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ADVANCED STREAMING AGENT DEVELOPMENT, VOLUME V: LABORATORY-SCALE STREAMING TESTS

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13. ABSTRACT (Maximum 200 words)

The objective of the overall effort is to develop new chemical compounds as Halon 1211 substitutes. Four families of compounds are particularly promising—phosphorus compounds, metal compounds, silicon compounds, and tropodegradable halocarbons. This report is Volume V of a 5-volume series of reports. Volume I covers silicon compounds; Volume II, metal compounds; Volume III, phosphorus compounds; and Volume IV, tropodegradable halocarbons. The portion of the work presented in this document reviews the laboratory-scale streaming testing of compounds from various families using the Laboratory-Scale Discharge Extinguishment (LSDE) apparatus. LSDE testing was performed on phosphorus compounds, hydrofluoro-polyethers, and metal compounds. Baseline materials included Halon 1211, Halon 2402, perfluorohexane (FC-5-1-14), methyl perfluorobutylether (HFE-44901) and water. LSDE testing indicates a significant fire suppression potential for phosphorus nitrides and other phosphorus compounds. Fluoroalkoxycyclotriphos-phazenes ($P_3N_3(OR)_6$), where R is a fluoroalkoxy group, exhibit particular promise.

14. SUBJECT TERMS

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PREFACE

This report was prepared by the Advanced Protection Technologies (APT) Division, New Mexico Engineering Research Institute (NMERI), The University of New Mexico, Albuquerque, New Mexico, for the Infrastructure Technology Section of Wright Laboratory (WL/FIVCF), Tyndall Air Force Base, Florida, and Applied Research Associates (ARA), Inc., Tyndall Air Force Base, Florida, under SETA Task 3.12, Air Force Contract S-5000.31, NMERI Number 8-32540. This document is Volume V of the final report and provides a summary of the laboratory-scale streaming tests.

The Start Date for the overall Advanced Streaming Agent Program was 9 August 1995, and the End Date was 30 April 1996. The WL/FIVCF Project Officer is Major Robert A. Tetla, the ARA Project Officer is Michael A. Rochefort, and the NMERI Principal Investigator is Robert E. Tapscott.

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EXECUTIVE SUMMARY

A. OBJECTIVE

The objective of the overall effort is to develop new chemical compounds that are highly efficient fire suppressants, are environmentally and toxicologically benign, have the same performance characteristics as Halon 1211, and are compatible with existing fire extinguishing equipment and aircraft materials. The effort includes (1) synthesis of the new compounds; (2) laboratory analysis of fire suppression characteristics; (3) analysis of environmental and toxicity parameters; and (4) analysis of stability, compatibility, and manufacturability factors. The outcome of the effort is the identification of the most promising replacement candidates for follow-on medium- and large-scale testing.

This document is Volume V of the final report and provides an analysis of laboratory-scale streaming test data to determine the effectiveness of selected advanced agents as potential substitutes for replacement of Halon 1211 in streaming applications. The five volumes of the final report are listed below:

Gobeli, G. W., Tapscott, R. E., and Kaizerman, J. A., *Advanced Streaming Agent Development, Volume I: Silicon Compounds*, WL-TR-96-XX, Vol. 1 of 5, Wright Laboratories (WL/FIVCF), Tyndall Air Force Base, Florida, and Applied Research Associates, Inc., Tyndall Air Force Base, Florida, May 1996. NMERI 96/1/32540

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B. BACKGROUND

Under the Montreal Protocol, an international treaty enacted in 1987 and amended in 1990, 1992, and 1995, the production of the fire and explosion protection agents Halon 1211 (bromochlorodifluoromethane, BCFC-12B1, $CBrClF_2$) and Halon 1301 (bromotrifluoromethane, BFC-13B1, $CBrF_3$) was phased out in the United States at the end of 1993.* To date, no environmentally acceptable halon substitute, equivalent to the existing halons in toxicity, effectiveness, and dimensionality, has been identified.

Halocarbons as replacements for halons have been well studied, and it is unlikely that new, exceptionally effective, halon replacements will be identified among the standard, saturated, non-iodinated halocarbons. The hydrochlorofluorocarbons (HCFC), perfluorocarbons (PFC or FC), and hydrofluorocarbons (HFC) are all less effective than the present halons in most scenarios and usually exhibit higher toxic gas emissions (primarily, hydrogen fluoride) during fire suppression. Moreover, all of these have some adverse global environmental impact (ozone depletion, global warming, and/or long atmospheric lifetime). PFCs and HCFCs are already subject to some restrictions, and such restrictions may eventually extend to HFCs. The single partial success among halocarbon replacements is the iodides, in particular, trifluoromethyl iodide (CF_3I), which is as effective as the existing halons. However, the cardiosensitization by the fluoroalkyl iodides that have been investigated restricts their use to only certain applications. There is, therefore, an increasing incentive to look at compounds other than the usual, saturated halocarbons. These compounds include the non-halocarbon candidates, known as "advanced agents." The most promising of these are the phosphorus compounds (particularly, the phosphorus nitrides, which include phosphazenes, phosphonitriles, and phosphazanes), metal compounds, and silicon compounds. In addition, the special class of tropodegradable halocarbons includes highly promising candidates, particularly for total-flood applications.

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* See Volume IV of this report for an overview of halocarbon numbers (e.g., "BCFC-12B1" and BFC-13B1") and general information on halocarbon nomenclature.

C. SCOPE

An investigation of advanced fire suppression agents is being conducted to find a replacement for Halon 1211 used in U.S. Air Force (USAF) flightline and portable fire extinguishers. Four separate tasks are included in the present overall effort.

Task 1: Technical Review. A technical review of syntheses, characterization, properties, toxicity, and fire extinguishment data (if any) for phosphorus nitrides, metal compounds, and silicon compounds that may have utility as fire and explosion protection agents is to be conducted.

Task 2: Synthesis of New Compounds. Samples of the most promising of these materials based on expected toxicity, availability/manufacturability, and environmental characteristics are to be prepared or obtained.

Task 3: Laboratory Evaluations. Laboratory-scale evaluations of fire extinguishment by these materials are to be performed. Preliminary analyses of global environmental impact, toxicity evaluations, and manufacturing/synthesis assessment of the candidates identified for follow-on testing are to be conducted.

Task 4: Final Report. The information obtained is to be used to prepare a final report detailing the work performed, the results obtained, and conclusions reached. The report will make recommendations for continuation of large-scale testing with the most promising agents.

D. METHODOLOGY

Testing was conducted with a laboratory streaming apparatus—the Laboratory-Scale Discharge Extinguishment (LSDE) apparatus, which is designed to simulated field-scale testing. Interchangeable nozzles, varied pressures, and a metering valve were used to produce varying flow rates. The fires were extinguished as quickly as possible, and the extinguishment times and masses of agent discharged were recorded.

E. APPROACH

Since many of the agents tested are available only in small quantities, an approach has been developed allowing comparisons between streaming performance of agents on a small scale. Moreover, due to low vapor pressures, cup-burner testing is impractical with many of the Halon 1211 substitute candidates. Modifications were made to the LSDE test apparatus used in the past to obtain more reliable and repeatable results. In Phase I tests, flow rates were varied by using different nozzles and pressures. The pressures ranged from 60 psi to 120 psi. In Phase II tests, larger nozzles were used, and pressures ranged from 40 psi to 100 psi. Flow rates were varied with a metering valve. Baseline data were obtained with Halon 1211 (bromochlorodifluoromethane, $CBrClF_2$), Halon 2402 (1,2-dibromo-1,1,2,2-tetrafluoroethane, $CBrF_2CBrF_2$), water, HFE-449s1 (1-methoxynonafluorobutane, methyl perfluorobutyl ether, $CF_3CF_2CF_2CF_2OCH_3$), and FC-5-1-14 (tetradecafluorohexane, *n*-perfluorohexane, $CF_3CF_2CF_2CF_2CF_2CF_3$). In most cases, compounds were blended with carriers to determine their effectiveness in comparison to the baseline materials.

F. RESULTS

The LSDE apparatus provides data for comparing streaming performance of candidate Halon 1211 substitutes. Baseline data were obtained with Halon 1211 and Halon 2402 and with some potential carriers (or carrier models) FC-5-1-14 and HFE-449s1. Tests of fluoroalkoxycyclotriphosphazenes, $P_3N_3(OR)_6$, where R is a fluoroalkoxy group, showed enhanced extinguishing capabilities compared to the carrier when blended with HFE-449s1. An acceptable carrier has not yet been found for the metal compounds now being evaluated (primarily, iron acetylacetonates and fluoroacetylacetonates). These metal compounds are only partially soluble in FC-5-1-14 and HFE-449s1 (concentrations of less than 0.6 percent by weight). Tests with the metal solutions showed no significant extinguishment enhancement compared with neat HFE-449s1 and FC-5-1-14. This is not an unexpected result considering the low concentrations achieved.

G. CONCLUSIONS

The LSDE apparatus provides a good means for agent comparison at small scale, a particularly useful procedure when only small amounts of agent are available. Although scaleable quantitative values may not be obtainable, relative effectiveness is obtained. None of the blended agents performed as well as Halon 1211 or Halon 2402; however, a noticeable improvement in extinguishment compared to the carriers is obtained by the addition of phosphorus nitrides. It is expected that as the carriers are varied and as concentrations of the active material are increased, performance similar to the halons will be obtained. Conclusions on the streaming effectiveness of metal compounds in blends cannot be made until a carrier allowing increased solubility is identified.

H. RECOMMENDATIONS

Future work should include continued testing of advanced agents using the LSDE apparatus. This laboratory test method appears to have significant utility for screening agents being considered in streaming applications. Following screening, tests at small- and medium-scale will allow more exact comparison of agent performance against Halon 1211. It is recommended that the next stage of streaming testing utilize 0.209-m^2 (2.25-ft^2) *n*-heptane fires. Extensive baseline data have been obtained recently with Halon 1211 on such fires using a 0.567-kg (1.25-lb) extinguisher.

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LIST OF ABBREVIATIONS

FC	(per)fluorocarbons
HFC	hydrofluorocarbons
HCFC	hydrochlorofluorocarbons
HFE	hydrofluoroether
LSDE	Laboratory-Scale Discharge Extinguishment
PFC	perfluorocarbons

LIST OF UNITS AND SYMBOLS

b	intercept on straight-line plot
g	gram
kg	kilogram
lb	pound
mL	milliliter
mm	millimeter
m	meter; slope on straight-line plot
M	mass
psi	pound per square inch
Q	flow rate
s	second
t	time

SECTION I

INTRODUCTION

A. OBJECTIVE

The objective of the overall effort is to develop new chemical compounds that are highly efficient fire suppressants, are environmentally and toxicologically benign, have the same performance characteristics as Halon 1211, and are compatible with existing fire extinguishing equipment and aircraft materials. The effort includes (1) synthesis of the new compounds; (2) laboratory analysis of fire suppression characteristics; (3) analysis of environmental and toxicity parameters; and (4) analysis of stability, compatibility, and manufacturability factors. The outcome of the effort is the identification of the most promising replacement candidates for follow-on medium- and large-scale testing.

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Halocarbons as replacements for halons have been well studied, and it is unlikely that new, highly effective, halon replacements will be identified among the typical non-iodinated haloalkanes. The hydrochlorofluorocarbons (HCFC), perfluorocarbons (PFC or FC), and hydrofluorocarbons (HFC) are all less effective than the present halons in most scenarios. Moreover, all of these have some adverse global environmental impact (ozone depletion, global warming, and/or long atmospheric lifetime). PFCs and HCFCs are already subject to some restrictions, and such restrictions may eventually extend to HFCs (for an overview, see Reference 1). The single partial success among halocarbon replacements is the iodides, in particular, trifluoromethyl iodide (CF_3I), which is as effective as the existing halons. However, the toxicity of the iodides restricts their use to only certain applications. There is, therefore, an increasing incentive to look at compounds other than the typical haloalkanes. These compounds are the non-halocarbon candidates, known as “advanced agents” and a special class of halocarbons, the “tropodegradable” halocarbons.

Recent work has identified several non-halocarbon (References 2 and 3) and low-atmospheric-lifetime halocarbon (Reference 4) substitutes for halon fire extinguishants. In particular, work by the Advanced Agent Working Group, AAWG, which includes U.S. Air Force (USAF) and U.S. Army participation, has shown that the most promising of the non-halocarbon compounds are phosphorus compounds (particularly, phosphorus nitrides, which include the phosphazenes, phosphonitriles, and phosphazanes), metal compounds, and silicon compounds (Reference 5). The AAWG work (which emphasizes chemical options for total-flood Halon 1301 applications) and work by the USAF also show that tropodegradable halocarbons, which contain features that induce short atmospheric lifetimes, exhibit high promise.

C. APPROACH

Four separate tasks are included in the overall effort.

Task 1: Technical Review. A technical review of syntheses, characterization, properties, toxicity, and fire extinguishment data (if any) for phosphorus nitrides, metal complexes, and silicon compounds that may have utility as fire and explosion protection agents is to be conducted.

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SECTION II
TEST APPARATUS AND PROCEDURES

A. TEST APPARATUS

The Laboratory-Scale Discharge Extinguishment (LSDE) apparatus is a modified version of a test apparatus used previously for studies of CF_3I blends (Reference 6). Past work with the LSDE apparatus employed fixed-position cone nozzles with a circular discharge pattern. The apparatus in this configuration did not operate satisfactorily with gaseous agents due to excessive fire disturbance. More recent work with lower vapor pressure agents, such as many of those tested in this project, and with the modified apparatus described here has given more reliable results.

As part of the LSDE refinement, Unijet® flat spray nozzles were obtained from

Spraying Systems Co.
Inquiry Handling Department
North Avenue at Schmale Road
P.O. Box 7900
Wheaton, IL 60189-9860

Specifications on the nozzles from the manufacturer's literature are provided in Table 1. The procedure and the LSDE apparatus were modified to use non-fixed nozzles. These changes allow the operator to control direction and distance manually to replicate field applications with handheld nozzles. The modifications gave significantly improved measurements over the fixed position nozzles used previously, but did not provide the desired reproducibility. A new apparatus using a pivoting nozzle was, therefore, designed and used for the tests reported here. The nozzle pivots up and down around a longitudinal axis perpendicular to the nozzle to pan line (Figure 1), allowing the discharge direction to be moved up and down (vertical motion), but preventing side to side horizontal motion.

TABLE 1. UNIJET® FLAT SPRAY NOZZLES USED IN LSDE.

Nozzle no.	Spray angle, degrees (capacity, gallons per minute)[a]				Equivalent orifice diameter, in. (mm)
	20 psi	40 psi	80 psi	200 psi	
400017	21 (0.012)	40 (0.017)	54 (0.024)	61 (0.028)	0.011 (0.279)
400025	22 (0.018)	40 (0.025)	53 (0.035)	60 (0.06)	0.013 (0.330)
400033	22 (0.023)	40 (0.033)	53 (0.040)	60 (0.07)	0.015 (0.381)
400050	22 (0.035)	40 (0.050)	53 (0.07)	60 (0.11)	0.018 (0.457)
500025	29 (0.018)	50 (0.025)	64 (0.035)	71 (0.06)	0.013 (0.330)
500033	30 (0.023)	50 (0.033)	62 (0.040)	68 (0.07)	0.015 (0.381)
500050	32 (0.035)	50 (0.050)	60 (0.07)	66 (0.11)	0.018 (0.457)
500067	35 (0.050)	50 (0.067)	60 (0.090)	66 (0.015)	0.021 (0.533)

[a]Spray angles and capacities are for water.

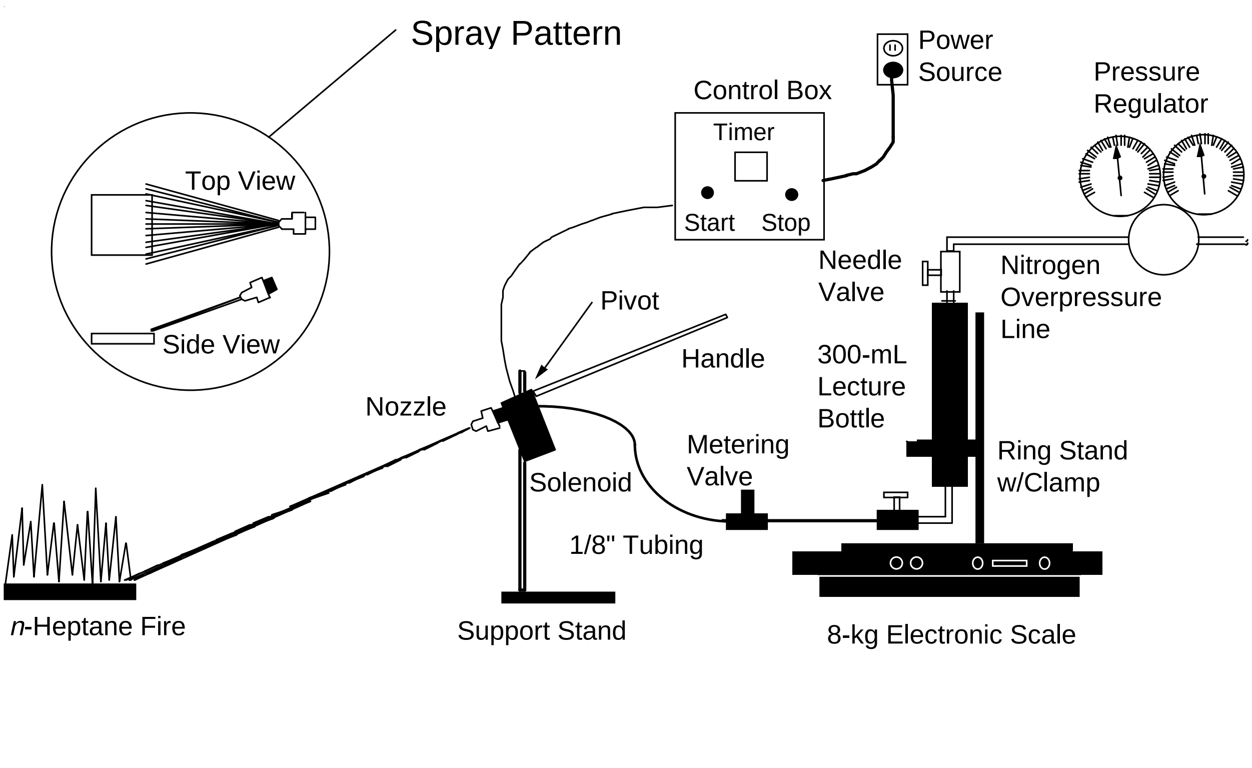

Figure 1. LSDE Apparatus.

Each nozzle produces a spray that covers slightly more than the entire width of the pan. The pivot arm is used to move the spray from the front of the pan to the back. This method of agent application reduces the amount of wasted agent.

The amount of agent discharged was determined with a Fisher Scientific digital readout laboratory scale (Model XD-8K), which has a precision of 0.01 g. Nitrogen overpressures ranging from 40 psi to 120 psi were delivered to the top of the holding cylinder to obtain desired flow rates. Agent was fed from the holding cylinder to the nozzle through a flexible nylon tube. A control box was used to activated a solenoid valve, which controlled the agent discharge. A timer, attached to the control box and having a precision of 0.01 sec, displayed the discharge time. The solenoid and timer were activated manually with toggle switches.

Two sizes of square steel fuel pans were used—8.9 by 8.9 by 2.5 cm (3.5 by 3.5 by 1 in.) with a surface area of 79.4 cm^2 (12.3 $in.^2$) and 10.2 by 10.2 by 2.5 cm (4 by 4 by 1 in.) with an area of 103.2 cm^2 (16.0 $in.^2$). In the first series of tests (Phase I), the smaller pan was used and was filled with 125 mL of *n*-heptane to produce a fuel depth of 1.58 cm (0.62 in.). In the second test series (Phase II), the larger pan was used with 90 mL of *n*-heptane floated on 90 mL of water to produce an overall depth of 1.74 cm (0.69 in.). In both cases, the pan was filled to approximately two-thirds of its capacity. A laser pointer was used to center the nozzle along a line dissecting the pan in a longitudinal direction. The fuel pan was contained within an aluminum structure closed on all four sides and open at the top (Figure 2). A door was located on one side for access into the box. The entire box was located within a laboratory hood to prevent the release of combustion products into the laboratory.

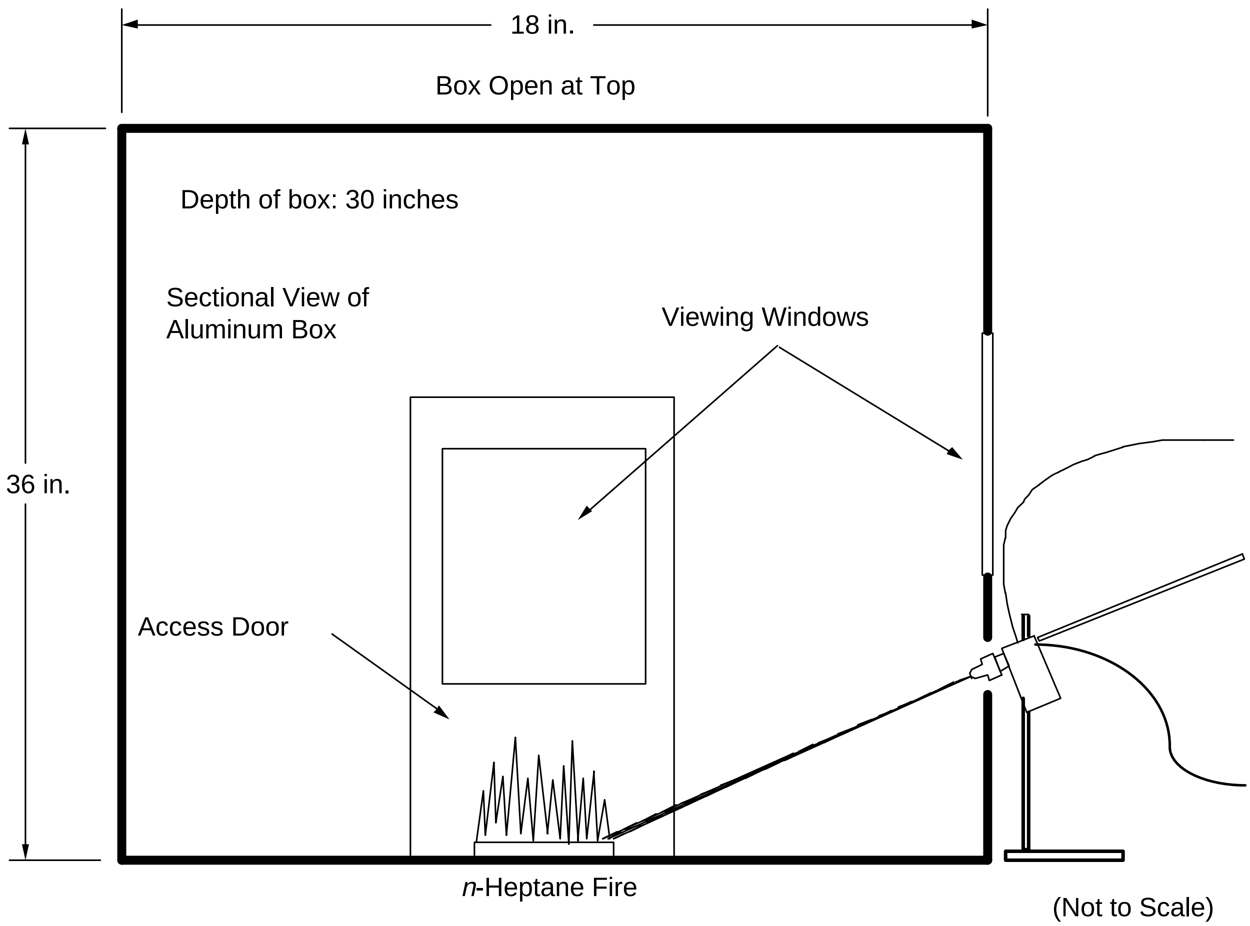

Figure 2. Test Chamber.

B. TEST METHOD

Tests on a small scale require that every effort be made to eliminate or minimize variations in those factors that can affect test results. The more significant variables require tighter control to obtain reliable, repeatable results. Prior to testing, pertinent variables were identified and measures were taken to mitigate their effects, as shown in Table 2.

TABLE 2. SOME TEST VARIABLES AND ACTIONS TAKEN TO MINIMIZE VARIATION.

Variable	Action taken
Agent application pattern	The same type of nozzles were used for all the tests. The nozzles produced a flat spray, creating a fan angle of approximately 55 deg. The nozzle was fixed to pivot in only one direction, thereby eliminating side to side motion.
Fire geometry	Square pans were used for all the tests. A square pan was chosen so that some comparisons can eventually be made with larger scale field data previously obtained with square pans.
Fuel depth	Care was taken to insure the fuel depth was measured prior to each test.
Contaminated fuel	The fuel (*n*-heptane) was replaced after every test.
Pan temperature	The pan was placed in a water bath after each test to cool it prior to the next test.
Preburn time	Preburn times were kept constant within each test series.
Air flow	All the tests were performed in an enclosed box placed inside a laboratory hood.
Ambient temperature	The laboratory was maintained at a near constant temperature of approximately 20 °C (68 °F).
Application technique	The same operator performed all tests.

Testing was conducted with the LSDE apparatus as described in the previous section. The procedure used for each test was to (1) apply the agent near the front of the pan, (2) drive the fire to the back of the pan, and (3) extinguish the fire as quickly as possible. A test was considered valid only if the operator believed that the fire was extinguished optimally. Frequently, the initial first few tests with a new nozzle and/or agent were considered invalid since the operator did not have a feel for the sweep rate from the front to the back of the pan necessary for optimized extinguishment. Examples of non-optimized extinguishment occurred when a sweep rate was too slow or when the agent stream was moved too quickly across the pan causing too much time to be required to gain control of the flame front. In both cases, agent was wasted. A technique was developed to minimize the agent needed for extinguishment and to give good repeatability.

SECTION III
COMPOUNDS TESTED

Materials tested with the LSDE apparatus included phosphorus compounds, metal complexes, a tropodegradable compound, and the baseline agents Halon 1211 (bromochlorodifluoromethane, $CBrClF_2$), Halon 2402 (1,2-dibromo-1,1,2,2-tetrafluoroethane, $CBrF_2CBrF_2$), water, HFE-449s1 (1-methoxynonafluorobutane, methyl perfluorobutyl ether, $CF_3CF_2CF_2CF_2OCH_3$), and FC-5-1-14 (tetradecafluorohexane, *n*-perfluorohexane, $CF_3CF_2CF_2CF_2CF_2CF_3$). Additionally, two experimental hydrofluoropolyethers (H-Galden 1183x and H-Galden 1164x), manufactured by Ausimont, Montedison Specialty Chemicals, Milan, Italy, were also tested. Table 3 lists all the agents tested.

TABLE 3. AGENTS TESTED WITH THE LSDE APPARATUS.

Common name	Chemical name (formula)
Halon 1211	bromochlorodifluoromethane, $CBrClF_2$
Halon 2402	1,2-dibromo-1,1,2,2-tetrafluoroethane, $CBrF_2CBrF_2$
water	H_2O
n-propyl bromide	1-bromopropane, $CH_3CH_2CH_2Br$
FC-5-1-14	tetradecafluorohexane (*n*-perfluorohexane), $CF_3CF_2CF_2CF_2CF_2CF_3$
HFE-449s1	1-methoxynonafluorobutane (methyl perfluorobutyl ether), $CF_3CF_2CF_2CF_2OCH_3$
Hexamethoxycyclotriphosphazene	$P_3N_3(OCH_3)_6$
Fluoroalkoxyphosphazenes	See Table 4
H-Galden 1183x	hydrofluoropolyether experimental product
H-Galden 1164x	hydrofluoropolyether experimental product
$Fe(hfac)_3$	tris(1,1,1,5,5,5-hexafluoro-2,4-pentanedionato(-1))-Iron(III) (iron hexafluoroacetylacetonate), $Fe(CF_3C(O)CHC(O)CF_3)_3$
$Fe(acac)_3$	tris(2,4-pentanedionato(-1))Iron(III) (iron acetylacetonate), $Fe(CH_3C(O)CHC(O)CH_3)_3$

The four fluoroalkoxy-substituted phosphazenes listed in Table 4 were tested on the LSDE apparatus during this project.* The formulas are given as $P_3N_3(OR)_3(OR')_3$. Compounds I and II may be a mixture of isomers. These phosphazene compounds were blended with HFE-449s1 as a carrier since the neat fluoroalkoxyphosphazenes were too viscous or very limited amounts were available. Attempts to blend the fluoroalkoxyphosphazenes with FC-5-1-14 failed due to low solubility.

TABLE 4. FLUOROALKOXYPHOSPHAZENES TESTED.

Compound	Designation	R	R'
I	C2C4	CH_2CF_3	$CH_2(CF_2)_2CF_3$
II	C2C5	CH_2CF_3	$CH_2(CF_2)_3CHF_2$
III	C5C5	$CH_2(CF_2)_3CHF_2$	$CH_2(CF_2)_3CHF_2$
IV	C2C2	CH_2CF_3	CH_2CF_3

*See Volume III of this report for information on the physical properties of these and other compounds tested.

SECTION IV
TEST RESULTS AND ANALYSIS

A. OVERVIEW

Laboratory tests with the LSDE apparatus provide a reasonable method for comparing streaming performance of candidate agents. Extinguishment time and quantity of agent required for extinguishment provide the primary test data. From these data, flow rates may be determined. The following discussion for Halon 1211, a baseline agent, provides an insight of the analysis of test data.

Data obtained give the minimum extinguishment time and minimum quantity of agent used for each test. For example, consider a test with Halon 1211 where the extinguishment time is 2.4 sec and the quantity of agent discharged is 4.8 g. This is ideally the quickest extinguishment time that could be achieved at this flow rate with the given test parameters. Obviously, the same fire could have been extinguished by moving the agent across the pan at a slower rate, thus resulting in a longer extinguishment time with a discharge of more agent; however, this would not provide pertinent information. The minimum extinguishment time and the corresponding quantity of agent discharged for given test conditions are the important data.

Use of various nozzles, pressures, and metering valve settings produced the data presented in Figure 3, which shows the quantity of agent required to extinguish the fire versus the extinguishment time for Halon 1211. The data in this figure were obtained with three sizes of nozzles—the 40017 nozzle, used with pressures of 40 psi and 60 psi; the 500025 nozzle, used with a pressure of 100 psi; and the 500067 nozzle, used with a pressure of 40 psi. A linear regression on the data gives a straight line for the quantity (mass) as a function of time (Equation 1 and Figure 3).

$$M = 1.13t + 2.19 \qquad [1]$$

where M = mass of agent discharged (g) and t = extinguishment (discharge) time.

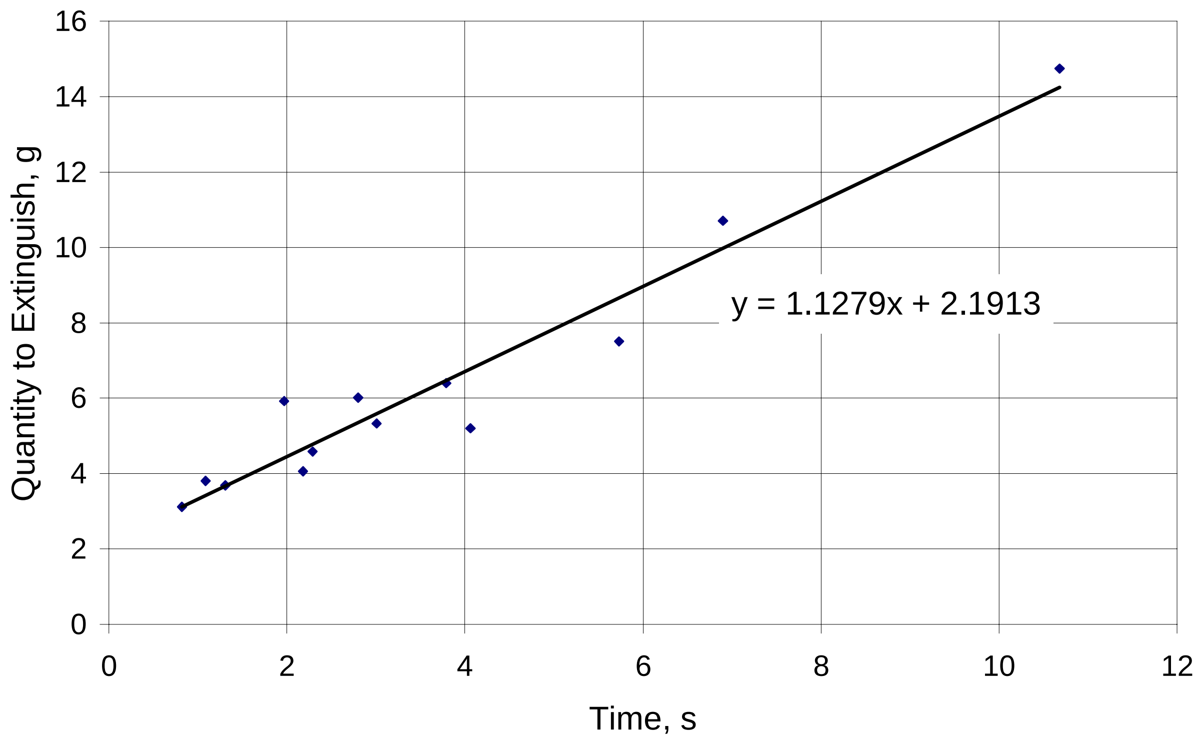

Figure 3. Quantity to Extinguish Versus Extinguishment Time for Halon 1211.

The flow rates can be plotted as a function of extinguishment time. The flow rate is determined by dividing the quantity to extinguish by the corresponding extinguishment time for each individual test (Figure 4). The data can be curve-fitted by dividing Equation 1 by time giving Equation 2,

$$Q = 1.13 + 2.19/t \qquad [2]$$

where Q = flow rate (g/s) and t = extinguishment (discharge) time (s).

Repeating this for the data obtained with FC-5-1-14 allows a comparison with Halon 1211, as shown in Figure 5. The curves obtained show that Halon 1211 is more effective than FC-5-1-14 since it has a lower flow rate versus extinguishment time curve, as would be expected.

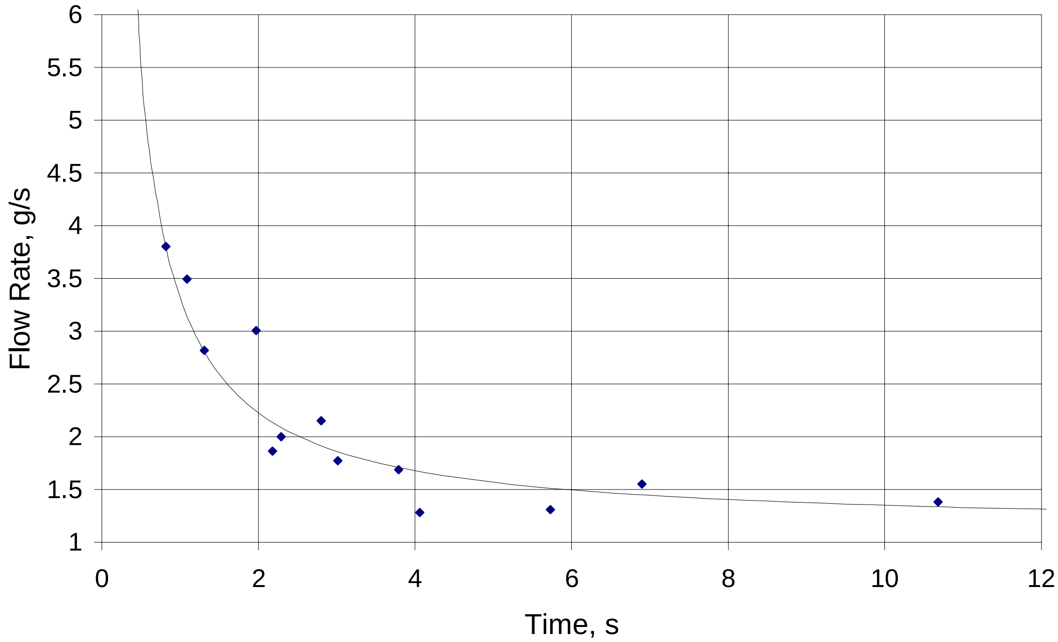

Figure 4. Flow Rate Versus Extinguishment Time for Halon 1211.

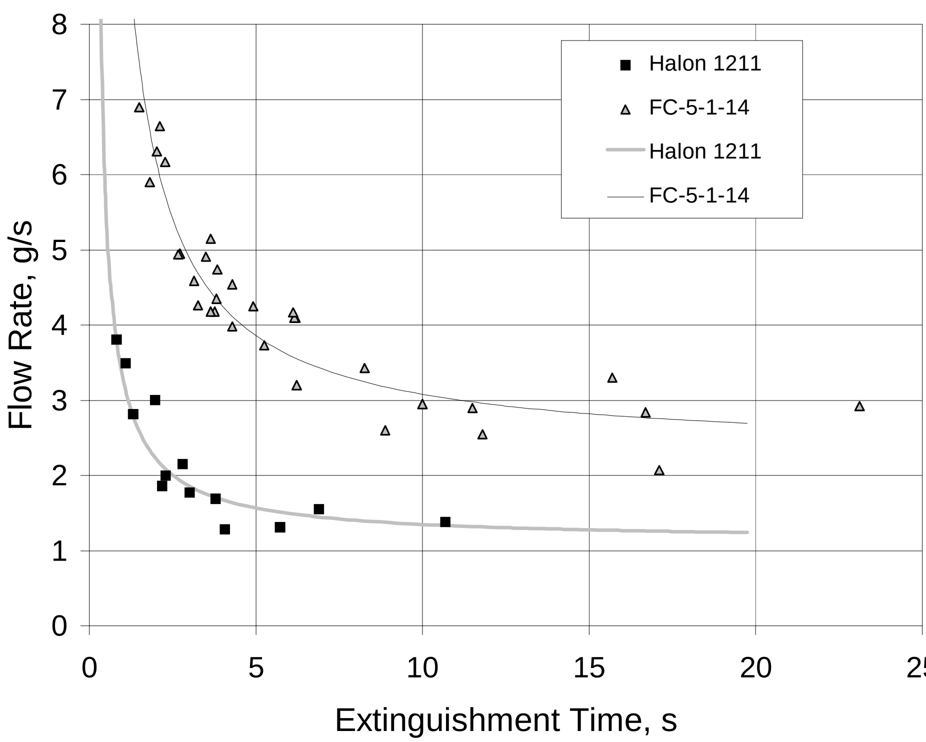

Figure 5. Comparison of Halon 1211 and FC-5-1-14 for a 103.2-cm^2 (16.0-$in.^2$) Fire.

The data can also be represented by plotting the quantity to extinguish versus flow rate. Such a plot is shown in Figure 6 for Halon 1211. A curve can also be fit to these data. To do this for Halon 1211, one solves Equation 2 for extinguishment time to obtain Equation 3.

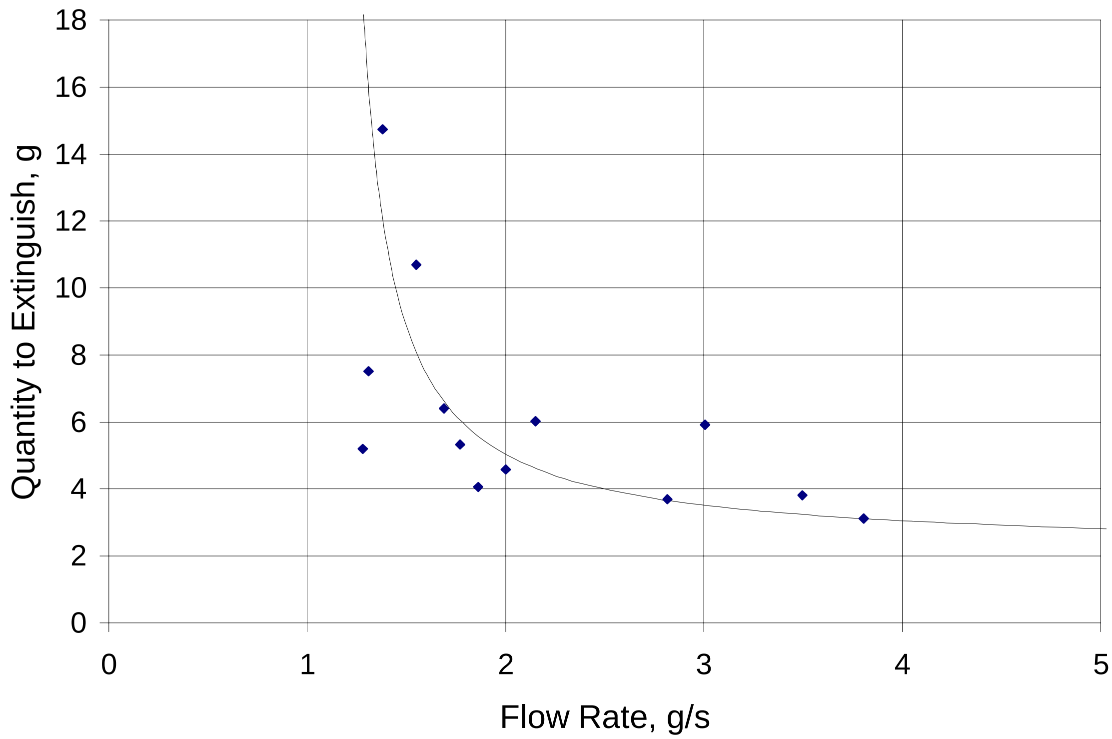

Figure 6. Quantity to Extinguish Versus Flow Rate for Halon 1211.

$$t = 2.19/(Q-1.13) \quad [3]$$

Substitution of Equation 3 into Equation 1 gives Equation 4.

$$M = 1.13 \times [2.19/(Q-1.13)] + 2.19 \quad [4]$$

Simplifying Equation 4, one obtains Equation 5.

$$M = (2.19Q)/(Q-1.13) \quad [5]$$

In Equation 5, 1.13 is the slope in Equation 1, and 2.19 is the y-axis intercept. Therefore, Equation 5 can be written in more general terms as shown in Equation 6.

$$M = (bQ)/(Q-m) \quad [6]$$

where m is the slope and b is the intercept of the quantity versus extinguishment time curve.

Applying Equation 6 to the data for Halon 1211, one obtains the curve shown in Figure 6. This figure allows an estimation of the quantity of agent required to extinguish a 103.2-cm^2 (16.0-$in.^2$) fire at a given flow rate. Note that this only applies to the 103.2-cm^2 (16.0-$in.^2$) fire tested in the lab. Comparisons between agents using this type of chart require that the fire sizes be the same.

Interpretation of the data often presents problems, particularly if the whole picture is not considered. The question is—How can one tell if one agent performs better than another? Does one look strictly at the amount of agent required to extinguish a fire or does one consider also the flow rate? These alternatives must be considered carefully. Suppose, for example, that an agent is being considered for a portable extinguisher. In such instances, the flow rate is very important. The extinguisher may be required to discharge agent for a minimum of 10 sec. If the agent has a high minimum flow rate (which will be discussed shortly), it may never meet this requirement. With this in mind, the importance of the different factors will be discussed, as will a method for comparing the effectiveness of the agents.

The minimum flow rate is the constant rate of discharge of an agent that is minimally capable of extinguishing a particular fire size. Equation 2, discussed earlier, represents the quantity of Halon 1211 required to extinguish a 103.2-cm^2 (16.0-$in.^2$) fire versus the corresponding extinguishment time. Equation 2 shows that as the time increases, the flow rate asymptotically approaches 1.86 g/s. Theoretically, the flow rate will never reach 1.86 g/s; however, this value is a good estimate for the minimum flow rate required.

The importance of the minimum flow rate cannot be overemphasized. At high flow rates, agents may appear to have similar extinguishing capabilities (Reference 7). Suppose, for example, that FC-5-1-14 is compared to Halon 1211 in a field test. Each is loaded into identical extinguishers, and appropriate nozzles are used such that relatively high but identical flow rates are used from the extinguishers. The agents are applied to identical fires, and each extinguishes their respective fires rapidly. In both cases, similar amounts of agent are used to extinguish each fire. This could lead one to believe that FC-5-1-14 is as efficient as Halon 1211, which is obviously incorrect. If one extrapolates the flow rate curves for Halon 1211 and FC-5-1-14 from the LSDE tests as shown in Figure 7, one can see how such an assumption could be made. With a relatively high flow rate of approximately 25 g/s, the extinguishment time for both agents would be approximately 0.5 sec, and the mass of agent required for extinguishment for both agents would be approximately the same.

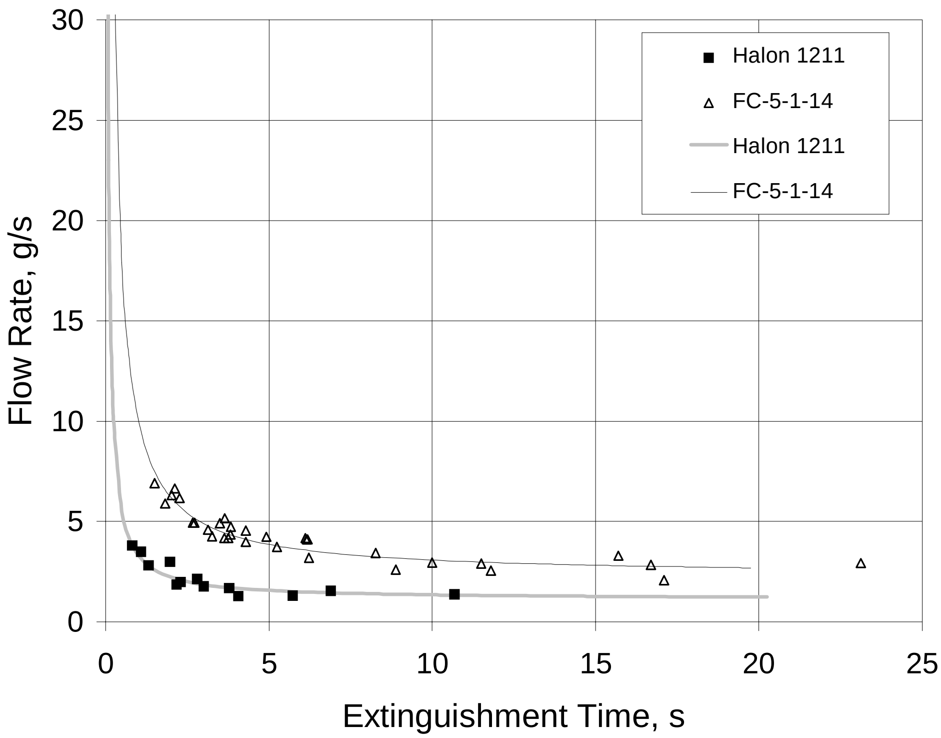

Figure 7. Extrapolation of Flow Rate Curves for Halon 1211 and FC-5-1-14.

With lower flow rates, such as 4 g/s, a significant difference in the performance of the agents begins to appear. Moving down the flow rate curves to approximately 2.5 g/s gives a point where the FC-5-1-14 is barely capable of extinguishing the fire, while Halon 1211 still achieves extinguishment readily. As a result, much more FC-5-1-14 is required to extinguish the same size fire since the FC-5-1-14 is being discharged closer to its minimum flow rate. At still lower flow rates, FC-5-1-14 becomes incapable of extinguishing the fire, while Halon 1211 remains effective.

The above example shows the importance of considering flow rate. Agents can appear to perform similarly if the flow rate is high enough. In such cases, agent is being wasted and no real comparisons can be made. The overall goal is to find an agent that gives a flow rate versus extinguishment time curve close to that of Halon 1211.

Laboratory tests were conducted in two series. In the first of these series a 79.0-cm^2 (12.3-$in.^2$) fire was used, using both the smaller and larger nozzles. In the second test series, it was desired to obtain a more definable distinction between agents, and to minimize the effects of the finer mists created by the smaller nozzle. Therefore, both a larger fire (103.2 cm^2 [16.0 $in.^2$]) and larger nozzles were used.

B. PHASE I TESTS

For Phase I tests, the data are presented into three groups: (1) Data obtained with the smallest nozzle (400017), (2) data obtained with the remaining nozzles (400025, 400033, 400050, designated as “Other Nozzles” in figure legends), and (3) data obtained with all nozzles (400017, 400025, 400033, 400050, designated as “All Nozzles” in figure legends). Water and 1-bromopropane data appear to be independent of nozzle size and are, therefore, grouped into “All Nozzles.” For other agents, such as FC-5-1-14, there is a noticeable difference between the 400017 nozzles and the remaining nozzles. Agents exhibiting this behavior were grouped into the “Nozzle 400017” and the “Other Nozzles” categories as discussed above.

Application of the equations developed above to the data, yielded flow rate versus extinguishment curves (Figure 8 and Figure 9). Individual data plots for individual agents are presented in Appendix A. These include quantity to extinguish versus extinguishment time, flow rate versus extinguishment time, and quantity to extinguish versus flow rate. Note that these tests were all performed with the smaller 8.9 by 8.9 by 2.5-cm (3.5 by 3.5 by 1-in.) pan, *n*-heptane fuel (125 mL), and a 60-sec preburn. Figure 8 shows results with the smallest nozzle

(400017). Water and 1-bromopropane are shown with all nozzles since nozzle sizes does not appear to have a significant effect on the results (Appendix A).

With the smallest nozzle, FC-5-1-14 appears to be the most effective agent since it has the lowest flow rate versus extinguishment time curve (Figure 8). This is almost certainly due to agent misting when the smallest nozzle is used with this agent. It has been shown that misting of perfluorocarbons such as FC-5-1-14 enhances their extinguishment ability (References 8 and 9). The Phase II tests, which use larger nozzles, were run, in part, to avoid the misting problem.

Water appears to be the least effective with the highest curve. Due to flow limitations, full curves could not be obtained with the small 400017 nozzle. With the larger nozzles (Figure 9) water showed an improved extinguishment, as did 1-bromopropane. FC-5-1-14 performed similarly to H-Galden 1164x, and all the agents performed superior to HFE-449s1. Similar trends were seen with the H-Galden 1164x, FC-5-1-14, and HFE-449s1 in the Phase II tests.

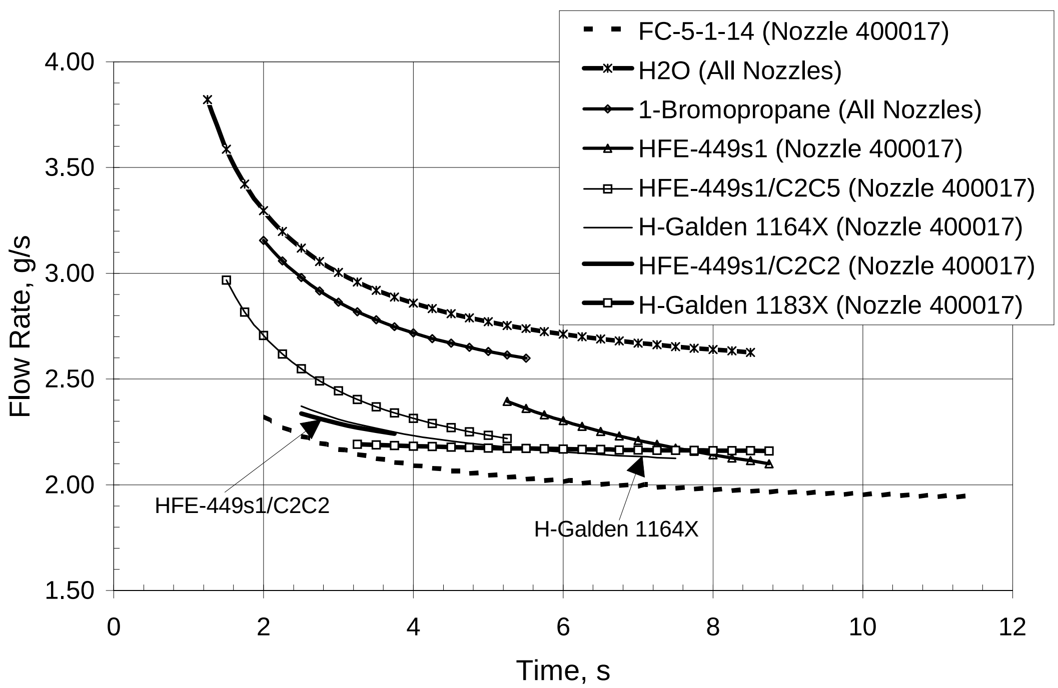

Figure 8. Flow Rate Versus Extinguishment Time for First Test Series Utilizing Smaller Nozzle.

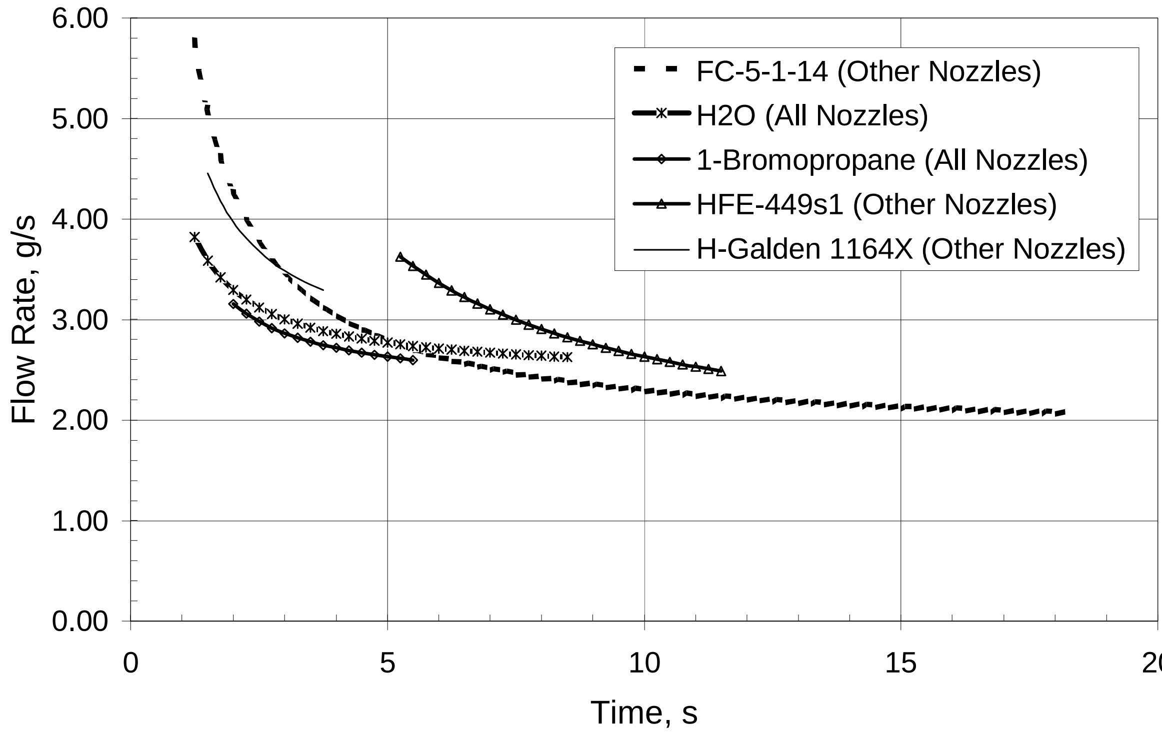

Figure 9. Flow Rate Versus Extinguishment Time for First Test Series Utilizing Larger Nozzles.

Results for the HFE-449s1/fluoroalkoxy phosphazene blends are of particular interest. The blend of 75 weight percent HFE-449s1 and 25 weight percent $P_3N_3(OCH_2CF_3)_3(OCH_2(CF_2)_3CHF_2)_3$ (designated "C2C5") showed enhanced extinguishment compared to the HFE-449s1 carrier alone. This can be seen from an examination of the flow rates between 2.0 g/s and 2.5 g/s in Figure 8. For example, at a flow rate of 2.5 g/s, the extinguishment time for HFE-449s1 is approximately 5 sec. This equates to 12.5 g of agent required for extinguishment. At the same flow rate of 2.5 g/s, the extinguishment time for the HFE-449s1/C2C5 blend is approximately 2.5 sec, equating to 6.3 g of agent for extinguishment. A plot of the quantity to extinguish versus flow rate shows this comparison directly (Figure 10). In the entire flow rate range of 2.0 g/s to 2.5 g/s, the quantity to extinguish is considerably lower for the HFE-449s1/C2C5 blend.

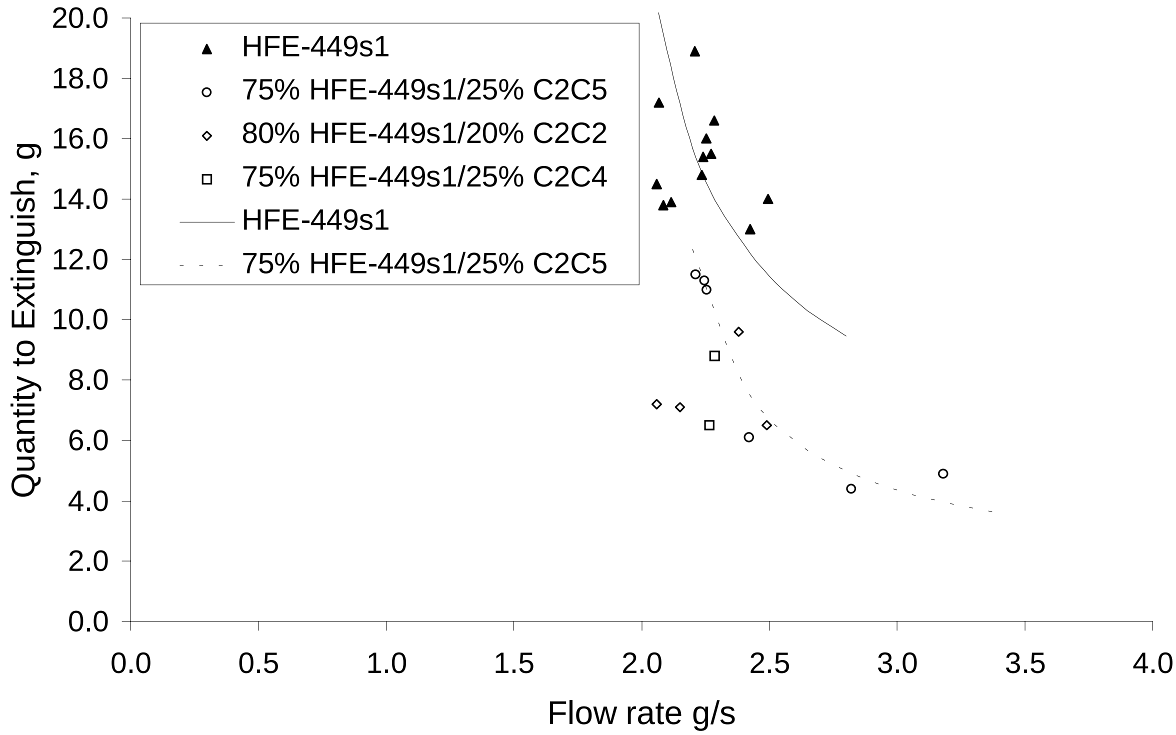

Figure 10. Quantity to Extinguish Versus Flow Rate for the HFE-449s1/Fluoroalkoxy Phosphazene Blends.

Similar results are seen with a 25 percent $P_3N_3(OCH_2CF_3)_3(OCH_2(CF_2)_2CF_3)_3$ (C2C4) blend. Data points from these tests fall close to the HFE-449s1/C2C5 curve in Figure 10. A blend of 80 weight percent HFE-449s1 and 20 weight percent $P_3N_3(OCH_2CF_3)_6$ (C2C2) performs similarly to the C2C5 blend (Figure 10). Each of the fluoroalkoxyphosphazenes, when blended with HFE-449s1, shows a significant improvement over neat HFE-449s1 in the flow rate range of 2.0 g/s to 2.5 g/s. Note that all of these comparisons are on a weight basis, not a volume basis.

During this phase of testing a new experimental hydrofluoropolyether, H-Galden 1164x, was tested. This material appears to have extinguishing capabilities similar to those of FC-5-1-14. Figure 8 shows that both compounds have similar flow rate versus extinguishment time curves in the range of 3.4 g/s to 4.4 g/s.

Limited testing of blends of $P_3N_3(OCH_2(CF_2)_3CHF_2)_6$ (C5C5) with the H-Galden 1183x gave inconsistent results. At a flow rate of 2 g/s, the fire was extinguished in 3.9 sec in one test, and 12.9 sec in another. With the limited amount of compound available, a reliable evaluation of effectiveness of a phosphazene blend with H-Galden 1183x could not be determined. It was noticed that a crackling sound occurred when the blend was sprayed onto the fire. The sound was similar to that heard during tests with water. The sound was not evident during tests with the other fluoroalkoxyphosphazene blends.

A 10 percent (by weight) aqueous solution of hexamethoxycyclotriphosphazene [$P_3N_3(OCH_3)_6$] did not effect extinguishment. The compound appeared to be flammable and was unable to push the fire off the front edge of the pan. Flammability is not unexpected with a nonfluorinated alkoxyphosphazene.

Although the water was the least effective of the agents tested with the smallest nozzle, it showed significant improvement with the 400025 and 400033 nozzles. With the larger 400050 nozzle, extinguishment was very difficult to attain with water (Appendix A), even at higher flow rates. This was also noticed in Phase II testing, where extinguishment did not occur with the larger 500050 and 500067 nozzles with the lower pressures.

Although labeled as flammable, the 1-bromopropane was effective in extinguishing fires. Figure 9 shows that this compound has the lowest flow rate versus extinguishment time curve. Visually, it appeared to act similar to Halon 1211. It was very effective in pushing the fire to the back of the pan and off the back edge. However, 1-bromopropane created a small fireball behind the pan, requiring slightly more agent for complete extinguishment. Since the nozzle size did not significantly effect test results, as it did with other compounds, 1-bromopropane did not appear to be as effective when compared to the other compounds based on the 400017 nozzle (Figure 8). The same holds true for water.

C. PHASE II TESTS

Phase II tests were performed with the larger 10.2 by 10.2 by 2.5-cm (4 by 4 by 1-in.) pan with 90 mL of *n*-heptane floating on 90 mL of water and using a 90-sec preburn. The purpose of the Phase II tests was to obtain data allowing fuller flow rate versus extinguishment time curves and to reduce the effects of the small particle mists created by the smallest nozzle (400017) combined with the high pressure (120 psi). Nozzles larger than the 400017 nozzle were used (with the exception of one test on Halon 1211, which requires a low flow rate due to its high effectiveness), and flow rates were controlled with a metering valve. Halon 2402 was included to obtain an additional baseline agent for comparison. Another hydrofluoropolyether, H-Galden 1183x, was also tested. Results from the Phase II testing are shown in Figure 11. Individual data plots for each compound are presented in Appendix B.

Figure 11 clearly shows that Halon 1211 and Halon 2402 outperformed all other compounds. Their flow rate versus extinguishment time curves are lower than all curves for the other compounds. At a flow rate of 1.5 g/s, both Halon 1211 and Halon 2402 extinguished the laboratory fire in approximately 4 sec with approximately 6 g of agent. None of the other compounds was able to extinguish the fire at such a low flow rate. For an extinguishment time of 4 sec, a flow rate of approximately 4.2 g/s is required for FC-5-1-14, H-Galden 1164x, and H-Galden 1183x. This corresponds to an extinguishment quantity of 16.8 g for these agents. The HFE-449s1 requires an even higher flow rate of 6.5 g/s and requires 26 g of agent. Overall, the performance of both of the H-Galden compounds is similar to that of FC-5-1-14, and the performance of both the H-Galden compounds and FC-5-1-14 is superior to that of HFE-449s1.

The effectiveness of a solution containing 75 weight percent HFE-449s1 and 25 weight percent C_2C_5 is inconclusive. Of the four tests performed, two showed significant enhanced performance compared to the carrier alone, and two did not. For example, at a flow rate of 3.6 g/s the extinguishment time was 16.4 sec, which is no enhancement compared to HFE-449s1 by itself. At a flow rate of 4.4 g/s the extinguishment time was 4.2 sec, a significant improvement over the extinguishment time of 8 sec for neat HFE-449s1. At a flow rate of 5 g/s, one test showed significant enhancement and one did not. The corresponding extinguishment times were 2.4 sec and 9.6 sec, compared to 7 sec for neat HFE-449s1. Insufficient C_2C_5 was available to make a further evaluation.

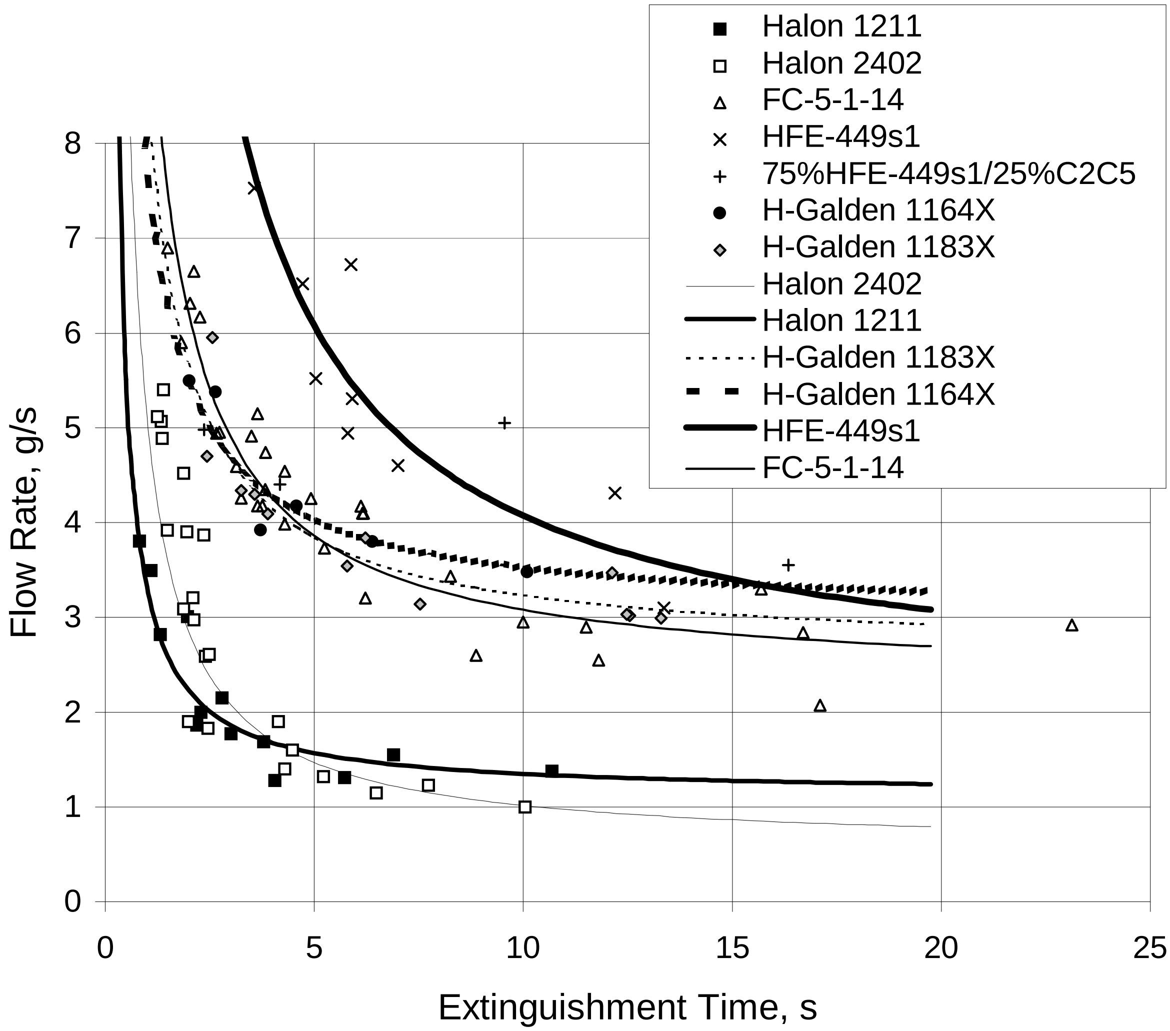

Figure 11. Flow Rate Versus Extinguishment Time for Phase II Tests.

Limited testing was performed with the metal compounds, which have a very limited solubility in FC-5-1-14 and HFE-449s1. One test on a blend of 99.8 weight percent HFE-449s1 and 0.2 weight percent $Fe(acac)_3$, which is insoluble in FC-5-1-14 and only partially soluble in HFE-449s1, showed no improved performance over pure HFE-449s1. At a flow rate of 4.8 g/s, the extinguishment time was 9.8 sec. This falls close to the HFE-449s1 curve shown in Figure 11.

$Fe(hfac)_3$ is only partially soluble in FC-5-1-14 and was not tested with HFE-449s1 due to its unavailability at the time of the testing. One test on a 99.6 weight percent FC-5-1-14 and

0.4 weight percent $Fe(hfac)_3$ solution showed no improved performance over neat FC-5-1-14. At a flow rate of 4.3 g/s the extinguishment time was 6.1 sec.

Since the iron compounds are soluble in methanol, a 75 weight percent methanol and 25 weight percent $Fe(hfac)_3$ solution was tested. This was quite ineffective; the fire could not be driven off the front of the pan, and it appeared that the agent blend made the fire larger. This is not unexpected since methanol is flammable.

Overall, the effectiveness of the metal compounds could not be determined with the limited tests that were performed. Different carriers are needed to increase the solubility of the metal compounds. With the very low solubilities of the metal compounds in the carriers used here, the absence of a definite extinguishment enhancement is not surprising.

SECTION V
CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

The modified LSDE apparatus provides valuable information for comparing agents. This is particularly true for the Phase II tests, in which distinct differences can be seen in agent performances. Some of the PN compounds were not tested in Phase II due to their limited availability. However, Phase I tests have shown that the fluoroalkoxy phosphazene compounds increase the performance of HFE-449s1. Although scaleable quantitative values have not been obtained, the relative effectiveness can be seen. None of the agents performed as well as Halon 1211 and Halon 2402, but there was a noticeable improvement in the extinguishment of carriers when phosphorus nitrides were added. Conclusions about the effectiveness of the metal compounds cannot be made until a carrier allowing increased concentrations of the metal compounds is identified.

B. RECOMMENDATIONS

Future work should include continued testing of advanced agents using the LSDE apparatus. This laboratory test method appears to have significant utility for screening agents being considered in streaming applications. Following screening, tests at small- and medium-scale will allow more exact comparison of agent performance against Halon 1211. It is recommended that the next stage of streaming testing utilize 0.209-m^2 (2.25-ft^2) *n*-heptane fires. Extensive baseline data have been obtained recently with Halon 1211 on such fires using a 0.567-kg (1.25-lb) extinguisher.

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APPENDIX A

TEST RESULTS FROM PHASE I TESTS

APPENDIX B

TEST RESULTS FROM PHASE II TESTS