FK-5-1-12 PERFORMANCE CHARACTERISTICS: RECENT DEVELOPMENTS

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ABSTRACT

This paper presents the recent developments that have been achieved through testing on FK-5-1-12, commercially known as 3M™ Novec™ 1230 Fire Protection Fluid. FK-5-1-12 has undergone multiple fire performance testing at 3M including inerting, cup burner, large scale and small scale testing. The data collected for the cup burner test for FK-5-1-12 was obtained for multiple class B fuels using a 3M cup burner apparatus. The DIS 14520-1: September 2003 Annex B was used for determining the flame extinguishing concentration.

Inerting tests conducted for FK-5-1-12 using propane and methane fuel are reported. The testing was conducted in accordance with the procedures outlined in ISO 14520:2000 Annex D to determine the inerting concentrations of FK-5-1-12 for various methane and propane air mixtures. The minimum demonstrated FK-5-1-12 inerting concentration for methane and propane is in the range of 8%-9% v/v.

Large-scale FK-5-1-12 fire performance testing is reported, conducted in a 100 m^3 test enclosure and an intermediate-scale, 1.28 m^3 enclosure. Thermocouples located directly in the discharge nozzle spray and other locations within the protected space were used to monitor the temperature. Discharge and post-discharge room temperature variations were recorded with respect to proximity of the discharge nozzle and location within the enclosure.

Alternative non-conventional system delivery methods are explored taking advantage of this high boiling, low vapor pressure fluid. Data now exist to validate agent delivery to the protected space in new and useful ways.

Introduction

FK-5-1-12 is an environmentally sustainable clean extinguishing agent that has undergone considerable testing to determine its fire performance characteristics as a clean extinguishing agent. Small, intermediate and large-scale testing recently conducted has quantified new information of the unique performance characteristics of FK-5-1-12. Standards like NFPA 2001 and ISO 14520 that recognize and now include FK-5-1-12 include protocols for small-scale cup burner and inerting tests, which are well recognized for determining fire performance characteristics of extinguishing agents. Intermediate and large-scale testing was conducted to collect a temperature profile in order to gain insight as to how the temperature of a room is affected by the discharge of FK-5-1-12, and to examine alternative methods for agent delivery conducive to the high-boiling, low vapor pressure FK-5-1-12.

Cup Burner Test Procedure

For the determination of the cup burner extinguishing concentrations the ISO DIS 14520 standard was used[1]. Several modifications were necessary to introduce a liquid agent into the standard cup burner apparatus. A cylinder of FK-5-1-12 was held in a heated bath of 3M™ Novec™ 7500 Engineered Fluid (B.P. = 130ºC) at 90 ºC. All lines that contained saturated FK-5-1-12 vapor between the cylinder and the cup burner apparatus were heated using heating tape and a variable transformer to maintain a minimum temperature of 90 ºC. A metering valve with a vernier handle was used to introduce the saturated agent vapor into the air stream of the cup burner apparatus. Once the agent is introduced into the air stream in gaseous form, it will not re-condense to the liquid phase, because it is well below its saturation concentration. The air/agent mixture was passed through a column filled with glass beads to ensure complete mixing before the gas sampling port. The apparatus is shown below in Figure 1. The mixed stream then entered the cup burner apparatus and the tests were run according to the ISO standard. All tests were run at a flow rate of 40 L/min, in accordance with Draft Annex B[2].

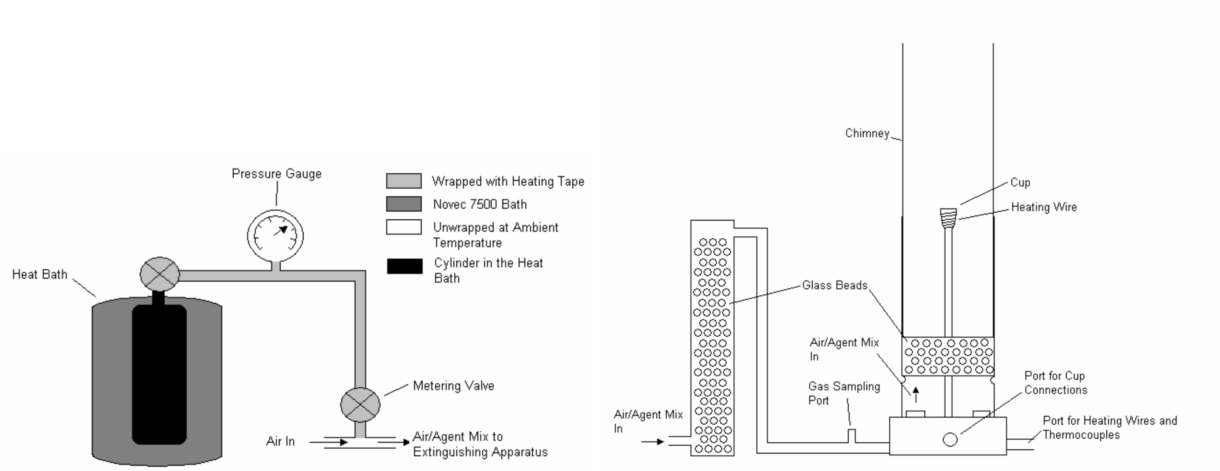

Figure 1: Supply Apparatus to Air Flow

In instances where heating of the fuel was required a different cup containing heating wires within the cup was utilized. A variable transformer was used to pass current through the cup in order to achieve the desired fuel temperature.

The flow rate was controlled with two Manostat 36-541-305 Rotameters. The rotameters were calibrated at ambient temperature and pressure through a six-point calibration using a DryCal DC-Lite Flow Meter. The ambient calibration was then converted to a standard temperature and pressure calibration using the ideal gas law. This STP calibration was then used before each test to adjust the airflow rate for differences in ambient temperatures and pressures.

The temperature of the heated bath is not critical but must only be maintained at a constant temperature. This temperature need only be sufficient to produce an adequate agent vapor pressure to overcome any pressure of the airflow. The temperature of 90 ºC was chosen as a result of trial and error. The temperature of the fuels was monitored using a K-type thermocouple and an Omega HHM29 digital multimeter.

Gas samples were obtained through the gas sampling port, as shown in Figure 1. A 100 mL gas-tight syringe was used to pull a sample from the gas sampling port. The sample was then injected into a 300 mL tedlar bag. The tedlar bag was then opened, the sample was pushed out and the bag was resealed. This procedure was repeated once more to ensure that the bag had been purged of any residual gas that may be in the bag. A 100 mL sample was again taken from the gas sampling port and injected into the sealed tedlar bag. A 5 mL gas-tight syringe was then used to transfer 1.0 mL of sample from the tedlar bag into an evacuated FT-IR gas cell. A PerkinElmer 1600 Series FT-IR was used to analyze three samples from each tedlar bag.

The cup burner was run at least five times for all fuels. An average mass concentration was then calculated from three separate absorbance peaks on the IR spectrum, 1303 cm^{-1}, 1265 cm^{-1} and 1175 cm^{-1}.

The average extinguishing concentration is reported with 95% confidence limits from this average. The confidence limits are calculated from equation (1):

$$\text{Confidence limit} = \overline{X} \pm 1.96\left(\frac{\sigma}{\sqrt{n}}\right) \qquad (1)$$

where $\overline{X}$ is the average value, σ is the standard deviation and n is the number of values.

The recommended extinguishing concentrations are a compilation of testing at 3M and testing conducted at Kidde Research in the UK, which was witnessed by the European laboratory and notified body Centre National de Prevéntion et de Protection (CNPP). Results to date for various fuels with FK-5-1-12 are shown below in Table 1.

Inerting Tests Procedure

The inerting testing was conducted in accordance with the procedures outlined in ISO 14520:2000 Annex D[3]. Testing was conducted to determine the inerting concentrations of FK-5-1-12 for various methane and propane air mixtures. The inerting test is conducted by attempting to ignite various concentrations of air-agent-fuel mixtures using a DC spark. The agent concentration is determined on a mass basis and the fuel concentration is determined by an equivalence ratio. The equivalence ratio is the actual fuel-air ratio relative to the stoichiometric fuel-air ratio. When the ignition spark is introduced to the mixture within the vessel, a pressure increase less than 1.0 psig (7.1 kPa) indicates that the mixture was inert; a pressure increase greater than or equal to 1.0 psig (7.1 kPa) indicates that the mixture was not inert[4]. The criterion stated in this standard is that a closed-vessel deflagration pressure rise greater than 7% of the initial pressure, or 1 psi (7.1 kPa) when $P_{ambient}$ is 14.7 psi (101.3 kPa), is considered combustible[5].

Fuel	Extinguishing Concentration Vol %
acetone	4.3
acetonitrile	2.9
cyclohexane	4.5
diesel fuel	3.5
ethanol	5.6
ethyl acetate	4.7
n-heptane	4.5
commercial heptane	4.3
isopropyl alcohol	4.9
isooctane	4.7
methanol	6.6
methyl ethyl ketone	4.5
methyl tert butyl ether	4.5
octane	4.4
n-pentane	4.7
1-propanol	5.4
tetrahydrofuran	5.0
toluene	3.5
transformer oil (Voltesso)	4.5

Table 1: 3M Recommended FK-5-1-12 Cup Burner Extinguishing Concentrations

In order to determine the maximum inerting concentration for each fuel, the equivalence ratio, or phi, of the fuel air mixture was varied from 0.6 – 1.2, in increments of 0.1. Phi is defined in equation (2) below:

$$\Phi = \frac{\left(x_{fuel} / x_{O_2} \right)_{actual}}{\left(x_{fuel} / x_{O_2} \right)_{stoichiometric}} \qquad (2)$$

where x is the mole fraction of the fuel or oxygen.

For the individual fuels the equations (3) and (4) were derived:

$$\Phi_{methane} = 9.52\left(P_{fuel} / \left(P_{ambient} - P_{agent} - P_{fuel} \right) \right) \qquad (3)$$

$$\Phi_{propane} = 23.8\left(P_{fuel} / \left(P_{ambient} - P_{agent} - P_{fuel} \right) \right) \qquad (4)$$

where p is the atmospheric pressure or partial pressure of the individual component.

Concentrations of the agent were then varied with each value of phi, in increments of 0.5%. Each combination of agent concentration and phi was then tested at least 3 times in order to determine reproducibility of the tests. Agent concentrations were studied at each value of phi to find the interval where the mixture passed through the inerting threshold. After each phi from 0.6 – 1.2 was examined the phi yielding the highest agent concentration to cross the 1.0 psig (7.1 kPa) threshold was then further studied to determine a more accurate inerting concentration.

The tests were run using a 6.37L spherical vessel as shown. Penetrations into the vessel provide access for instrumentation. Pressures were monitored using Omega™ PX303-050AV and PX425 pressure transducers. An Omega™ 0.002 inch (0.051 mm) diameter chromel-alumel K-type thermocouple located in the upper hemisphere of the vessel was used to monitor the temperature. Pressure transducer and thermocouple data were collected by a National Instruments™ DAQCard-6063E. Data was collected at 100 Hz to ensure adequate resolution. The data collection system was run on an IBM™ ThinkPad™ T21 using LabVIEW™ version 6.1 software.

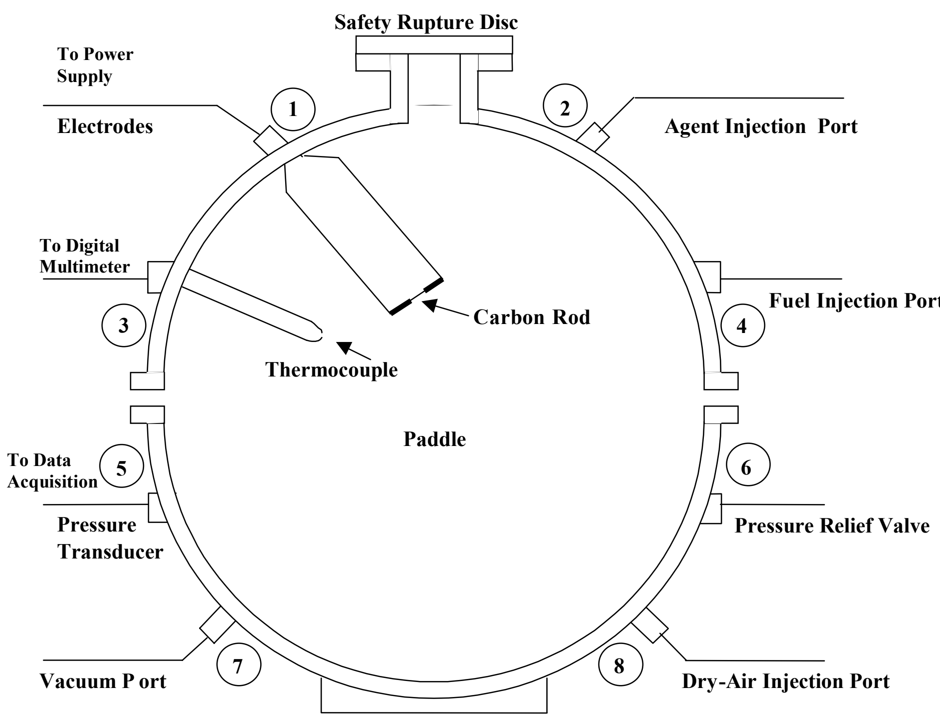

Figure 2: Schematic of Inerting Vessel

The mixture was loaded into the evacuated vessel in the following order; agent, fuel then air passed through a Drierite column. This order was used to allow for increased safety. A measured mass of agent was added to the test vessel via syringe injection. The fuel was then added using the PX425 pressure transducer, as it was more accurate than the PX303-050AV pressure transducer at pressures below atmospheric. Dry air was then introduced to bring the vessel back to atmospheric pressure. The mixture was then mixed for 30 seconds by rocking the vessel to agitate the internal paddle. After mixing, the DC power supply was then allowed to charge to 200 VDC for 60 s, providing spark energy of 20 joules. This spark energy is well above the minimum ignition spark energy of a fuel-air mixture. At 55 s the data logging was turned on and at 60 s the ignition spark was discharged. The data was logged for 15 seconds after ignition to record any transients. Pressure changes within the vessel were recorded with the PX303-050AV pressure transducer. The data was then examined to determine the maximum temperature and pressure achieved during the test. The vessel was evacuated three times prior to each test to purge any remaining gases of the previous test. Also the vessel was opened and wiped clean with a wet paper towel to remove deposits after every third test. The inside was dried before reassembly. Tests where an experimental error occurred were ignored in the final analysis.

Inerting Test Results

The results of the testing for methane and propane are shown below in Figure 3 and Figure 4.

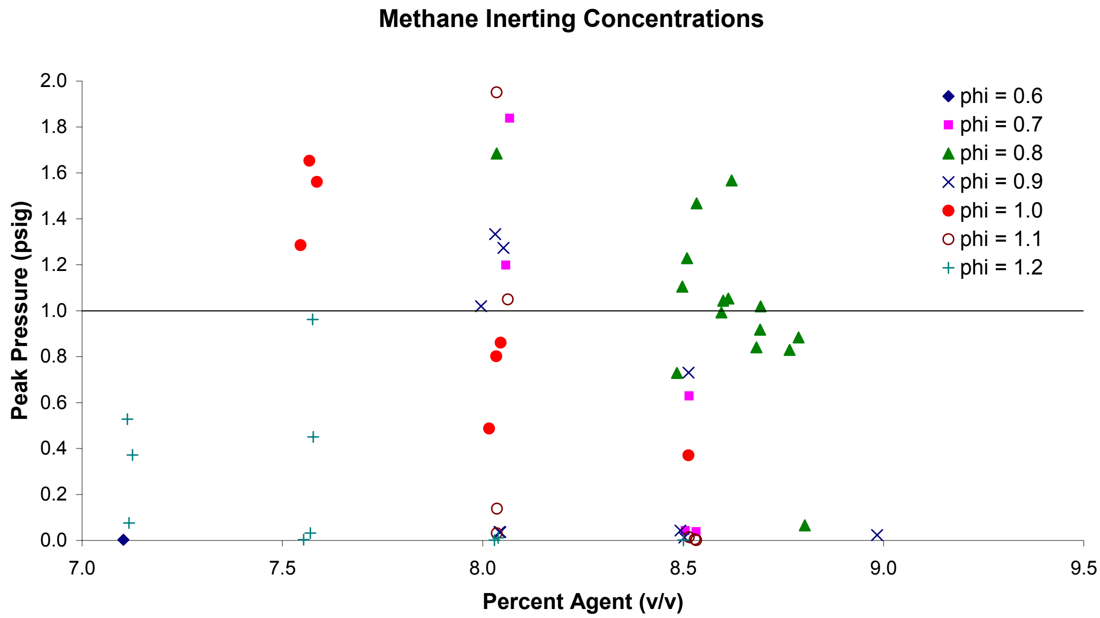

Figure 3: Methane Inerting Concentrations

Propane Inerting Concentrations

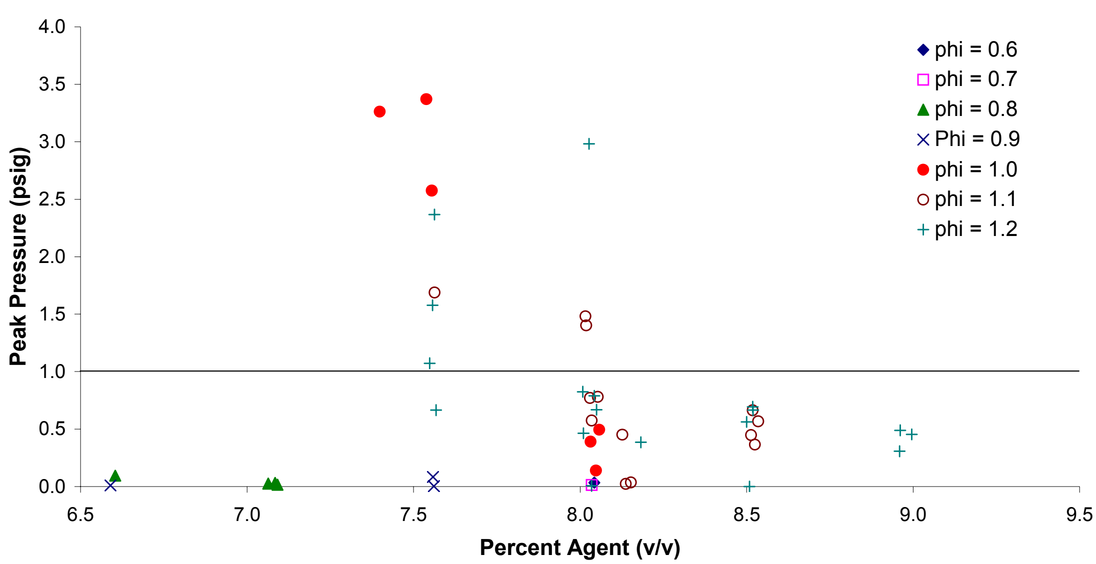

Figure 4: Propane Inerting Concentrations

The minimum inerting concentrations demonstrated by FK-5-1-12 for propane and methane test results are shown in Table 2.

Demonstrated minimum FK-5-1-12 inerting concentration		
	Methane	Propane
Equivalence Ratio	0.81	1.11
Inerting Concentration	1.17 kg/ m^3	1.06 kg/m^3
Volume Percent	8.79% v/v	8.06% v/v

Table 2: Minimum propane and methane inerting concentration

Temperature at Discharge

FK-5-1-12 fire performance testing is reported and conducted in a large-scale, $100m^3$ test enclosure and intermediate-scale, $1.28m^3$ enclosure at 3M facilities. Thermocouples located directly in the discharge nozzle spray and other locations within the protected space were used to monitor the temperature. Discharge and post-discharge room temperature variations were recorded with respect to proximity of the discharge nozzle and location within the enclosure. Data is contrasted with system performance involving other commercially available halocarbons.

Several cold discharge tests were performed to determine the room temperature affects of FK-5-1-12 during discharge without the added heat of a fire. The tests results shown in Figure 5 were performed in a $1.28m^3$ enclosure and temperature readings were taken at 1mm, 0.33m, 0.62m, 0.94m, and 1.25m beneath the discharge nozzle. Cold discharge testing was also conducted in a 100m^3 test enclosure with thermocouples located directly beneath the nozzle, 3.87m, 2.15m, and 0.43m above the floor under the nozzle as shown in Figure 6.

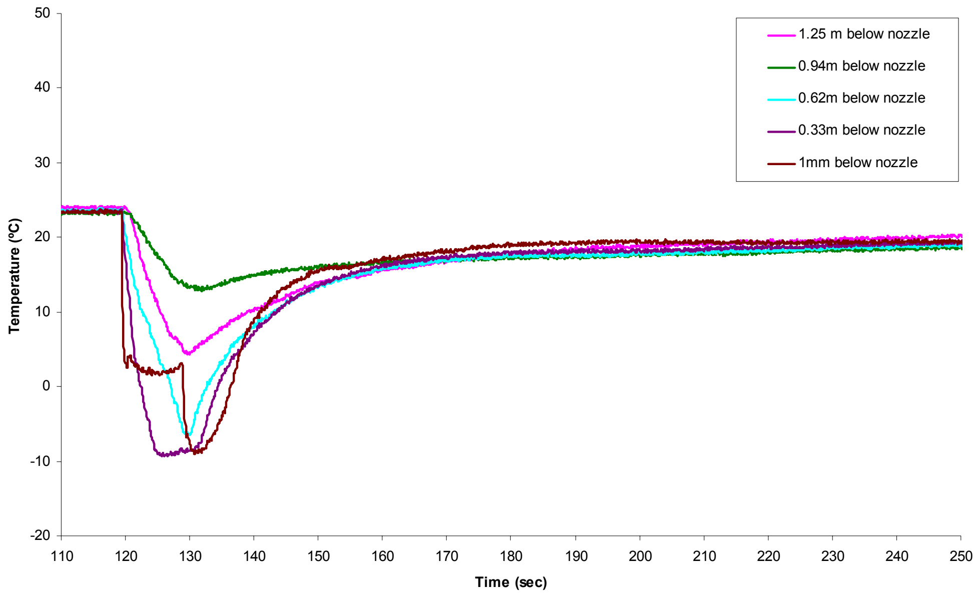

Figure 5: Temperature Profile in 1.28m3 enclosure of FK-5-1-12 discharge at a concentration of 5.9% from a 42 bar system.

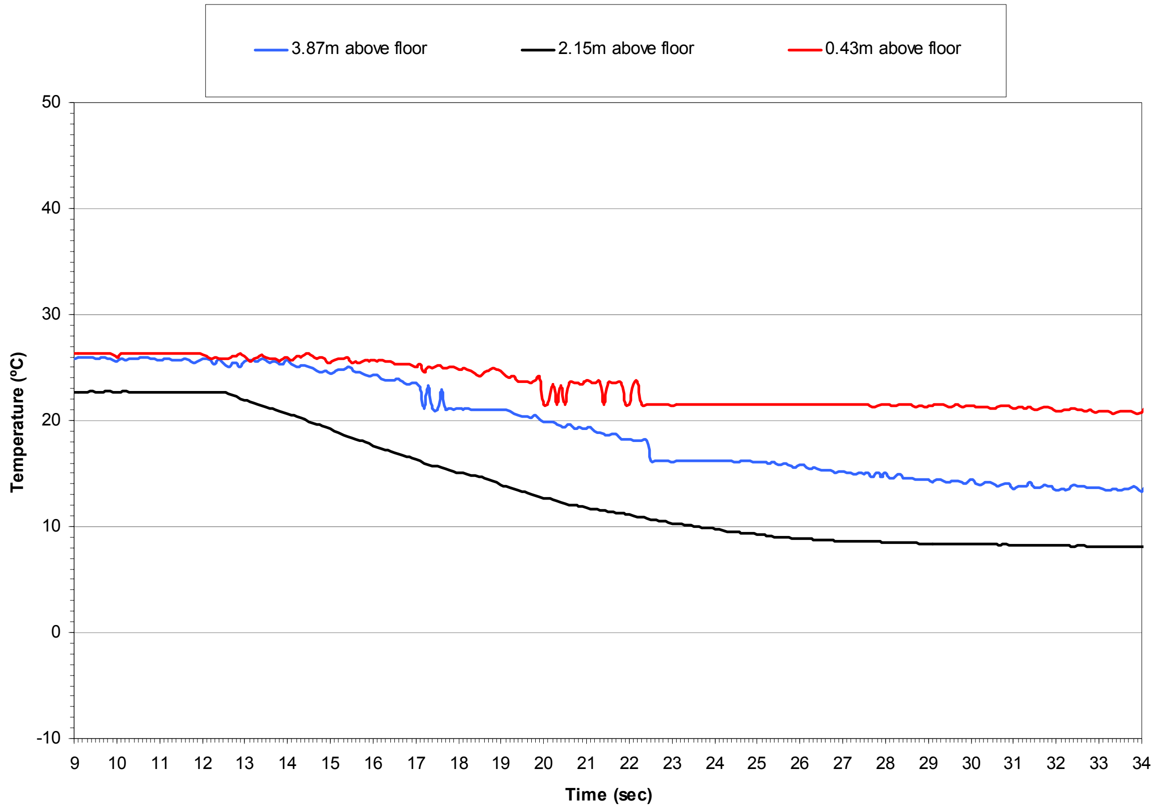

Figure 6: Temperature Profile in 100m^3 enclosure of FK-5-1-12 at a concentration of 4.0% from a 42 bar system.

The most dramatic temperature change occurs directly under the nozzle during discharge. The farther away from the nozzle the thermocouple is located, the smaller the temperature difference noted.

Alternative Delivery Methods

FK-5-1-12 has a higher boiling point and lower vapor pressure when compared to previous clean agents. These key properties allow it to be delivered to the protected space using novel methods. As opposed to superpressurizing a pressure-rated cylinder with nitrogen for a conventional system containing FK-5-1-12, the technology now exists for the delivery of the agent in a manner similar to what has been termed "new technology delivery"[6] or "piston flow"[7]. The technology examined in the previous work uses nitrogen stored in a separate high-pressure cylinder connected to a standard non-superpressurized agent cylinder. Upon actuation, the nitrogen cylinder opens to push the agent from the agent cylinder through a system of piping to be discharged through a special nozzle into the protected space. Two methods alternative to this, but accomplishing similar results, have been examined.

The first alternative method used with FK-5-1-12, has been described in research on small applications using FK-5-1-12 and a solid propellant gas generator (SPGG) [8]. In this research, it was concluded that hybrid fire extinguisher testing on a small scale has demonstrated that higher-boiling fluids, such as FK-5-1-12 can be successfully discharged when pressurized by a solid propellant gas generator. But, it has now been demonstrated that SPGG technology can effectively be utilized in conventional large-scale room applications. In nozzle distribution verification tests in accordance with UL 2166 with an SPGG fixed to a conventional cylinder head, FK-5-1-12 has been shown to discharge in a piston flow fashion from a cylinder generating a nozzle pressure trace that looks remarkably similar to a conventional superpressurized system as shown in Figure 7 below. Once actuated, the SPGG vigorously vents inert gas into the cylinder head space to push FK-5-1-12 from the cylinder through the cylinder dip tube and into the system piping. The difference is that the cylinder in storage remains under only the low vapor pressure of neat FK-5-1-12.

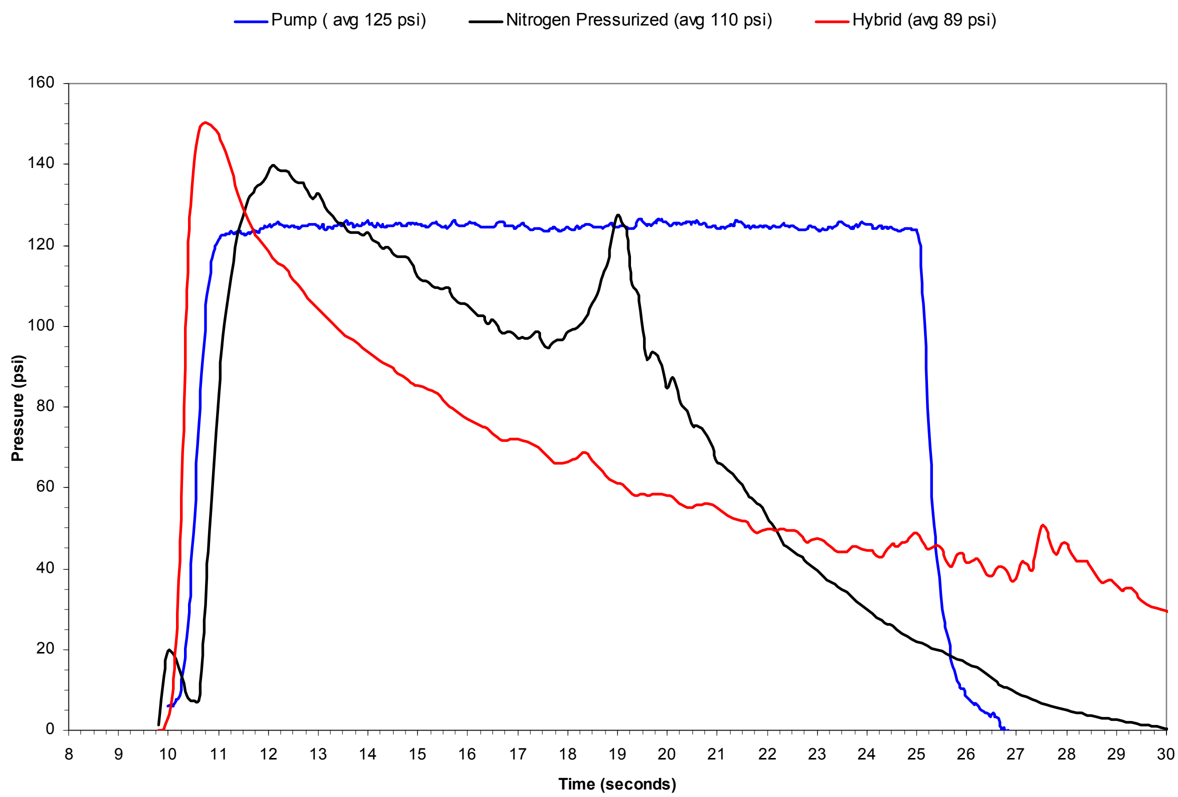

Figure 7: Nozzle pressure comparison, in 100m3 enclosure, standard UL2166 NDVT scenario

Also note in Figure 8 is an example of a nozzle pressure trace from a heretofore untried method for clean agent delivery in a conventional fire protection application. In this application, a specially engineered, multi-stage centrifugal pump takes suction from a reservoir at atmospheric pressure that is filled with FK-5-1-12 and delivers it to the protected space through standard piping and fittings. At reasonable flow rates and system and nozzle pressures, this agent delivery method into a standard test enclosure can effect extinguishment of UL 2166 nozzle distribution verification test fires for low ceiling maximum area and maximum height enclosures. Recent research and development has taken place to validate this concept.[9]

Conclusions

Product and commercial development of FK-5-1-12 continues including systems applications. Fire performance for this internationally recognized clean extinguishing agent has been validated using small-scale apparatus to determine inerting and cup burner concentrations as well as fire performance in large-scale testing as part of laboratory listing programs. All fire performance testing has been conducted in accordance with recognized international standards.

Physical properties testing have progressed in a continuing effort to define the characteristics of this product. Alternative non-conventional system delivery methods have been successfully attempted to take advantage of the unique properties of FK-5-1-12. Further testing continues to augment data that now exist validating agent delivery to the protected space in new and useful ways.

References

[1] ISO/WD 14520-1: September 2003 Annex B *Determination of flame-extinguishing concentration of gaseous extinguishants by the cup burner method.*

[2] ibid, reference 1

[3] ISO 14520-1:2000(D) © ISO 2000, *Method of evaluating inerting concentration of fire extinguishing vapour,* ISO copyright office, Case postale 56 CH-1211 Geneva 20.

[4] ibid, reference 3, page 51.

[5] Ibid, reference 3, page 51.

[6] Senecal, Joseph A, Kidde Fenwal, Inc, *New Technology Delivery System for FM-200® Clean Agent*, Halon Options Technical Working Conference, 24-26 April, 2001, Albuquerque, NM

[7] Robin, Mark, et al, *US Patent No. 6112822, Method for Delivering a Fire Suppression Composition to a Hazard ...,* September 5, 2000.

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[9] Reser**,** Thomas W.**,** Smith**,** David M., CAPS Fire, Inc., *Examination of an Alternative Delivery Method for a Sustainable Clean Agent in a Total Flooding System*, Halon Options Technical Working Conference, 4-6 May 2004, Albuquerque, NM