n-PROPYL BROMIDE AND BROMOALKANE TESTING

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INTRODUCTION

The Montreal Protocol on substances that deplete the ozone layer and the U.S. Clean Air Act of 1990 originally ceased the production of halons by the year 2000. The Copenhagen Amendments accelerated the production phaseout to 1 January 1994. "DoD Directive 6059.9" directs DoD components to conduct R&D on replacement agents and to adopt suitable substitutes that are consistent with mission requirements. The halon production phaseout has caused a need to develop and evaluate substitute agents for halons used to protect high-value military combat equipment. Under research sponsorship by the U.S. Air Force Wright Laboratories (USAF/WL), the Center for Global Environmental Technologies (CGET), at The University of New Mexico (UNM) has identified, developed, and tested a variety of halon replacements for use in large (150-lb) flightline fire extinguishers. This research has identified several candidate agents ("first- and second- generation" and "advanced-agents"). Several of these candidates are now being commercialized as streaming agents. Testing of the candidates has shown that although some compounds and blends are not **very** effective on large fires (150 ft') and in flightline extinguishers (requiring long throw distances), these compounds also demonstrate effectiveness on small-scale fires (< 10 ft^2) such as those experienced within combat vehicles. A project was initiated by the US Army to investigate further these potential replacements in handheld extinguishers on small fires applicable to combat vehicle specific applications. While testing commercially available compounds, CGET has also identified several advanced agents. A list of acceptable agent criteria has been included in Table 1.

Table 1. List of Acceptable Agent Criteria.

Criteria	Value
ODP	0.00
Atmospheric Lifetime	< 250 yrs.
GWP (100-yr relative to CO,)	< 4000
Availability	Being manufactured in bulk now or in the near future.
Effectiveness	Halon Weight Equivalent (WEq) < 2.0
Boiling Point Range	-5 to 200 °C
Toxicity	30-min, rat, LC_{50} > 5.0 vol. %, Cardiac NOAEL > 2.0 vol. %
Operating Temperature Range	-25 °F to 140 °F
Agent **Cost**	< $50 per lb

COMPOUND IDENTIFICATION AND SELECTION

CGET first proposed halocarbon compounds and blends of these compounds **as** halon replacements in 1989 [1]. The focus was on commercially available nonbrominated halocarbons. Several of these proposed candidates have been commercialized as fire extinguishing agents (Table **2).** However, the current commercially available halon replacements are less effective than the present halons in most scenarios, have marginally acceptable toxicity, and/or have some adverse global environmental impact (deplete ozone, high global warming, and/or long atmospheric lifetime). In 1995 CGET began investigating advance agents termed "tropode-gradable" halocarbons [2]. Preliminary testing of halogenated bromoalkene (HBA) compounds has demonstrated their utility as halon replacements when used in blends. In the fall of 1996, limited USAF funds were used to evaluate HBA blends as streaming agents [3]. In the spring of 1997, **TACOM** sponsored field-scale streaming and decomposition testing at CGET focusing on the commercially available hydrofluorocarbon (HFC) based halon replacement. Halon 1301 was tested as the baseline. During the TACOM project, limited HBA blend tests were performed [4]. In the summer of 1997, under ***USAF*** sponsorship, additional laboratory cup-burner tests and field-scale streaming jet fuel tests (comparing with Halon 1211) were performed [5]. This paper summarizes the test data that have been developed to date on HBA blends and makes recommendations for further research activities towards their applicability as halon streaming agent replacements for use in combat vehicle applications.

RELEVANT LITERATURE REVIEW

Sheinson et al. [6], Noto et al. [7], and Hammel et al. [8] have estimated the chemical and physical contributions of various compounds on fire extinguishment. For chemically acting agents (Halons 1211 and 1301, CF_3I, etc.), the chemical suppression contribution has been estimated to be 60 to 80% with **a** physical contribution estimated to be 20 to 40%. For physically acting agents (HFC-227ea, HFC-236fa, etc.), rhe suppression contribution has been estimated at 20 to **40%** chemical and 60 to 80% physical. Noto et al. [7] states that a chemical reaction saturation concentration is reached with chemically acting agents during the fire suppression process, and the physical contribution is, therefore, required to provide complete suppression. Thus, the use of **a** composite agent composed of **a** blend of an effective chemically acting agent ("chemical component") with **a** high heat capacity carrier ("diluent component') may function as an ideal halon replacement (e.g., HBA blends). This has been the rationale for developing the HBA blends discussed in this paper.

LABORATORY TEST RESULTS

Most tropodegradeable nonfluoroniated **HBA** compounds are listed as flammable in the chemical catalogs. Initially, the HBA I-bromopropane (n-propyl bromide, $CH_3CH_2CH_2Br$) was selected for evaluation. It has a low number of hydrogen atoms (an indication of flammability), the lowest known LC_{50} value, and is available at low cost. The compound is also being developed as **a** solvent replacement, and additional toxicity and global environmental information (e.g., ODP and atmospheric lifetime) are becoming available [9].

A synergistic effect was shown in the cup-burner data when various HBAs were blended with various carriers (e.g., adding 10 wt.% 1-bromopropane to 90 wt.% HFC-236fa reduced the cup-burner extinguishment concentration of pure HFC-236fa from 6.7 vol.% to 5.2 vol.% for the

Table 2. Commercially Available Halon Replacement Streaming Agents and Those Considered as Carriers During HBA Blend Tests.

Compound	Cardiac NOAEL, Vol.%	Cardiac LOAEL, Vol.%	Heptane Cup Burner Ext. Conc., vol.%	ATM. Lifetime, yrs.	GWP, 100 yr. Rel. to CO_2
"HCFC-123 (FE-232™)	1.0	2.0	6.3	1.4	93
[a]HCFC-124 (FE-241™)	1.0	2.5	6.7	6.0	480
[b]HFC-227ea (FM-200"')	9	10.5	6.3	41	3300
[b]HFC-236fa (FE-36™)	10	15	5.6	200	6300
[b]HFC-236ea	2.5	3.5	6.6	6.2	110
[b]HFPE-1164X	---	---	5.1	8	---
[a]HCFC Blend B (Halotron I)	–1.0	–2.0	–6.3	–1.4	–93
"HCFC Blend C (NAF P-III)	–1.0	–2.0	–6.4	1.4- to 15	93 to 1300
[a,b]NAF P-IV	–1.0	–2.0	–6.4	1.4- to 15	93 to 1300
FIC-1311 (Triodide™)	0.1	0.2	3.2	<1 dav	<5

"Compounds have an ODP value of –0.017.
[b]Considered as a potential carrier compound.

blend (Figure 1). Also, laboratory streaming tests of various blended compounds at a number of blend ratios showed that the flammability of the HBAs was nonexistent (depending on the blend ratio). Several tropodegradeable HBA blends have been investigated including flame suppression testing and acquiring known toxicity, cost, availability, and environmental effects data. Table **3** lists the most attractive HBAs considered to date.

NMERI FIELD TESTING

A variety of fire tests have been performed. Underwriter's Laboratories (UL) 2B, 5B, and 10B fire pans and test procedures were used (1-min preburn). The UL-2B pan was used for the primary testing, and the 5B and 10B pans were used to test each of the agents to its fullest extent. The test fuels included heptane and Jet **A-1** (ASTM D1655) (equivalent to JP-8), which were floated on 2 in of water, leaving a freeboard of 4 in. The extinguishers used included US Army 2.5-lb CO_2 portable extinguishers, Amerex™ handheld halon extinguishers, and a prototype extinguisher being developed by Metalcraft, Inc.

PORTABLE FIRE EXTINGUISHER CONSIDERATIONS

Nozzles—A critical element of the extinguisher is the nozzle. The nozzle affects two primary items: agent flow rate and pattern. An approach has been used whereby existing halon portable extinguisher nozzles are used for initial testing and modifications are made accordingly. Nozzle design parameters have been developed to predict accurate agent flow rates. At the same time

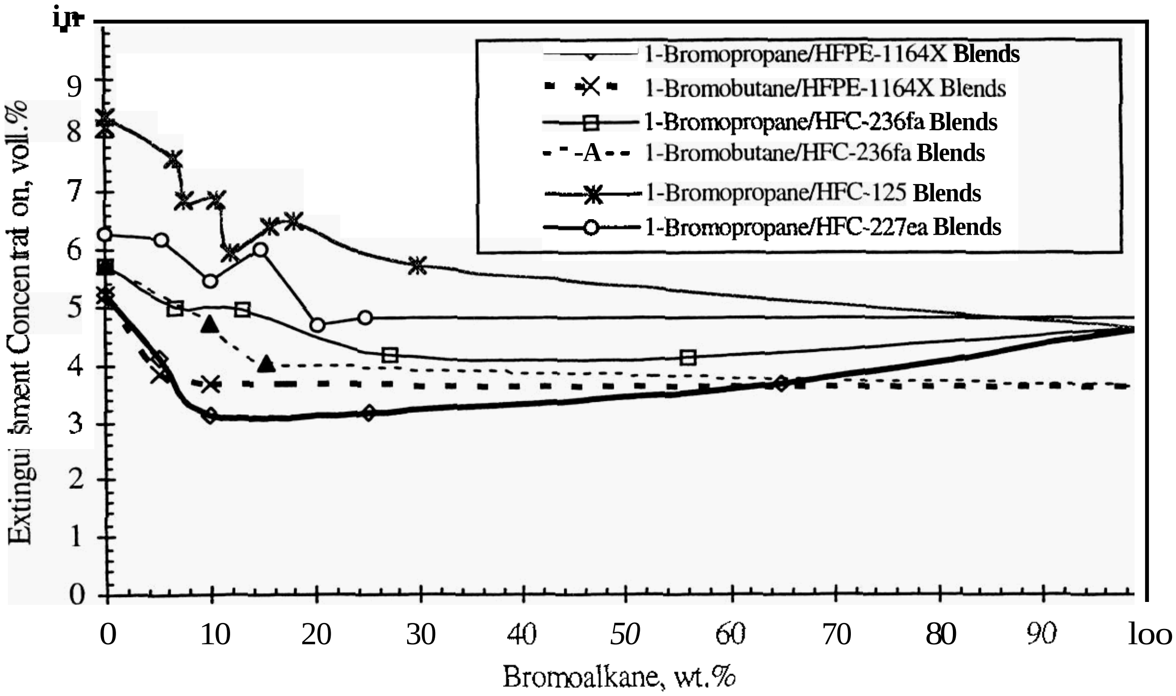

Figure 1. Extinguishment concentrations versus weight percentage of **HBA.**

Table 3. **HBAs** Considered **for** Blend Testing.

Compound Name	Mole wt., g/mol	Known Toxicity Value, vol.%	Heptane Cup Burner Ext. Conc., vol.%	Flash Point, °C	Carbon/ Bromine Ratio	Hydrogen / Bromine Ratio
1-Bromopropane	123.01	Rat ihl LC_{50}-30 min: 5.03	4.6	25	311	7/1
1-Bromobutane	137.04	Rat ihl LC_{50}-30 min: 4.2	---	23	4/1	911
1,2-Dibromopropane	201.90	**Rat** ihl LC_{50}-30 min: 0.19	---	none	1.5/1	311
1,3-Dibromobutane	215.93	---	---	none	211	411
2,3-Dibromobutane	215.93	---	---	>110	211	4/1
1,4-Dibromopentane	229.95	---	---	>110	2.5/1	511
1-Bromo-3-chloropropane	157.44	---	---	none	311	611

the nozzle needs to produce an effective pattern with optimum droplet sizes. The more liquid compounds tend to be completely ineffective when applied as a mist. Large droplets are required for effective extinguishment.

Liquid Fill Densities — The liquid fill density of an extinguisher refers to the liquid volume occupied by the extinguishing agent. The importance of fill density cannot be over-emphasized. Fill densities that are too high cause serious problems such as low flow rates. There is less room available for the nitrogen pressure charge. As a result, the pressure drops off more quickly during the agent discharge and causes a rapid decrease in agent flow rate. This problem is amplified when the extinguishers are cooled because cooling a pressurized extinguisher reduces the internal pressure. Another problem occurs when the extinguisher is heated. At elevated temperatures the internal pressure increases. In general, a 70 to 75% liquid fill is optimum.

Technique—The current commercial halon replacement streaming agents are very sensitive to firefighter technique. Technique is one of the most important variables with halon replacement agents and it must be treated as such. The objective is to use an effective optimized technique for each agent and to maintain this technique during testing, so that valid comparisons are made between agents. In general, streaming agents are initially applied to the front edge of the fire pan in a side-to-side sweeping motion, with approximately two sweeps per second. Once the fire "breaks" off the front edge of the pan, it is pushed to the back of the pan by applying the sweeping motion to the leading edge of the fire, maintaining the 70-deg "angle-of-attack." The side-to-side sweeping motion, i.e., keeping the agent directed at the base of the fire, is continued until the fire is extinguished (Figure 2).

FIRE SUPPRESSION **TEST** DATA

A variety of fields tests have been conducted including baseline streaming tests with Halons 1211 and 1301. Listed in Table 4 are summaries of tests conducted at CGET. **A** complete test listing is provided in References 3, 4, and 5, which give extinguisher size, nozzle size and type, charge pressure, discharge time, quantity discharged, and average flow rate for each test. As shown, the candidates listed in Table **4** extinguished the pool fires with small differences in weight requirements. However, slight extinguisher parameter changes significantly affected performance. With the right nozzle, fill density, pressure combination and technique, the test fires could be extinguished easily. Without the right combination, extinguishment could not be attained. The pure compounds generally required 2 to 3 times as much as halon to extinguish equivalent fires. The addition of the HBAs to the pure compounds proved to be the difference between extinguishment and nonextinguishment of most of the tested fires. The most effective blending percentages ranged from 10 to 15 wt.% HBA. A 30 to 40% improvement has been measured with the HBA blends. Although a limited number of tests have been performed, the results and firefighter comments signify a definite enhancement. A similar enhancement was seen with both the heptane and jet fuel tests. Note, a similar **30%** extinguishment improvement was measured using the cup burner.

Figure 2. Firefighter finishing attacking the fire. **As** shown, it is important to hold the extinguisher in an upright position, maintain a steep "angle-of-attack," keep the agent directed at the base of the fire ("at the leading edge"), and allow the agent concentration to build up over the fuel surface, while using a side-to-side sweeping motion.

Another factor to consider, when looking at the HBA blends, is technique. These blends provided a chemical action during the extinguishment process (chemical inhibition), similar to that afforded by the halons. The blends are less technique sensitive and tend to help compensate for varied techniques of different ("inexperienced") firefighters.

Funding and time limitations have prevented extensive repeatability testing of all the difference combinations of **HBA** and carrier combinations and optimization of extinguisher parameters. However, based upon the results to date, further optimization of the HBA blends for use in streaming applications, in particular with 1-bromopropane, is warranted.

NMERI DECOMPOSITION TESTING

Adding halons and/or halon replacements during the fire suppression event increases the amount and types of combustion products. The resulting species are characterized as decomposition products and can be severely toxic. In addition to increased carbon monoxide (CO) and carbon dioxide (CO_2), acid gases, such **as** hydrogen fluoride (HF) and hydrogen bromide (HBr), as well as carbonyl fluoride (COF_2), are formed. In some cases, chemical intermediaries (e.g., perfluoropropene from HFC-227ea) have also been identified. The decomposition product concentrations have been measured to be 10 to 1000 times those limits set by the Occupational Safety and Health Administration (OSHA) and other safety and health organizations. The concentration of toxic products generated by the current halon replacements generally exceeds levels generated by the existing halons by 2 to 10 times depending upon the fire scenario.

A limited series of preliminary tests was conducted to compare the decomposition product concentrations from one agent to another and not necessarily to measure the exposures one would expect in suppressing a typical fire. The decomposition product tests were conducted inside an enclosed 645-ft^3 (18.27-m^3) compartment using a 2.25-ft^2 fire pan and Jet A-1 fuel. The test compartment was equipped with an automatic ventilation system. Prior to agent discharge, the motorized damper was closed, and the ventilator was turned off. Immediately after the fire was

Table 4. Summary of Representative NMERI Streaming Test Fires.

Fire Size, Fuel, and Agent	Extinguishment Quantities, lbs	Extinguishment Time Range, sec	Flow Rates, lbs/sec	No. of Tests
2.25 ft^2 Heptane				
Halon 1211	0.28 - 0.65	1.1-4.0	0.12 - 0.47	18
HFPE-1164X	0.94, 1.22	2.4, 2.5	0.38, 0.49	2
HFC-236fa	1.7- 2.6	8.2 - 13	0.17 -0.21	4
HFPE/1-bromopropane	0.76 - 1.6	2.6 - 6.0	0.17 - 0.40	4
HFC-236fa/1-bromopropane	0.67 - 1.1	3.4 - 6.4	0.17 - 0.22	14
UL-2B (5.0 ft^2) Jet Fuel				
CO_2	0.98 - 1.7	5 - 10	---	3
HFFE-I 164X	2.1 - 4.6	8 -10	0.21 - 0.42	22
HFC-236fa	1.7 - 2.3	3.5 - 7.5	0.3 -0.5	11
HFC-227ea	1.1 -2.7	3.3 - 7.5	0.16 - 0.39	12
HFPE/1-bromopropane blends	3.0 - 1.8	6 - 8	0.3 - 0.4	5
HFC-227ea/1-bromopropane	1.1 - 1.7	3.7 - 5.7	0.3	3
UL-5B (12.5 ft^2) Jet Fuel				
Halon 1211	2.3	5	0.46	I
HFC-236fa	3.8	6 - 10	0.46 - 0.50	3
HFC-227ea	3.9	---	---	Unable to ext.
HFFE-I 164X	2.8	5	0.6	1
HFC-236fa/1-bromopropane	3.0	4 - 16	0.22 - 0.47	5
UL-5B (12.5 ft^2) Heotane				
Halon 1211	1.75	8	0.21	Typ. UL Ext.
HFC-236fa	6.0	6 - 10	0.64	Typ. UL Ext
Halotron I	5.0 - 6.0	8 - 10	0.60	Typ. UL Ext
HFC-236fa/1-bromopropane	3.4	6.0	0.28 - 0.59	8
UL-10B (25 ft^2) Jet Fuel				
Halon 1211	3.12	4.8	0.65	1
HFC-236fa	9.1	8 - 10	1.0	4
HFC-236fa/1-bromopropane	5.4 - 7.5	6 - 10	0.73 - 1.1	11
HFC-236fa/1,2,-dibromo-propane	6.4	6.5	0.98	1
HFC-236fa/1-bromobutane	8.4	8.6	0.98	1

extinguished, the compartment doors were closed and secured. The top vent of the chamber remained open throughout the test event, allowing convective ventilation to take place.

It was found that the quantity of decomposition products present is dependent upon the time required to extinguish the fire and the amount of extinguishing agent discharged (Table 5 and Figure 3). As shown in Table 5, the extinguishment time, discharge time, and amount of agent discharged varied for each of the tested agents. The HF and COF_2 concentrations exceeded those generated by with Halon 1301 by 2 to 3 times. HFC-227ea generates higher HF concentrations than HFC-236fa or HFPE-1164X. The lowest HF concentrations were generated with HFPE-1164X based upon equal extinguishment times. The addition of 1-bromopropane to HFC-227ea does not appear to reduce the amount of decomposition products generated for similar extinguishment times. The addition of I-bromopropane did reduce the extinguishment time; therefore, lower HF and COF_2 concentrations will result if the HFC-227ea/1-bromopropane blend is used. The HFC-236fa generated less HF than HFC-227ea and HFC-227ea blended with 1-bromopropane and slightly more than the HFPE-1164X. As expected, the least amount of HF was formed with Halon 1301. Interestingly enough, HF levels for the HFPE-1164X blended with 1-bromopropane were similar to Halon 1301. Higher levels of COF_2 were also measured during the HFC-227ea tests when compared to HFC-236fa. The HFPE-1164X generated higher COF_2 than either of the other tested compounds/blends. There was little to no COF_2 formed during the HFC-236fa and HFPE-1164X tests.

HALON 1211 VERSUS THE REPLACEMENTS

There are currently three halon replacement agents that are commercially available in Underwriters Laboratory (UL) listed extinguishers. These agents are HFC-227ea (FM-200™), HFC-236ea (FE-36™), and a HCFC-123 blend (Halotron I™). Another HCFC-123 blend (NAF-PIV) is also available in equipment listed in Europe. There have been numerous advertisements indicating that these replacements equal Halon 1211 in effectiveness. Therefore, an assessment of agent effectiveness compared to Halon 1211 has been performed.

There are three primary applications in which halon-like agents are used (1) Class A Fires - wood cribs, wood panels, computers, data acquisition equipment, cabling, clothing, etc.; (2) Class **B** Fires - aircraft flightlines, ships, including flightdecks, oil and gas facilities, chemical laboratories, etc.; and Class C Fires - telecommunication sites, etc. Also, combinations of these fire **types** are also possible. Table 6 shows the current Class A and B UL listed ratings and agent quantities for Halon 1211 and typical replacement agent extinguishers. These data, for n-heptane, as well as Jet-A1 (jet fuel) (Class B fires) data for the tested HBA blends, are shown in Figures 4 and 5.

Table *5*. Decomposition Product Test Data Comparison.

Agent	No. of Tests	Ave Ext. Time, sec	Ave Amt. of Agent, lbs	[a]Ave Max. HF Conc., ppm	[a]Ave Max COF_2 Conc., ppm	Ave. HF Conc. (Oto 15 min), ppm	Ave. COF_2 Conc. (0 to 15 min), ppm	Ave. HF Conc. (2 to 10 min), ppm	Ave. COF_2 Conc. (2to 10 min), ppm
[b]Halon 1301	3	5.3	0.6	370	0.07	260	0.0	270	0.0
HFC-236fa (FE-36)	3	4.0	0.9	720	1.2	405	0.17	450	0.05
HFC-227ea (FM-200)	4	4.0	1.3	1020	4.1	650	2.1	710	2.4
HFPE	*2*	7.6	2.4	780	7.3	545	4.7	550	5.3
[b]HFPE *I* Bromo Blend	1	2.1	0.75	485	1.5	280	0.2	310	0.0
[b]FM-200 / Bromo Blend	3	3.7	1.1	900	6.5	715	2.0	730	2.1

"Values based upon removal of the spike that occurred at the beginning (< 0.4 min) **of** the test.
[b] HBr levels were below the detection limit (<100 ppm) of the instrumentation.

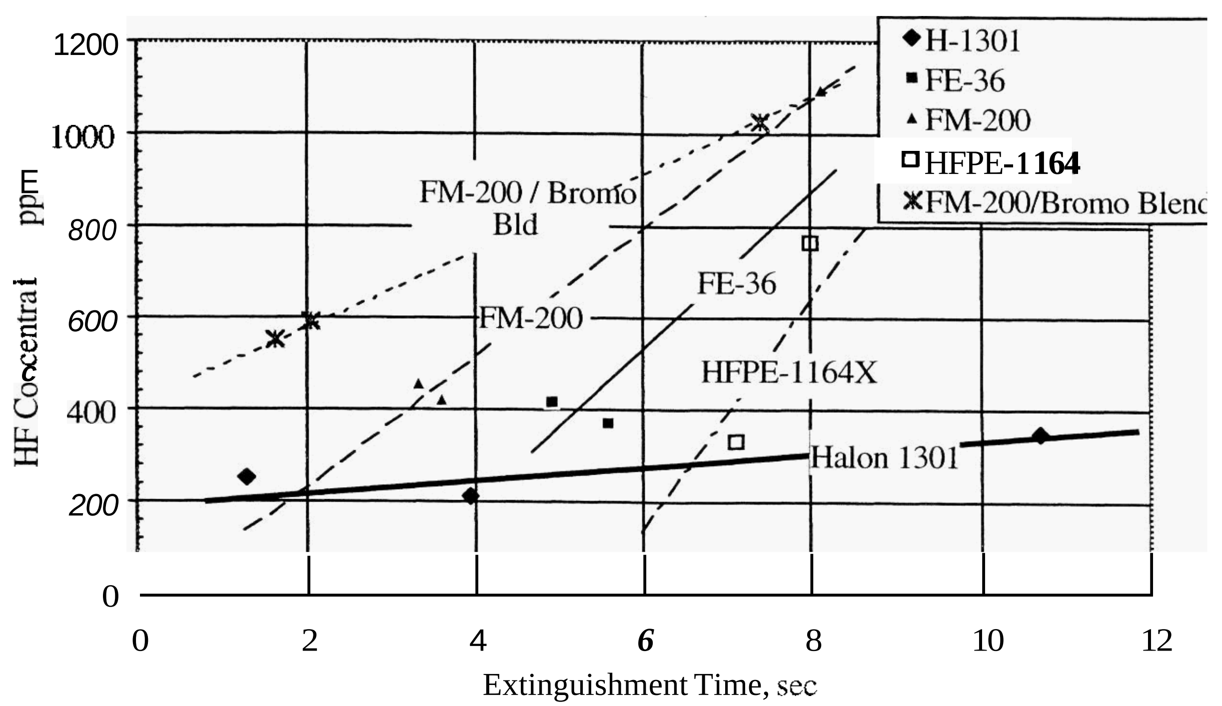

Figure 3. HF Concentration versus extinguishment time.

From an analysis of the above test results and manufacturers data two important conclusions are drawn: (1) the Class A performance of the replacement agents is similar to Halon 1211 and (2) Halon 1211 is 2 to **4** times more effective than the replacement agents on Class B fires (fires typical of combat vehicles, flightlines, and flightdecks). If you look strictly at the 1A 10B:C rated extinguisher data it appears that the FE-36™ and Halotron I extinguishers are indeed similar in effectiveness to Halon 1211 (e.g., 10 - 12 lbs of agent for the replacements versus 9 lbs for Halon 1211). Upon further investigation (e.g., looking at the 5B:C, and 2A rated extinguisher data), it is obvious that Halon 1211 is superior in performance to the commercial replacements (2 - 4 times more effective). To obtain a 5B:C rating the replacement agent extinguishers require over twice as much agent as Halon 1211 (e.g., **6** lbs versus 2.5 lbs for Halon 1211). When one looks at the 2A rated extinguishers, 13 lbs of Halon 1211 are required to obtain the 2A rating, a quantity approximately equivalent to the replacement agents; however, this quantity **of** Halon 1211 extinguishes a 40B:C fire (this is a Class B fire four times larger than the replacement agents will extinguish).

Using these data one can predict the quantity of replacement agent that would be required to he equivalent to a Halon 1211 20-lb portable (4A 80B:C) or 150-lb wheeled (240B:C) extinguisher. Figures 6 and 7 show that quantities of 100 lbs and 290 lbs of replacement agent, respectively, would be required (e.g., 2 - *4* times more replacement agent than Halon 1211 to extinguish the same Class B fire).

CONCLUSIONS

The agents that appeared to be most effective on the small fires (<UL-10B) were HFC-227ea, HFC-236fa, and blends with I-bromopropane. Although the other compound, HFPE-1164X, was the least effective, it should not be ruled out since it has the advantage of being less volatile, thereby releasing less of the compound in the vapor phase, which in turn reduces the quantity breathed should the compound be discharged into an occupied crew compartment. The compound may also be more effective on Class A fires since it is a liquid and may have more of **an** ability to penetrate the fire. It also has a short atmospheric lifetime and lower GWP compared to the other compounds. When blended with 1-bromopropane, the performance of the tested compounds, including HFPE-1164X, was enhanced approximately 30%, as has been observed in previous testing conducted by CGET. There is a two-fold benefit when blending the tested compounds with 1-bromopropane: (1) increased agent performance and (2) reduced decomposition products. This is especially true for HFPE-1164X.

RECOMMENDATIONS

- Additional **HBA** blend fire suppression and decomposition testing should be conducted to evaluate further their applicability as halon replacements.
- Toxicity and exposure. studies of neat agent discharges should be performed.
- Materials compatibility and long-term storage stability studies of HBA blends should be performed.

Table 6. Decomposition Product Test Data Comparison.

UL Extinguisher Rating	FE-36™ Extinguisher, lbs	Halotron I Extinguisher, lbs	Halon 1211 Extinguisher, lbs
5B:C	6.0	5.0	2.5
10B:C	---	---	5.0
1A 10B:C	10	11	9
2A 10B:C	14	15.5	---
2A 40B:C	---	---	13
4A 80B:C	---	---	17 (20)
240B:C	---	---	150

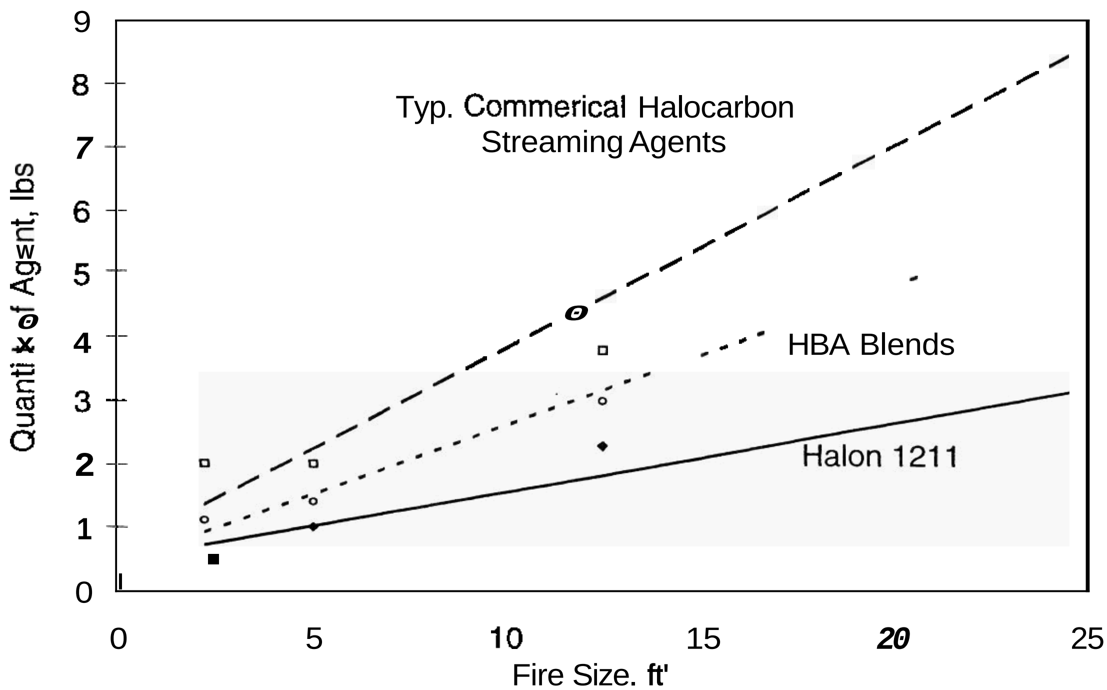

Figure 4. Quantity of Agent Required Versus Fire Size for Jet Fuel (Jet A-1).

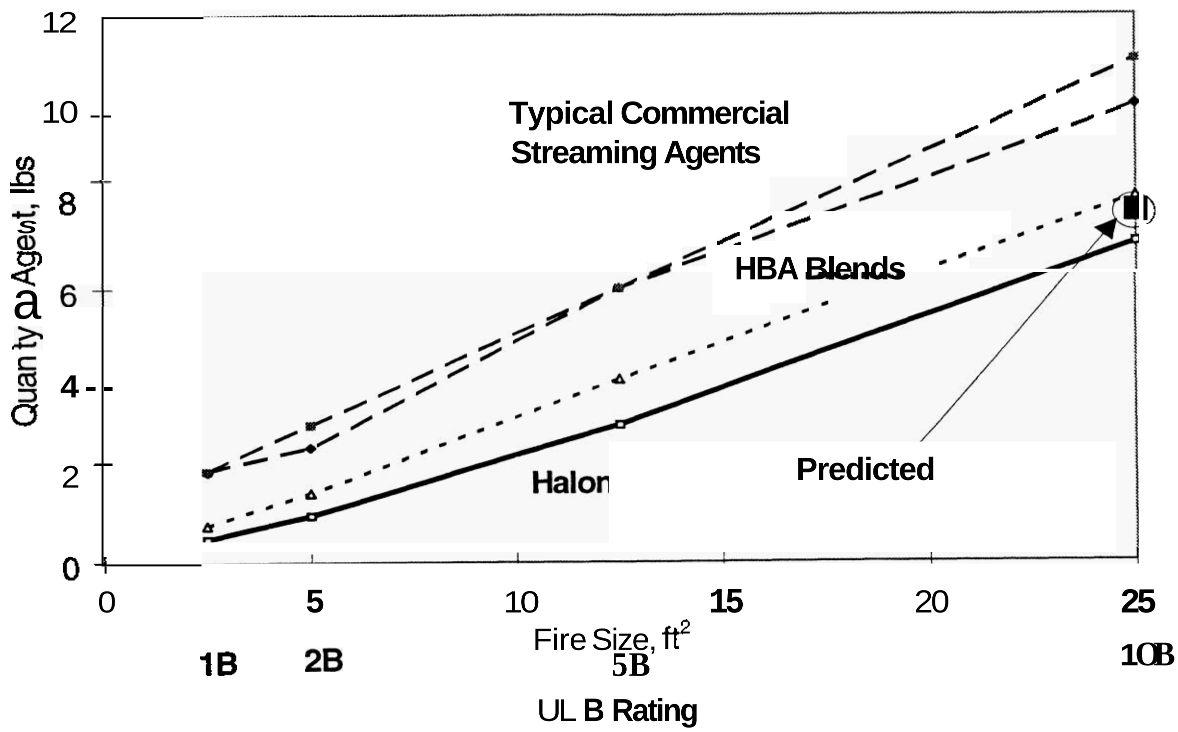

Figure 5. Quantity of Agent Required Versus Fire Size for n-Heptane.

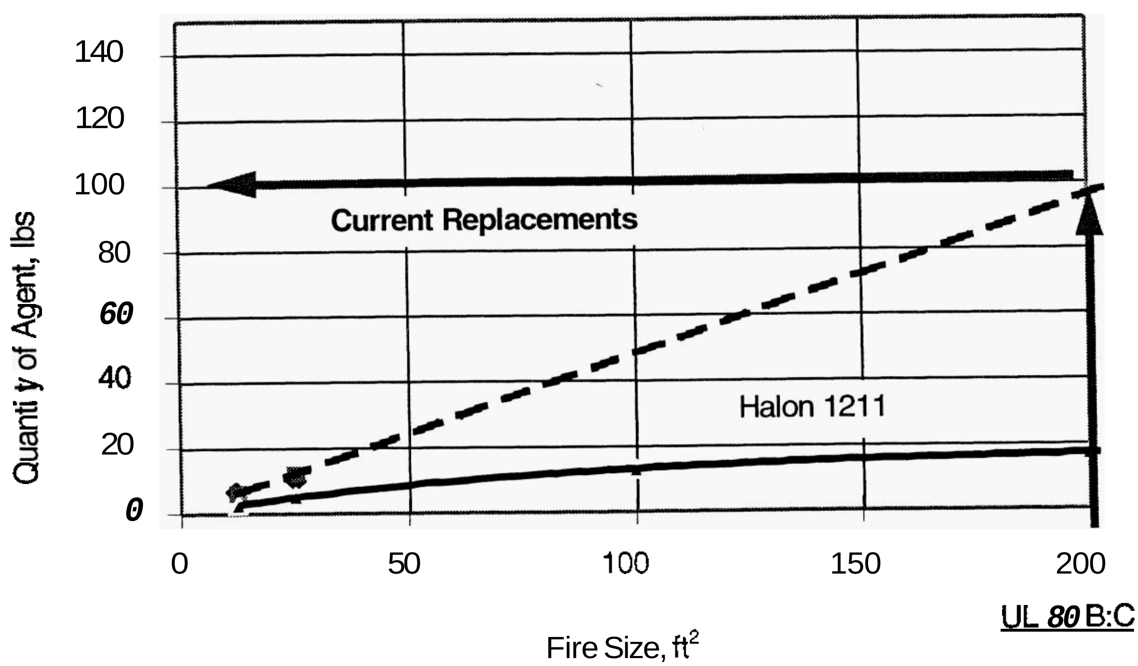

Figure 6. Quantity of Agent Required Versus Fire Size for 20-lb Halon 1211 Extinguisher (n-Heptane Fuel).

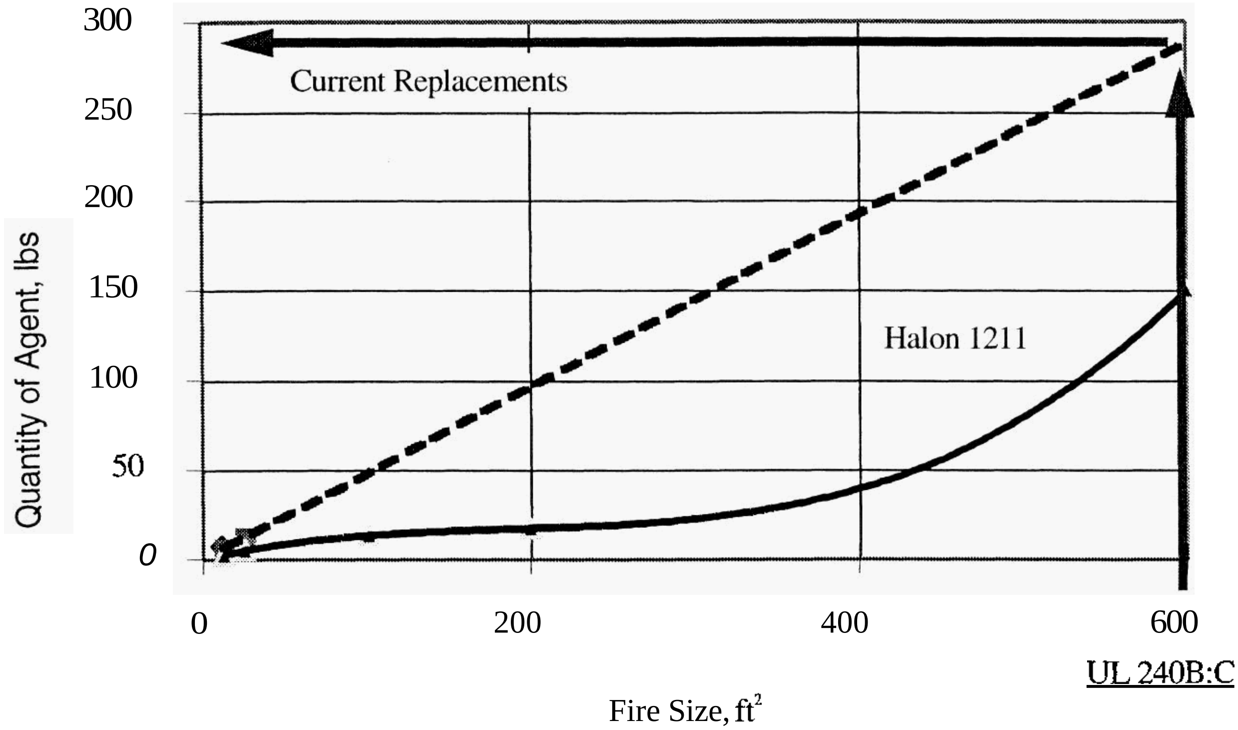

Figure 7. Quantity of Agent Required Versus Fire Size for 150-lb Halon 1211 Extinguisher (n-Heptane Fuel).

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