# Advanced Propellant/Additive Development for Fire Suppressing Gas Generators

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The "Advanced Propellant/Additive Development for Gas Generator" project is a collaborative effort between Primex Aerospace Company (PAC) and the Naval Air Warfare Center-Weapons Division (NAWCWD) at China Lake, CA. The objective of this NGP project is to develop new highly efficient, environmentally acceptable, chemically active fire suppressant capabilities based upon solid propellant gas generators; improve understanding of propellant and additive effectiveness in fire suppression.

Cooled propellant formulations were developed based upon novel high-energy, high-nitrogen fuels in order to reduce overall combustion temperatures while maintaining ballistic robustness. These fuels included 5-amino tetrazole and the new high nitrogen compound BTATZ ( $C_4H_4N_{14}$ ) (courtesy of Mike Hiskey, Los Alamos National Laboratory), and were formulated with oxidizers such as potassium perchlorate or strontium nitrate, and an elastomeric binder added to facilitate pressing of pellets and increase abrasion resistance.

Chemical additives are incorporated into the propellant formulations, and take advantage of the high combustion temperatures to volatilize reactive agents and/or produce them directly via the propellant combustion process. A variety of additive candidates have been identified and subsequently categorized as "combustion" additives (e.g. decabromodiphenylether or metal compounds such as K(acetate) and ferrocene) which take an active role in propellant combustion, and "inert" additives (e.g. alkali metal halides or carbonates) which undergo no substantial role in the combustion process other than to decompose thermally.

Some of the identified chemical agents have been tested in a fire test fixture to evaluate their effectiveness in fire suppression for a controlled JP-8 fire. The agents were vaporized and delivered into the fire zone by high-temperature exhaust gases produced by a neutral-burning solid-propellant gas generator producing a blend of  $CO_2$ ,  $N_2$  and  $H_2O$ . Preliminary test results indicate that  $K_2CO_3$  and KI are more effective than KBr under similar test conditions. Results of this testing will be incorporated directly into propellant formulations and tested using the fire test fixture to assess effects of agent loading and combustion temperatures upon agent effectiveness.

This presentation will summarize results to date of propellant formulation and fire suppression effectiveness testing, and attempt to correlate additive effectiveness with additive composition and role in the propellant combustion process.

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## Introduction

Most replacement candidates for Halon-1301 are fluorocarbon agents like HFC-125 (C<sub>2</sub>HF<sub>5</sub>) and HFC-227 (or FM-200, C<sub>3</sub>HF<sub>7</sub>)[1]. The absence of heavy halogen atoms (like Cl, Br or I) make these agents primarily physically acting suppression agents, functioning through cooling and dilution effects[2]. As such, these replacement agents are much less efficient that Halon-1301 and so require greater concentrations to attain the same effectiveness.

A proven approach to non-Halon fire suppression is based upon technology similar to that used in automobile airbag devices. In this approach the fire suppression agent is a mixture of inert gases which are stored not in pressure bottles but in the form of solid propellants[3, 4]. On combustion in a solid propellant gas generator (SPGG), the solid propellant produces large quantities of inert gases such as nitrogen, carbon dioxide and water vapor. The compact nature of the SPGG device makes it a remarkably efficient means for chemically storing gaseous agents in a solid form. Early developments of this approach were pioneered at NAWC-China Lake in 1986[5] and later refined[6] and demonstrated in full scale testing by PAC in a variety of vehicle platforms, including military aircraft drybays and engine bays, military land vehicle engine and crew compartments, and commercial automobile engine compartments[4].

The current state of the art SPGG fire suppression system – PAC's FS01-40 – is size competitive with Halon-1301 systems on both mass and volume bases. It is clean, effective and fast acting, environmentally rugged and reliable, non-toxic and SNAP approved. Current products are based on commercial automotive airbag technology, thereby providing for a smooth transition from military to commercial applications.

A class of "superagents" has long been known, which exhibit fire suppression effectiveness much greater than Halon-1301[7]. The foremost example of these agents is iron pentacarbonyl, Fe(CO)<sub>5</sub>, but other examples include chromyl chloride, CrO<sub>2</sub>Cl<sub>2</sub>, and tetraethyl lead, Pb(C<sub>2</sub>H<sub>5</sub>)<sub>4</sub>, as well as powdered materials such as K<sub>3</sub>Fe(CN)<sub>6</sub>. While effective, these superagents are highly toxic, and so are not used in general suppression applications. However, much insight into the mechanism of chemically active fire suppression has been gleaned by a study of the chemical kinetics associated with the action of these superagents upon hydrocarbon combustion[8, 9]. For instance, chemical kinetic modeling of "superagent" suppressants indicates that the Fe(CO)<sub>5</sub>-based scavengers, or iron oxide species like FeO and FeOH, are more effective than halogens (like I•, Br•) in flame suppression, because they are more easily regenerated. The key to iron pentacarbonyl's effectiveness is the gas phase iron-oxo chemistry

Next-generation SPGG fire suppression systems will capitalize upon the current technology and further enhance performance by utilizing chemically active additives such as found in "superagent" sysems.. SPGG technology provides an extremely flexible agent platform whereby one can readily incorporate additives which can be converted into the high-performance metal-oxo species of the superagents – without the associated toxicity risk[10]. These next generation systems are expected to provide a means to reduce agent loads by 2–5x compared to current baselines.

In this NGP project, the PAC/NAWC team has developed propellant compositions that provide significant improvements in fire protection performance. These developments have resulted in cooler, more efficient and more compact fire suppression agent systems. These accomplishments are made possible by taking advantage of the great flexibility of solid propellant technology.

### Results

Initial efforts on the PAC/NAWCWD, China Lake NGP effort have considered propellant modifications that can be readily compared with PAC's FS01-40 chemically inert solid propellant formulation. The formulations studied on the NGP program have thus far attempted to both reduce combustion temperatures – by increased levels of nitrogen generation and coolant addition – and incorporation of additives to provide some amount of chemical activity to the propellant exhaust.

# **Cooled Propellant Formulations**

Improved cooling and more efficient agent generation have been accomplished via variations in the overall solid propellant composition. Different combinations of fuels and oxidizers have been modeled and tested; these results are summarized in Table 1. Several high-nitrogen fuels were considered (see Figure 1). These additives increased the production of the inerting agent nitrogen. Several compositions have been designed to deliver agent at a very rapid rate; this rate can be tailored by the incorporation of endothermically acting chemical coolants.

One reduced combustion temperature formulation is based upon 5-amino tetrazole(5-AT) and potassium perchlorate (KP), with an elastomeric binder added to facilitate pressing of pellets and increase abrasion resistance. This formulation burns well at 1000 psi (BR1000 = 0.2 in/s) but is somewhat sensitive to combustion pressure (n=0.8); this sensitivity limits commercial use of the formulation.

A new high nitrogen compound BTATZ (C<sub>4</sub>H<sub>4</sub>N<sub>14</sub>) (courtesy of Mike Hiskey, Los Alamos National Laboratory) is currently under evaluation. Initial results indicate that this compound burns well at low pressures and the burn rate has a low dependence on pressure (slope = 0.2, to be confirmed). Impact, friction and electrostatic sensitivity data have been obtained. We have included BTATZ in several formulations in combination with 5-AT in an attempt to increase efficiency of combustion while keeping oxidizer content to a minimum. A composition (with 10% KP has a very low dependence of burn rate on pressure, with calculated combustion temperature approximately 1700 °K (2600 °F), and gas production efficiencies yield over 4 mol gas/100g, nearly 60% of which is N2.

Other high nitrogen compounds[11] – e.g. GAZT, BT, GBT and TAGN – were tested in formulations and in thermochemical calculations. At this level of added oxidizer and with no coolants, all of the formulations exhibited high combustion temperatures and very high burn rates. fact that the burn rates were so high indicates that at a lower level of oxidizer (or by addition of an oxidizer that promotes cooler burning) combustion will be efficient in terms of nitrogen generation as well as burning cooler.

Figure 1: High-nitrogen fuels used in CL/PAC propellant development

**Table 1: Cool, High Nitrogen Developmental Propellant Compositions** 

Composition	Current SOA	5AT	5AT/BTATZ	5AT/BTATZ	
			(1)	(2)	
Exhaust species (vol% @ T <sub>c</sub> ))	N2 45 CO2 35 H2O 20	N2 57.1 H2 35.4 CO 7.5 KCl(s) 5.3g	N2 66.4 H2 32.1	N2 61.4 H2 30.2 CO 8.5 KCl(s) 5.3g	
Tex, °K (°F)	1000 (1400)	874 (1113)	908 (1175)	967 (1281)	
Gas Output, mol/100g (mol/100cc)	2 (5)	4.41 (7.41)	4.14 (6.87)	4.01 (6.86)	
Pressure exponent, n	0.5	0.80	1.6 (500-1000psi)	0.7 (1000-2750 psi)	
BR <sub>1000</sub> , in/s	0.5	0.17	0.55	1.31	

Composition	GAZT	BT	GBT	TAGN	
Exhaust species (vol% @ T <sub>c</sub> )	N <sub>2</sub> 37.0 H <sub>2</sub> O 28.3 CO <sub>2</sub> 15.3 CO 11	$\begin{array}{c cccc} B & H_2^2O & 15 \\ CO_2 & 25 \end{array}$	H.6 N2 42.9   5.0 H2O 28.8   5.8 CO2 20.1   6.6 CO 12.8	N <sub>2</sub> 28.3 H <sub>2</sub> O 40.6 CO <sub>2</sub> 16.4 O <sub>2</sub> 11.5	
	KCl(s) 32.	$6g \mid KCl(s) \mid 33$	3.6g   KCl(s) 33.1g	KCl(s) 5.3g	
Tex, °K (°F)	1436 (2125)	1761 (2711)	1457 (2163)	1421 (2098)	
Gas Output, mol/100g (mol/100cc)	2.51 (4.99)	2.13 (4.28)	2.19 (4.40)	2.44 (4.86)	
Pressure exponent	ND	ND	ND	ND	
BR <sub>1000</sub> , in/s	>2.5	>2.5	>2.5	>2.5	

## Chemically active additives

Fire suppression effectiveness of SPGG systems was also increased by incorporation of additives capable on interfering with, and termination of, radical processes associated with hydrocarbon combustion. Because the operational conditions <u>inside</u> a SPGG are similar to those in a fire, radical-trapping intermediates can be generated inside the GG and delivered with the inert gas coolant to the fire zone. Some additives tested in the course of this project include various alkali halides (e.g. KBr, KI), alkali carbonates (e.g. K2CO3) and polyhalogenated aromatics (pentabromophenyl ether). The physical properties of several chemically active suppressant compositions are summarized in Table 2.

**Table 2: Properties of Chemically Active Developmental Propellant Compositions** 

Composition	Current SOA	0807-Mod KN	0807-Mod K- Carb	0807-ModD	5AT/DECABr	
Exhaust species (vol% @ T <sub>c</sub> , or g/100g propellant)	N <sub>2</sub> 45 CO <sub>2</sub> 35 H <sub>2</sub> O 20	N2 44.1 CO2 35.2 H2O 20.7 (K2CO3 9.0g) (0.13 mol K)	N2 42.6 CO2 37.6 H2O 19.9 (KCO3 9.0g) (0.13 mol K)	N2 44 CO2 36 H2O 20 Fe2O3 5g (0.06 mol Fe)	N <sub>2</sub> 56.1 H <sub>2</sub> 34.0 CO 7.8 KCl(s)2.0g KBr(s) 5.4g	
T <sub>ex</sub> , °K (°F)	1000 (1400)	1102 (1524)	1206 (1711)	1098 (1517)	874 (1113)	
Gas Output, mol/100g (mol/100cc)	(5)	1.61	1.78 (4.09)	1.88 (4.87)	4.24 (7.29)	
Pressure exponent	0.5	0.66	0.59	0.62	1.0	
BR <sub>1000</sub> , in/s	0.5	0.41	0.52	0.64	0.1	

Two different approaches were evaluated for incorporation of potassium into propellant exhaust: one begins with potassium nitrate, the other with potassium carbonate. Both formulations resulted in slightly reduced gaseous agent output when compared to an inert baseline composition, but had comparable ballistic characteristics. Likewise, iron oxide containing formulations exhibited viable rates of agent generation.

Propellant compositions were formulated both with and without pentalbromophenyl ether, added as a chemically active flame inhibitor, and underwent preliminary ballistic characterization. Larger pellets of the chemically active formulation were burned in a motor and the temperature of the gaseous products measured. The outlet (nozzle) temperature was approximately 675°C which was in good agreement with the temperature predicted from thermochemical calculations. The chemically active formulation was also found to be impact, friction and electrostatically insensitive. Suppression effectiveness was demonstrated by directing the exhaust from propellant combusted in an Adam Motor into a 2 ft3 vented box, where it successfully extinguished a JP-8 pool fire. UV camera data indicated that no H2 burned during the discharge event.

# SPGG's and Fire Effectiveness Testing

Fire suppression effectiveness testing was undertaken using a mid-scale Fire Test Fixture (FTF), developed by PAC and representative of typical military aircraft fire scenarios (see, e.g. reference[12]). Operational parameters for the PAC FTF are given in Table 3, with a drawing of the fixture in Figure 3.

Table 3: Summary of Test Fixture Parameters, PAC Fire Test Fixture

Airflow rate		Stoichiometry	
Mass flow rate, m•, g/s	384	m• <sub>air</sub> /m• <sub>fue</sub>	19
(lbm/s)	0.85	Equivalence Ratio	
Volumetric Flow Rate, L/s	316		
$(ft^3/s)$	11	Fire Zone Dimensions	
Linear Flow Rate, cm/s	670	T, °K (°F)	1200 (1700)
	(in pipe)		
(ft/s)	22 (in pipe)	Intensity, kW	1000
		Length, cm	180
Fuel Flow Rate		Cross-Sectional Area, cm <sup>2</sup>	3700
Fuel	JP-8	Volume, L	670
Mass flow Rate, m•, g/s	20.23	V, ft <sup>3</sup>	24
(lbm/s)	0.0446	residence time, s	0.27
Volumetric flow rate, mL/s	25		
gal/s	0.0661	Injection Interval, ms	Up to 8000

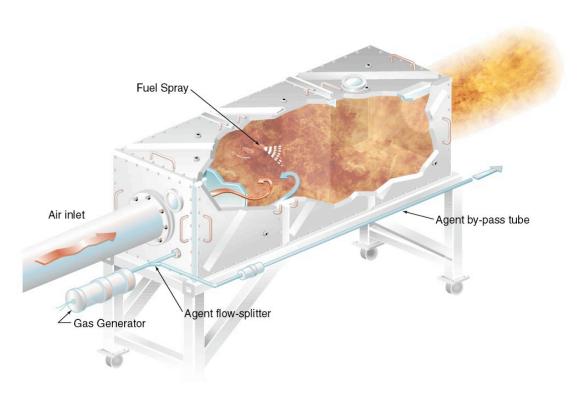
The fixture is located in a concrete reinforced test cell. The floor is painted with fuel-resistant, non-absorbing material. The test facility consists of several major subsystems including:

- Main Test Chamber
- Air Supply System
- Fuel Supply System
- Ignition System
- Suppressant Discharge System
- CO<sub>2</sub> Emergency Extinguishing System
- Control and Data Acquisition System:

## NGP FIRE TEST CONFIGURATION

For tests conducted under this program, the test fixture was usually operated at air to fuel mass ratio of about 19. The fuel flow rate was about 0.04 lbm/sec (or 20 gm/sec). Using a fuel heating value of 46.4 MJ/kg, the fire intensity was about 1.0 MW.

Selected chemical agents were tested in Fire Test Fixture to evaluate their effectiveness in fire suppression for the controlled JP-8 fire. The agents were loaded inside the discharge section of a neutral-burning solid-propellant gas generator (SPGG) (Figure 4). The exhaust of SPGG consisted mainly of CO<sub>2</sub>, N<sub>2</sub> and H<sub>2</sub>O. During SPGG discharge, the chemical agents were supposed to be vaporized and delivered into the fire zone by the high-pressure and high-temperature exhaust gases. Fine-grid metallic meshes and porous plates were used downstream of the agents to enhance the mixing between SPGG exhaust and agents prior to entering the distribution tube.



**Figure 3: PAC Fire Test Fixture** 

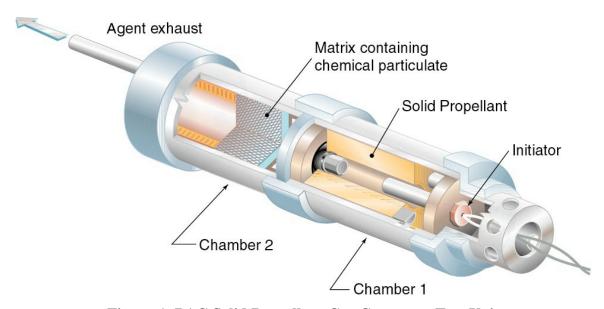


Figure 4: PAC Solid Propellant Gas Generator Test Unit

The distribution tube was split into two lines, one enters the test fixture, and the other discharges directly to ambient (Figure 4). By varying the split ratio between these two lines, the amount of agent entering the fire zone can be controlled. In the tests, the split ratio was fixed to be 50%, and the initial agent quantity in the SPGG was varied. Thus, the objective of the tests was to determine the amount of initial agent required to put the JP-8 fire out. The relative efficiency of different agents was then ranked based upon their threshold quantity.

Various instrumentation was installed in the SPGG and test fixture to monitor and control test variables, and to make sure they are in the similar ranges from test to test. The SPGG was fired and the agent discharged after a steady-state fire was established. A waiting period of 45 seconds between JP-8 fire on and SPGG firing was kept to assure steady-state, repeatable test conditions.

Some of the identified chemical agents, including KI, KBr, K2CO3, Fe2O3, Fe(oxalate), Ferrocene and pentabromophenyl ether, were tested in a fire test fixture to evaluate their effectiveness in fire suppression for a controlled JP-8 fire. The results of this testing are summarized in Table 4. The agents were vaporized and delivered into the fire zone by high-temperature exhaust gases produced by a neutral-burning solid-propellant gas generator producing a blend of CO2, N2 and H2O.

The test results indicated that K2CO3 and KI are more effective than KBr in suppressing JP-8 fire in PAC's Fire Test Fixture. The threshold amount for successful fire extinguishment seemed to be around 40 g of neat K2CO3 or KI powder used in each SPGG. As evidenced by some tests using agents with more distinctive colors, the powder seemed to be ejected out of the SPGG throughout the entire test duration of about 7.5 to 8 seconds. Testing with ferrocene resulted in growth of needles of ferrocene crystals on the exit port of the delivery tube.

**Table 4: Summary of NGP FTF Data** 

Neat agent	wt. (gm)	Test No.	Result	Neat agent	wt.	Test No.	Result
					(g)		
KI	20	027-06	fire not out	Fe2O3	40	038-01	fire not out
	40	027-07	fire not out		80	038-02	fire not out
	40	027-05	fire out				
	60	027-04	fire out				
KBr	40	032-02	fire not out	Ferrocene	40	039-01	fire not out
	60	032-03	fire not out		80	039-02r	fire not out
	60	032-01	fire not out				
K2CO3	20	035-03	fire not out	Iron	40	040-01	fire not out
				Oxalate			
	40	035-02r	fire out	PBPE	60	041-01	fire not out
	60	035-01	fire out				

## Discussion

The cooler, high gas output propellant compositions described in Table 1 represent an important step towards increased efficiency SPGG fire suppression devices. Gas temperatures were reduced in some case by 10-20% when compared to the current baseline. The findings of increased burn rate compositions, while maintaining relatively low gas temperatures, provides a means for further increases in agent cooling when these compositions are further modified with endothermic chemical coolants.

Compositions incorporating combustion inhibitors were demonstrated to burn efficiently and were shown to be effective in fire suppression. It is interesting to note that the incorporation of species to suppress fires does not prevent combustion of the propellant.

Since the key to superagent effectiveness – e.g. iron pentacarbonyl's – is the gas phase iron-oxo chemistry[8, 9], we considered developing alternative means to generating gas-phase or fine particulate iron oxide – or other metallo-oxo species. This route then provides a means of delivering the chemical activity of Fe(CO)5 without the associated toxicity. The routes explored to date – involving different means for potassium-oxo and iron-oxo containing discharges, have yielded ballistic characteristics that are amenable to further study.

With the test results available, it seemed that the iron-based agents and PBPE are less effective than KI and K2CO3, at least within PAC's Fire Test Fixture environment. The similarity in performance between the iodide and the carbonate is similar to that observed in turbulent spray burner testing at PAC; that data was interpreted as reflective of the greater role of the potassium ion in suppression effectiveness rather than the anion.

Assuming a uniform agent discharge, the agent to propellant weight ratio is about 4.3% for threshold KI quantity. This is significantly lower than the 21% of KI in one of PAC's active propellants. Since PAC's KI-based active propellant has already been proven to be very effective in suppressing fires in some large-scale testing, the SPGG and propellant design could be further optimized to reduce weight and volume.

The lesser performance of the iron- and pentabromo species seems to conflict with observations in some of the earlier literature. Several possible causes might contribute to this discrepancy. The residence time – and/or time at temperature for the given particle size of agent – may not be long enough for the agents to break down. Inadequate time at temperature is supported by the observation of ferrocene crystal growth at the end of the delivery tube – implying sufficient stability of ferrocene in air at  $\sim 1000$  °C for 0.3 s. Together, this indicates that adequate agent delivery mechanism need to be carefully designed and implemented in the solid-propellant fire extinguisher in order to fully utilize the chemical capabilities of such agents.

Although we have tried to keep all of the test variables as consistent as possible from test to test, the nature of this type of tests has shown certain degree of poor-repeatability in the test results. This non-repeatability is representative of the large-scale fire tests that PAC has participated in the past. It is also possible that the poor-repeatability in some of the tests was due to the fact that the agent quantity used was in the threshold range. The threshold quantity may not be a sharp line separating the regions between fire out and fire not out. It might be a band accommodating variations in test variables.

As a caution, ferrocene and its derivatives have been used in propellants as burn rate modifiers with great success. However these compounds tend to migrate to the surface of the propellant where they are oxidized to Ferrocinium compounds. Ferrocinium derivatives in combination with propellant ingredients gives friction sensitive materials[13]. This would tend to limit the use of ferrocene derivatives to hybrid systems where the propellant and the additive were stored separately.

## Summary

Several key findings have been made to date, whereby cooler propellant compositions have been used in conjunction with chemically active additives, or combustion radical scavengers. Adding "radical traps" to the exhaust provides means for cooling, dilution and chemical termination of the combustion process, hence increasing the overall effectiveness of the fire suppressant. Current propellant formulations are lighter in weight than Halon-1301 for comparable fire suppression effectiveness, but without the hazards associated with Halon's ozone depletion capability.

The findings of this year's NGP effort suggests a more in-depth examination of the role and optimal delivery requirements for highly active, radical-scavenging chemical additives as fire suppressants. In addition to "pure" solid propellant approaches, the use of solid propellant/liquid (or gaseous) agent "hybrid" technologies shall also be explored.

Propellant development activities are planned in efforts to further optimize cool-burning, highnitrogen content solid. Additionally, chemically active additives will be incorporated directly into propellant formulations and tested using the fire test fixture to assess effects of agent loading and combustion temperatures upon agent effectiveness.

Further optimization testing and of these compositions – in terms of agent temperatures, additive identity and loading – can be expected to result in substantial reductions in agent requirements. A system utilizing high-output, cool gas and chemical activity can be expected to result in agent loads of 1/3 - 1/5 that of the current baseline.

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