow gas generation rates. Nevertheless, using the strategy outlined above, concentrations of K_2CO_3 in the exhaust were varied over a range of 4x, and evaluated for suppression effectiveness in the GD Fire Test Fixture as described in the following section.

Fire Test Fixture Testing: SPGG

The majority of the fire test data presented in this paper were collected during the latter part of phase II and the first part of phase III. The results from the first part of phase II were presented in a previous paper[2], but selected data is included in this paper for comparison.

Both inert and chemically active propellant can be utilized in SPGG devices, and these devices are effective fire suppression tools. SPGG technology is able to produce large quantities of inert gases, such as nitrogen, carbon dioxide and water, in a very rapid fashion. Along with the inert gases, chemically active species are volatilized and delivered to the fire when chemically active propellant is incorporated into an SPGG. A typical SPGG device is illustrated in Figure 5 below.

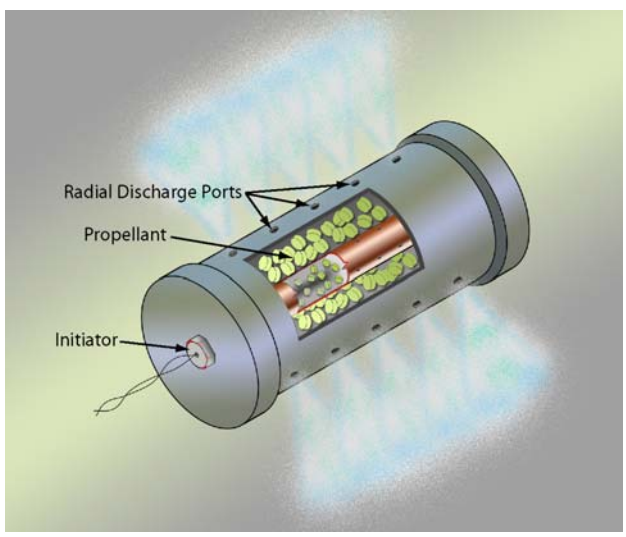


Figure 5: SPGG

The inert SPGG device that utilizes GD's FS01-40 propellant was chosen as a baseline for comparison. The relative effectiveness of each SPGG configuration is determined by a normalized FSN (FS01-40) value, which is equal to the agent threshold value divided by the FS01-40 threshold value. GD's active GD-04 propellant blend was shown to have the greatest fire suppression efficiency during Phase II testing[2]. The Phase III SPGG test results are

summarized in Table 2 (FS01-40 and GD-04 test results are included for comparison). The values represent the threshold amount of agent needed to extinguish the fire. The threshold amount was typically determined by the following procedure:

1. Determine initial agent load and conduct a live fire test.
2. Increase (or decrease) agent load until fire is extinguished (or not extinguished) to determine threshold value.
3. Conduct repeated tests at the suspected threshold value.
4. Define threshold value as amount of agent needed to extinguish the fire at least two out of three times.

Table 2. SPGG FTF Data Summary

Agent	FS01-40	GD-04[a]	GD-05[a]	GD-06[c]
Active additive	-	K_2CO_3	K_2CO_3	K_2CO_3
Gas Fraction	50%	50%	50%	50%
MW, g/mole	30	30	30	30
Mole active (K)/100g	0	0.145	0.0724	0.145
GG Load	347	105	157[b]	105
Discharge mass, g	173.5	52.5	78.5	52.5
Mole active (K) discharged	0	0.152	0.114	0.152
FSN(FS01-40)	1	0.303	0.454	0.303

(a) K_2CO_3 incorporated into the propellant blend as an additive.

(b) Anticipated threshold value: only 1 test was conducted using 157 g (fire out), two tests were conducted using 105 g (both fires not out), and one test was conducted using 189 g (fire out).

(c) KNO_3 incorporated into the propellant blend as an additive.

(d)

Fire Test Fixture Testing: HFE

Hybrid Fire Extinguishers combine a SPGG with a fire suppressing fluid (referred to as hybrid fluid in this document). The exhaust gases from the SPGG are used to pressurize the fluid to a specified pressure range and then the fluid is discharged. Figure 6 is shown below and it contains an illustration of a HFE.

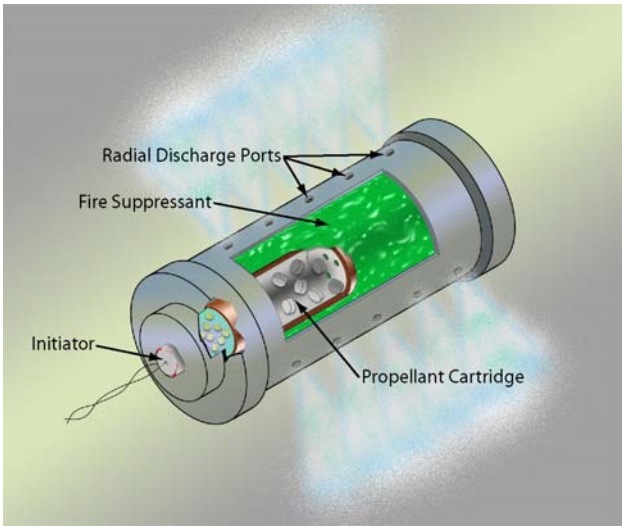


Figure 6: HFE

HFE devices are also able to utilize inert and active agents. The active agents in an HFE can be incorporated either into the propellant or the hybrid fluid. This paper includes test results from the following agents:

- Propellant:
  - FS01-40 (inert)
  - GD-02 (chemically active – KI)
  - GD-04 (chemically active – K_2CO_3)
- Hybrid Fluid:
  - Water (inert)
  - Water:Potassium acetate:Soap solution 48:48:4 by weight (chemically active – $KC_2H_3O_2$)
  - HFC-227 (inert)
  - 3M Novec™ Engineered Fluid HFE-7100 (inert)
  - 3M Novec™ 1230 Fire Protection Fluid (inert)
  - CF_3I (chemically active – CF_3I)

Again selected results from the first part of Phase II testing have been included for comparison[2]. The inert HFE with GD's FS01-40 propellant and HFC-227 hybrid fluid was used as the baseline and a normalized FSN (FS01-40/HFC-227) value is used to evaluate the effectiveness of the other HFE configurations.

The results of HFE testing have been broken into two separate categories: fluorocarbon systems and aqueous systems. The fluorocarbon test results are listed in Table 3 and the aqueous test results are listed in Table 4.

Table 3. HFE Fluorocarbon System Data Summary

Agent	FS01-40/ HFC-227[(a)]	FS01-40/ HFE-7100	FS01-40/ Novec 1230	GD-04/ HFC-227[(a)]	FS01-40/ CF_3I
Active additive	-	-	-	K_2CO_3	CF_3I
MW, g/mol	170	250	316	170	196
HFE Load (g)	358	-	358	228	98[(c)]
Discharge mass, g	340	-	340	217	93
Mole (K or I) discharged	0	-	0	0.046	0.429
FSN (FS01-40/HFC-227)	1	>1[(b)]	1	0.637	0.274

(a) Tested during the first part of Phase II[2].

(b) Tests conducted using 358 g (same agent weight as the FS01-40/HFC-227 system) did not extinguish the fire. Testing was suspended since the amount of agent needed would be more than the baseline system.

(c) Anticipated threshold value: two tests were conducted using 130 g (both fires out) and two tests were conducted using 98 g (one fire out, one fire not out).

The threshold values in the above table were determined by conducting ambient tests. An additional test of the 130 g FS01-40/CF_3I HFE was conducted after cold soaking the HFE and it also extinguished the fire. The HFE was conditioned to –65 °C, but the CF_3I temperature rose to –27 °C before the HFE was operated (note the boiling point of CF_3I is –22.5 °C).

Table 4. HFE Aqueous System Data Summary

Agent	GD-02/ Water	GD-04/ Water	FS01-40/ Water:KOAc:Soap[(b)]
Active additive	KI	K_2CO_3	$KC_2H_3O_2$
MW, g/mol	18	18	30
HFE Load	-	-	228
Discharge mass, g	-	-	217
Mole (K) discharged	-	-	0.96
FSN (FS01-40/HFC-227)	>1[(a)]	>1[(a)]	0.637

(a) Testing conducted using 358 g (same agent weight as the FS01-40/HFC-227 system) did not extinguish the fire. Testing was suspended since the amount of agent needed would be more than the baseline system.

(b) 48:48:4 weight ratio of Water:Potassium acetate:Soap.

Discussion

Direct incorporation of coolant compounds into the propellant composition was shown to be an effective means for reducing exhaust temperatures, typically resulting in a linear relationship between T_c and mass-fraction of coolant, as well as a linear ln(burn rate) vs. $1/T_c$ relation, indicative of an activated reaction process. While direct prediction of burn rates does not appear possible using these relations, trends can be predicted within a composition family.

Propellant formulations incorporating the new high nitrogen compound BTATZ ($C_4H_4N_{14}$), while structurally similar to the 5-aminotetrazole (5AT) fuel used in the GD FS01-40 propellant (Figure 4), appear to provide increased means for reducing propellant combustion temperatures. Several new BTATZ compositions were formulated to yield cooler exhausts. Preliminary ballistic testing indicates that burn rates may be maintained within workable constraints at the same time that exhaust temperatures are reduced below current baseline levels.

The presence of chemically active additives greatly enhances the fire suppression efficiency of SPGG and HFE devices. The most effective solid propellant composition tested remains GD-04, which delivers a mixture of an inert gas blend with potassium carbonate additive. Previous testing indicated that GD-04 is nearly 3x more effective than the inert FS01-40 and nearly 1.5x more effective than compositions having an equimolar amount of potassium iodide (GD-02)[2]. Testing reported here indicates that reducing the potassium carbonate by 50% results in a falloff in effectiveness versus GD-04, to a level comparable to GD-02. In light of reports from other workers[6,7] indicating a saturation of chemically active agent effectiveness beyond some critical concentration, it appears that these new compositions (e.g. GD-05) contain additives at levels below saturation.

As noted above, the propellant weight of an SPGG can be decreased by 50-70% with the addition of active additive, but the HFE weight was only reduced by approximately 30% during previous testing[2]. We theorized that the difference in weight reduction was due to the fact that the active propellant comprised 100% of the total SPGG weight, but only 14% of the HFE agent weight, thus limiting the molar fraction of chemically active additive[2]. The results from the FS01-40/CF_3I and FS01-40/Water:KOAc:Soap HFE tests support this theory. The molar fraction of the chemically active additive was increased and the results show a 64% weight reduction for the FS01-40/CF_3I HFE (versus FS01-40/HFC-227) and a >36% weight reduction for the FS01-40/Water:KOAc:Soap HFE (versus GD-04/Water). Figure 7 below illustrates these results.

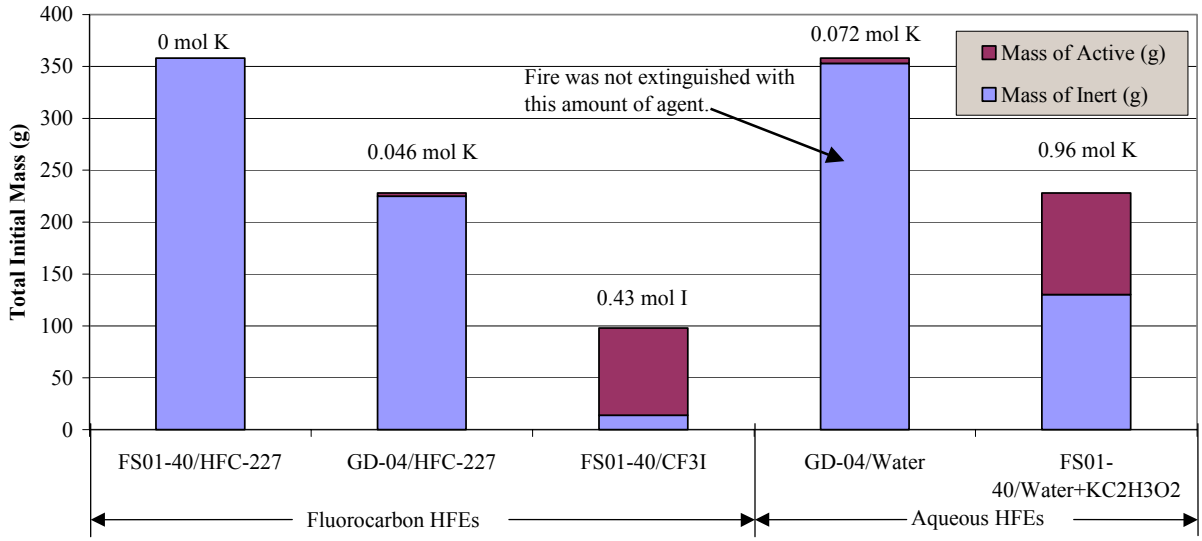


Figure 7: HFE Results – Decrease in Total Agent with Increase in Active Additive

HFE testing with aqueous agents provided additional insight into necessary levels of active agent required to effect suppression. Tests using an inert propellant configuration with pure water were found to require agent loads in excess of threshold levels of inert propellants and HFC-227. Similar findings were obtained when the active GD-04 propellant was used in conjunction with pure water. Previous work[2] had demonstrated a 30% performance enhancement in HFC-227 HFEs when substituting GD-04 for the inert FS01-40 propellant composition, thereby suggesting that the threshold level for FS01-40/water HFE is >30% over the FS01-40/HFC-227 threshold. This relatively poor performance of the water hybrids is not well characterized at present, but is likely a function of insufficient distribution of the hybrid stream into the fire compartment of the FTF. The effectiveness of the water-potassium acetate HFEs demonstrates that taking advantage of chemically active additives can offset this insufficient distribution.

HFE testing demonstrates that higher-boiling fluids, such as HFE-7100 and Novec-1230, can be successfully discharged when pressurized by a solid propellant gas generator. This opens opportunities for alternate suppression fluids that, on the basis of vapor pressure alone, may not appear to be desirable candidates as fire suppressants. The decision to include HFE-7100 and Novec 1230 in our test matrix was motivated by earlier reports their effectiveness in cup burner testing[8,9] We were unable to extinguish fires using this agent when used in quantities up to the threshold determined for HFC-227 hybrid units. Several tests at this agent loading resulted in a post-discharge flare-up in the test fixture. Testing with FS01-40/Novec-1230 HFEs indicated that its effectiveness is, on a mass basis, comparable to that of FS01-40/HFC-227 hybrid units. This suggests that the overall heat capacity of both discharges is similar; modeling is underway to verify this.

The lowest HFE agent loads were found when using CF_3I as the hybrid fluid. Incorporation of CF_3I into a hybrid configuration was examined in light of its poor low temperature dispersion[10]. Coupling this agent with a solid propellant gas generator did result in improved performance at low temperature, with effectiveness levels approaching ambient temperature test levels. This

finding indicates that CF_3I HFE configurations provide a viable solution to the use of CF_3I in low temperature applications.

Summary

A strategy for obtaining cooled propellant formulations incorporating the new high nitrogen compound BTATZ ($C_4H_4N_{14}$) has been defined, with preliminary results indicating the viability of these platforms for reducing propellant combustion temperatures. This strategy, involving direct incorporation of coolant species into the propellant composition, may be effective in reducing exhaust temperatures by as much as 30% vs. current baselines.

Fires testing with chemically active compositions indicate that the GD-04 composition is the most effective. This composition incorporates potassium carbonate in its discharge, and is ~3x more effective per unit mass than the inert baseline FS01-40. Testing with compositions of lower active-agent loading resulted in less effective performance. This indicates that the additive loading in GD-04 is below (or at) the saturation level reported in sub-scale testing with numerous other chemically active suppressants.

HFE testing with HFE-7100 and Novec-1230 indicate that high boiling agents such as these may warrant closer inspection than initially thought: their low vapor pressures can be offset by the heating and pressurizing power of the solid propellant driven HFE. Results with HFE-7100 were not as encouraging as initial indications suggested, and in fact resulted in flashback in fire tests. We found that FS01-40/Novec-1230 HFEs were comparably effective to FS01-40/HFC-227 HFEs on a mass basis. Hence other factors – e.g. atmospheric lifetimes – may provide the discrimination between their use. Incorporation of CF_3I into HFEs proved a useful means of overcoming its poor cold-temperature dispersion. However, while poor dispersion in the case of water-based HFEs probably resulted in their lackluster performance, this effect could be largely offset by blending in chemically active species such as potassium acetate.

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