

**DIODE LASER MEASUREMENTS OF HF CONCENTRATIONS PRODUCED FROM
HEPTANE/AIR PAN FIRES EXTINGUISHED BY
FE-36, FM-200, FE-36 PLUS APP, OR FM-200 PLUS APP**

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ABSTRACT

Tunable diode laser absorption spectroscopy (TDLAS) is used to measure the time evolution of hydrogen fluoride (HF) concentrations produced from a series of enclosed heptane/air pan fires extinguished by either FE-36, FM-200, FE-36 plus ammonium polyphosphate (APP), or FM-200 plus APP. For the fires studied, the change in HF gas concentration with time is dependent upon the fire fighting chemical used to extinguish the fire. The presence of APP is observed to reduce HF concentrations in the fire enclosure. Visible attenuation spectroscopy is also used to measure the amount of light attenuation (obscuration) that occurs as a hand held fire extinguisher containing powdered fire fighting agent is released in the crew space of a M1-Abrams land combat vehicle.

Key Words: Diode laser, HF, FE-36, FM-200

INTRODUCTION

Fire protection on military platforms, including ground fighting vehicles, is being challenged by the impending loss of the ubiquitous fire fighting agent halon 1301 (CF_3Br) due to environmental concerns related to the destruction of the stratospheric ozone layer. Replacement fire extinguishment agents need to be found that will satisfy numerous criteria including: fast fire suppression, minimum

production of toxic gases when used, low toxicity, compatibility with storage materials, and environmental acceptability.

The U.S. Army's search for halon replacement agents has largely involved an empirical approach of testing and evaluation of commercially available compounds/systems. Testing and evaluation of two candidate halon replacement agents FE-36 ($C_3F_6H_2$) and FM-200 (C_3F_7H) is presented here from full scale fire suppression tests conducted at the Aberdeen Test Center. The fluorinated propane agents, though effective at extinguishing fires, are deficient in terms of increased weight and volume requirements needed for fighting the most difficult military fires. The increased amounts of replacement agent required for extinguishment, relative to halons 1301 and 1211, pose a concern with the levels of HF formed as the primary decomposition product. Thus, the tests presented here measure the concentration of gas phase hydrogen fluoride (HF) using near-infrared tunable diode laser absorption spectroscopy (NIR-TDLAS) [1]. A recent article from this laboratory details the use of NIR-TDLAS as the most effective diagnostic for gas phase HF measurements [2].

The two mechanisms by which HF concentrations can be decreased during and following fire extinguishment by FE-36 or FM-200 are by 1) reducing the time required to extinguish the fire, thereby minimizing the time that the fluorine containing suppressant is exposed to flame temperatures, or 2) releasing a scavenging agent in conjunction with the fire suppressant chemical to remove HF after it is produced. A potential chemical scavenging agent of gaseous HF is ammonium polyphosphate (APP), which is a commercially available chemical (Phos-chek™) with fire retardant properties. One of the drawbacks to the release of a powder agent in an occupied space is the possibility of reduced visibility.

HF is produced here from a series of enclosed heptane/air pan fires extinguished by either FE-36 ($C_3F_6H_2$), FM-200 (C_3F_7H), FE-36 plus APP, or FM-200 plus APP. By measuring how much the HF

concentrations are reduced in fires extinguished with FE-36/APP or FM-200/APP mixtures compared to fires extinguished by FE-36 or FM-200 alone, the effectiveness of APP as a HF reduction and potential scavenging agent will be demonstrated. In addition, spectroscopic measurements of the amount of light attenuation (obscuration) that occurs as a hand held fire extinguisher containing powder fire fighting agent is released in the crew space of a M1 land combat vehicle will be discussed.

EXPERIMENTAL

Figure 1 shows a schematic diagram of the test facility containing the NIR-TDLAS experimental apparatus and signal processing electronics. TDLAS has proven to be a valuable, non-intrusive optical diagnostic for species concentrations and temperatures in combustion systems even when the local soot level is high [3,4,5,6,7,8,9]. The test facility is a 1.5 m³ cubic enclosure, which contains a liquid heptane pan fire (fire size approximately 30 KW). The fire suppressants used were FE-36 or FM-200. For the extinguishers with APP added, the following combinations of FE-36 plus APP and FM-200 plus APP were tested: FE-36 plus 7 % APP, FE-36 plus 15 % APP, and FM-200 plus 15 % APP.

The test protocol was as follows: A 250 ml pan filled with heptane fuel was placed underneath 0.5 m high steel table within the cubic container. The NIR-TDLAS HF gas measurement apparatus was situated on top of the metal table where a HF gas cell was placed in the line-of-sight path between the GRIN lens from which the 1.3 micrometer laser radiation was emitted and the InGaAs detector. The HF gas cell was used to provide a HF spectrum to tune the laser and data collection system. The HF gas cell was removed prior to fire testing. A hand held bottle-type extinguisher charged with 770 g FE-36 plus scavenging agent (when applicable) and pressurized with N₂ gas was situated on top of the cubic container. The extinguisher bottle output nozzle was attached via tubing to a spray nozzle protruding into the cubic container.

The heptane pan fire was ignited using a butane electric match and the NIR-TDLAS HF gas measurement was initiated immediately after an internal cubic container fan was turned on and the container door was closed. The heptane pan fire was allowed to burn for 15 seconds at which time the hand held extinguisher's contents were discharged into the interior of the cubic container producing total flooding inhibitor conditions (10 % by volume FE-36 concentration). A video camera mounted within the cubic container recorded the fire event during the tests. HF data collection occurred for 180 s after ignition of the fuel, at a rate of 1 spectrum per second.

Following each test, the interior of the cubic container was rinsed with an aqueous solution of sodium bicarbonate to neutralize any acid residues on the interior surface and the container was allowed to air dry. The experimental details and analysis of NIR-TDLAS HF spectra have been described previously [2] and will only be summarized here as described below.

A sawtooth modulation (≈ 100 Hz) supplied from a Function Generator (Tektronix Model FG 504) was used to rapidly scan over the desired spectral frequency range. Coincident with the 100 Hz modulation was a small amplitude but higher frequency modulation, ≈ 20 KHz, taken from the sine output of a SRS Model 830 DSP Lock-in Amplifier. The application of the two modulations to the laser diode current facilitated wavelength modulation spectroscopy (2f detection) which was needed for enhanced signal sensitivity [10,11,12,13,14]. Wavelength modulation spectroscopy (WMS) is convenient for analysis because of the linear relationship that exists between the measured absorption signal and the analyte's concentration.

When WMS is employed, the second derivative of the diode laser probe beam intensity with respect to wavelength is usually measured, since the wavelength at which the second derivative is a

maximum coincides with the wavelength of maximum light absorption. To quantify a 2f absorption signal, denoted as x'' , the Bouguer-Lambert law is written as [6]

$$\frac{x''}{I^\circ} = H_2(\nu - \nu^\circ) \cdot S(T) \cdot g(\nu^\circ) \cdot P_j \cdot L \quad \text{Equation 1}$$

From **Equation 1**, H_2 is the second Fourier component of the modulated absorption coefficient, $S(T)$ is the temperature-dependent line strength for the transition, $g(\nu^\circ)$ is the lineshape factor evaluated at the line center, P_j is the partial pressure of the absorbing analyte, L is the path length, and I° is the incident laser intensity. The implementation of **Equation 1** for the quantification of measured absorption signals requires knowledge of the quantities H_2 , S , and g . For TDL measurements, Hanson et al. [15,16,17] demonstrated that the product of the linestrength S ($\text{cm}^{-2} \text{atm}^{-1}$) and the lineshape factor $g(\nu - \nu^\circ)$ (cm) yields the absorption coefficient, $\alpha_\nu = S \cdot g(\nu - \nu^\circ)$, which is normally used when quantifying a

measured signal in terms of absorbance, i.e., $\ln\left(\frac{I^\circ}{I}\right)_\nu = \alpha_\nu \cdot P_j \cdot L$. To quantify a 2f absorption signal,

Reid and Labrie [10] determined that the modulation of the absorption signal is accounted for in the Bouguer-Lambert law with the addition of the second Fourier component of the modulated absorption coefficient, H_2 , which describes the modification of the spectral lineshape. Hence, the absorption coefficient for a 2f absorption signal can be re-written as, $\alpha_\nu = H_2 \cdot S \cdot g(\nu - \nu^\circ)$. The values for α_ν are easily determined from signal calibrations of the 2f signal measured in an atmospheric pressure continuous flow cell, pathlength of ≈ 12 cm, containing known concentrations of HF in N_2 . For calibrations,

Equation 1 is simplified to:

$$\frac{x''}{I^\circ} = \alpha_\nu \cdot P_j \cdot L \quad \text{Equation 2}$$

From **Equation 2**, the slope obtained from a plot of x''/I° , the normalized 2f signal peak height (peak to trough height, the distance between the maximum and minimum points on the 2f signal), versus $(P_j \cdot L)$ provides the value for α_ν . Once the α_ν value is known, the unknown gas pressure (P_j) may be obtained directly from **Equation 2**. Calibrations are performed at the beginning and end of each measurement set so that changes in optical surfaces during measurement (which may occur when measuring corrosive gases such as HF) do not affect the value of α_ν .

The emitted infrared light from the diode was launched into a fiber coupled to the laser housing. Fiber optic patch cables were used to deliver infrared light into the test facility. The fiber is terminated by a gradient index (GRIN) lens (Sentech Systems, Inc.), which collimates the laser radiation. The GRIN lens-tipped fiber is placed into a “pitch and catch” arrangement which directs the infrared radiation over a 14 cm open path to a InGaAs detector (Epitaxx Model ETX 1000 T). The signal observed at the detector was directed to a phase sensitive, lock-in amplifier (Stanford Research Systems Model 830) which acquired the $2f$ absorption signal. The $2f$ output signal from the SRS lock-in amplifier was then sent to a digital oscilloscope (LeCroy Model 9654). Resulting spectra were acquired every second for a three minute time period and then sent to a Pentium™ based laptop computer for storage and analysis. For experiments described here, the P(2) transition of the first vibrational overtone of HF was monitored at 7665 cm^{-1} [5].

Figure 2 presents a schematic representation of the experimental apparatus used for attenuation testing. The apparatus consists of a HeNe laser (Oriel Model 6611 output 632 nm), an Optical Chopper (Stanford Research Systems Model SR540), and a fiber optic fiber coupler (Newport Optics Model F-916T). The experimental equipment was mounted to a 46 x 46 cm optical bread board which was placed outside the crew compartment on top of the vehicle. A separate 41 cm optical rail supported the fiber collimating/ projection optics and a 15 mm^2 (active element) photodetector (Centro Vision Inc. OSD5-5T, 350-1100 nm). The optical rail was placed inside the vehicle in the driver’s seat. Not shown is a plastic enclosure that was placed over the fiber collimating/projection optics to minimize the optic devices exposure to the powder agent. A 18 m BNC cable and extension cord provided remote signal communications and power to the chopper and laser while another 18 m BNC cable transported the detector signal out to the detection electronics. The tests consisted of an occupant releasing a Kidde™ 2.75lb hand held fire extinguisher containing either FE-36 plus APP or NaHCO_3 plus N_2 at the personnel heater while inside the vehicle and all outside hatches were closed. With the hatch doors closed, the

vehicle's nuclear and biological containment (NBC) system was operated to create a positive pressure inside the crew compartment.

Light attenuation is determined by monitoring the percent transmission of HeNe laser radiation that is incident on the detector as powdered agent is dispersed over the measurement region. Thus, if 100 % transmission occurs, all the light that exits the laser source is incident upon the detector and no attenuation occurred. The laser radiation is passed through an optical chopper, which modulates the laser radiation at ≈ 1000 Hz. After passing through the chopper, the laser radiation is launched into a 8m long visible fiber optic cable (3M™ multi-mode, 1000 mm diameter core, glass substrate optical fiber). Light passes out the opposite end of the fiber where it is terminated into a 11 mm SMA fiber ferrule (Oriol Model 77670). The terminated fiber is inserted into a glass collimating beam probe (Oriol Model 77645). The collimated light exits the beam probe and is directed into a visible Nikon™ objective lens, which apertures the beam to approximately 5-6 mm and directs it over a 0.4 m free space where the laser radiation is incident onto the 15 mm² photodetector. The free space through which the laser radiation passes is the region in which the powdered agent, if present, will attenuate the laser radiation. The light intensity reaching the detector is converted to a voltage, which is then measured using a lock-in amplifier referenced to the chopper frequency. A DC voltage (0-5 V full scale), proportional to the detector signal, is output to the digital oscilloscope (LeCroy Model 9654), which is also triggered by the optical chopper, and is processed and recorded using a Pentium based laptop computer at a rate of 2 Hz.

RESULTS

Figure 3 presents average HF concentration profiles from extinguishment tests with FE-36, FE-36 plus 7 % APP, FE-36 plus 15 % APP, FM-200, and FM-200 plus 15 % APP. For the FE-36 plus 7 % APP and FE-36 plus 15 % APP tests, three individual profiles for each extinguisher concentration were used to construct each test's average profile. For the FE-36 profile, test data from five different profiles,

collected over the entire testing period, compose the average profile. The FM-200 average profile is constructed from 10 separate tests while the FM-200 plus 15 % APP profile consists of four separate tests. Statistically the 1σ variances for the profiles were 41, 42, 31, 56, and 54 percent for FE-36, FE-36 plus 7% APP, FE-36 plus 15 % APP, FM-200, and FM-200 plus 15 % APP. For the FM-200 and FM-200 plus APP tests, the large statistical deviations are attributed to inconsistent fire extinguishment times from test to test, which was not as evident in the FE-36 tests. **Table I** lists the maximum HF concentrations and time weighted averages from each profile in **Figure 3**. The results from **Figure 3** and **Table I** indicate that fires extinguished by FM-200 produce almost twice as much HF as fires extinguished by FE-36. For fires extinguished by FE-36 plus APP or FM-200 plus APP, the data show that HF is reduced with respect to fires extinguished by the neat agents alone. **Table I** also lists the time weighted average HF values which represent the average dose of HF one would be exposed to from the time of extinguisher release at $t = 15$ seconds until the end of the measurement period, $t = 180$ seconds. The time weighted average is very important because the primary toxicity concern for HF exposure is not the maximum HF one experiences but the average concentration one is exposed to over a period of time. It should be noted that the target non-toxic HF TWA level for these tests was 500 ppm or less. From **Table I** the HF TWA values imply that for the tests with FE-36, FM-200, and FM-200 plus 15 % APP the HF dose is toxic, while the tests with FE-36 plus 7% APP and FE-36 plus 15 % APP achieve minimum to very acceptable HF reductions respectively.

Measurement of HF gas concentration versus time provides a monitor of the fire history, and of the effectiveness of any HF reduction agent used. That is, the time from fire suppressant release until the maximum HF concentration occurs is a measure of the time required for fire extinction (also verified visually using a video recorder), while the rate at which the HF concentration decreases following extinguishment provides a measure of the effectiveness of scavenging agent (when used), or a measure of the rate at which HF gas reacts with the walls of the enclosure. Reduction in fire out times reduces HF

levels by reducing the time the agent is exposed to flame temperatures which causes agent decomposition to HF. **Table II** lists the fire out times from the data in **Figure 3**. Statistically the difference in fire out times between fires extinguished by FE-36 and FE-36 plus 7 % APP are insignificant with a 1σ error of 22 percent. For fires extinguished by FE-36 plus 15 % APP and FM-200 plus 15 % APP, the fire out times were reduced by approximately 33 to 40 percent relative to the fires extinguished by the respective neat agents. The decrease in the TWA HF levels in **Table I** for fires extinguished using FE-36 and FM-200 compared to fires extinguished by FE-36 plus 15 % APP and FM-200 plus 15% APP (approximately a factor of 30 and 2.5 respectively) is partially attributable to the fire suppression properties of APP (reflected by the shorter fire out times).

Figure 3 and **Table I** indicate that the presence of APP, regardless of the percentage in the extinguisher, helps reduce the overall HF levels with respect to those fires extinguished by only neat agents. A possible explanation for the HF reductions is that without APP in the enclosure, fires extinguished by the neat agents are able to produce HF more rapidly than fires extinguished by neat agents plus APP. This statement is supported by **Figure 4** that plots HF concentrations for each extinguisher from $t = 16$ seconds until $t = 31$ seconds which are the time locations from the extinguisher release to just before the HF concentrations reach their respective maximums in **Figure 3**. Linear regression analysis of this data indicates that FM-200 produces HF 39 percent faster than FE-36. For the fires extinguished by FE-36, HF is formed 21 percent faster than the fires extinguished by FE-36 plus 7 % APP while fires extinguished by FM-200 form HF 12 percent quicker than those fires extinguished by FM-200 plus 15 % APP. Obviously the data in **Figure 4** are dependent on the fire out times, and previous studies [18] have shown that as the fire out times increase this is accompanied by an increase in the amount of HF present in the system. Nevertheless the presence of APP appears to slow HF production.

In real fires reduction of fire out times are difficult to control, and if the time duration is extended some measures must be taken to control the HF levels. APP is added to these extinguishers because it is believed that APP can heterogeneously scavenge HF from the post flame gases of a fire situation extinguished by a fluorinated fire fighting agent. To evaluate APP's scavenging abilities, the time rate of change of the HF concentration must be measured.

The rate of change in HF concentration can be compared between extinguishers with and without APP using data from **Figure 3** starting at the maximum HF concentration time (t_0) and plotting the natural logarithm of the HF concentration versus the natural logarithm of the elapsed time from the HF maximum, as seen in **Figure 5**. As the HF concentration for tests with FE-36 plus 15 % APP were well below the target concentration of 500 ppm, no further analysis of the data was warranted [19]. Results from linear regression analysis of the data in **Figure 5** are presented in **Table III**. The difference in slopes for between FE-36 and FE-36 plus 7 % APP tests is approximately 13 percent greater for the fires extinguished by FE-36 plus 7 % APP, while the difference between the FM-200 and FM-200 plus 15 % APP is approximately 5 percent. Thus the faster decreases in HF concentrations from fires extinguished by FE-36 plus APP and FM-200 plus APP versus FE-36 and FM-200 respectively is attributed to the presence of APP.

The primary concern with combining a powder substance like APP in a hand held extinguisher with the fluorinated agents is when the extinguisher contents are released, the powder is temporally suspended in the air forming a visibly dense "cloud" that could be difficult to see through. To address and quantify this situation, a series of obscuration measurements were conducted. **Figure 6** presents results from the obscuration measurements with temporally resolved percent transmission profiles measured during and following the release of the FE-36 plus APP and the NaHCO_3 plus N_2 extinguishers inside a M1-Abrams combat vehicle. The profiles indicate that the maximum light attenuations were 0

percent transmission for the NaHCO_3 plus N_2 extinguisher and 18 percent transmission for the FE-36 plus APP extinguisher. Empirical correlations using a VHS video taken inside the vehicle during the extinguishers discharge indicate that clear visibility to the human eye correlates to an attenuation level of approximately 70 percent transmission. Thus, all percent transmission levels recorded below 70 percent transmission correspond to an obscured field of view at a distance of 30.48 cm. The time duration that visibility is less than 70 percent transmission for the NaHCO_3 extinguisher is 63 seconds while the FE-36 plus APP extinguisher experiences less than 70 percent transmission for 49 seconds. The more rapid return to visibility, i.e. ≥ 70 percent transmission, using the FE-36 plus APP extinguisher is attributed to the fact that the powder is not released in a dry state, rather it is "wet" and thus falls faster to the vehicle floor.

CONCLUSIONS

NIR-TDLAS has been demonstrated to measure HF in a practical field application. The results presented here indicate that HF concentrations produced from fires extinguished by FE-36 plus APP and FM-200 plus APP are being reduced in the cubic test container and that the presence of APP accelerates this reduction. Thus the combination of APP in an extinguisher containing FE-36 or FM-200 appears to reduce HF levels. Visibility reduction during extinguisher deployment was measured inside an actual combat vehicle for extinguishers containing FE-36 plus APP and NaHCO_3 plus N_2 . From an experimental standpoint, more tests should probably be conducted to analyze the reacted APP to understand how APP reacts with HF. Future tests will attempt to meet this concern in order to develop a chemical kinetic mechanism for post fire HF activity.

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TABLES AND FIGURES

Table I: Maximum HF concentrations (ppm) and time weighted average HF concentrations (ppm).

Table II: Fire out times (seconds) from average profiles in **Figure 3**.

Table III: Slope (ppm/second) values from linear regression analysis of HF dissipation rates.

Figure 1: Schematic diagram of experimental HF measurement apparatus.

Figure 2: Schematic diagram of light attenuation measurement apparatus.

Figure 3: Average HF concentration profiles versus measurement time. The () symbols are HF concentrations from fires extinguished by FE-36, the ()) symbols are HF concentrations from fires extinguished by FE-36 plus 7 % APP, the (+) symbols (bottom trace) are HF concentrations from fires extinguished by FE-36 plus 15 % APP, the (,) symbols are HF concentrations from fires extinguished by FM-200, and the (()) symbols are HF concentrations from fires extinguished by FM-200 plus 15 % APP.

Figure 4: Plot of HF concentrations versus measurement time from $t = 16$ seconds to $t = 31$ seconds to illustrate HF production rates. The () symbols are HF concentrations from fires extinguished by FE-36, the ()) symbols are HF concentrations from fires extinguished by FE-36 plus 7 % APP, the (+)

symbols are HF concentrations from fires extinguished by FE-36 plus 15 % APP, the (,) symbols are HF concentrations from fires extinguished by FM-200, and the (()) symbols are HF concentrations from fires extinguished by FM-200 plus 15 % APP. The straight solid lines represent linear regression analysis of the data.

Figure 5: Rate plot of \ln (HF) concentrations versus \ln (time) for fires extinguished by FE-36 only (,), FE-36 plus 7 % APP ()), FM-200 (+), FM-200 plus 15 % APP (,). The straight solid lines represent linear regression analysis of the data.

Figure 6: Percent transmission profiles collected from measurement of light attenuation while an extinguisher containing either FE-36 plus APP (solid line) or FE-36 plus NaHCO_3 (dashed line) was released inside an M1 combat vehicle. The horizontal line drawn across the graph is the 70 percent transmission level which corresponds to the minimal level of clear visibility to the human eye.

	FE-36	FE-36 + 7 %	FE-36 + 15 %	FM-200	FM-200 + 15 %
		APP	APP		APP
HF Maximum, ppm	1394	996	73	2667	1638
HF TWA, ppm	712	493	23	1626	646

	FE-36	FE-36 + 7 %	FE-36 + 15 %	FM-200	FM-200 + 15 %
		APP	APP		APP
Fire Out Times	18	20	12	20	12

	FE-36	FE-36 + 7 %	FM-200	FM-200 + 15 %
		APP		APP
Slopes (ppm/second)	-0.65	-0.75	-0.52	-0.55

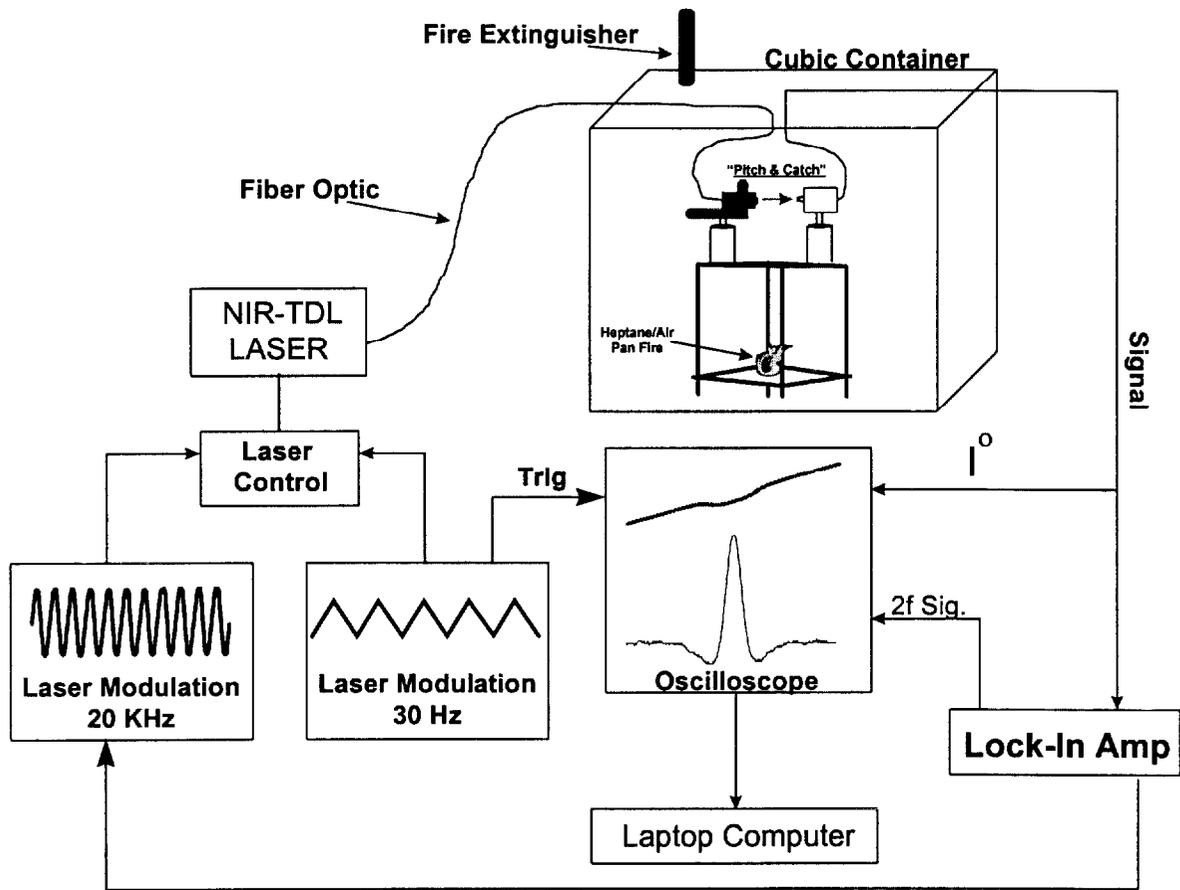


Figure 1

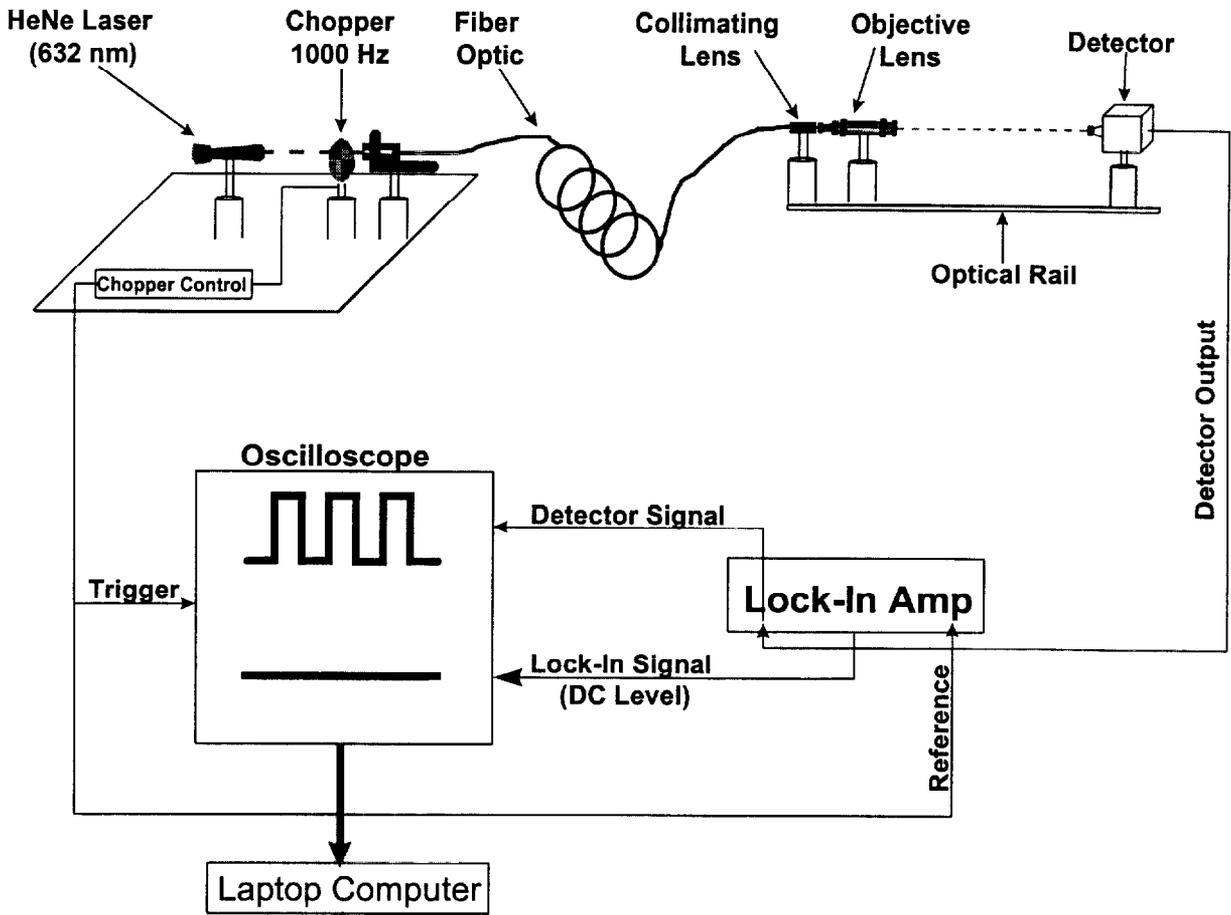


Figure 2

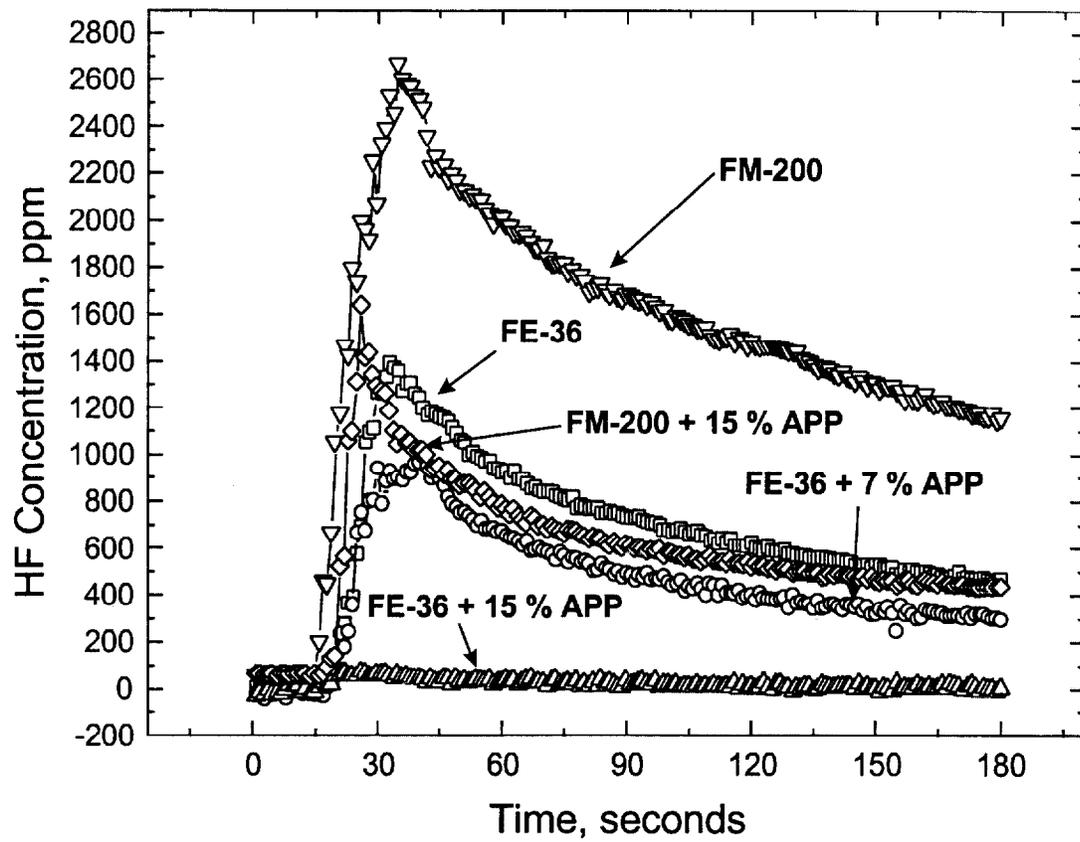


Figure 3

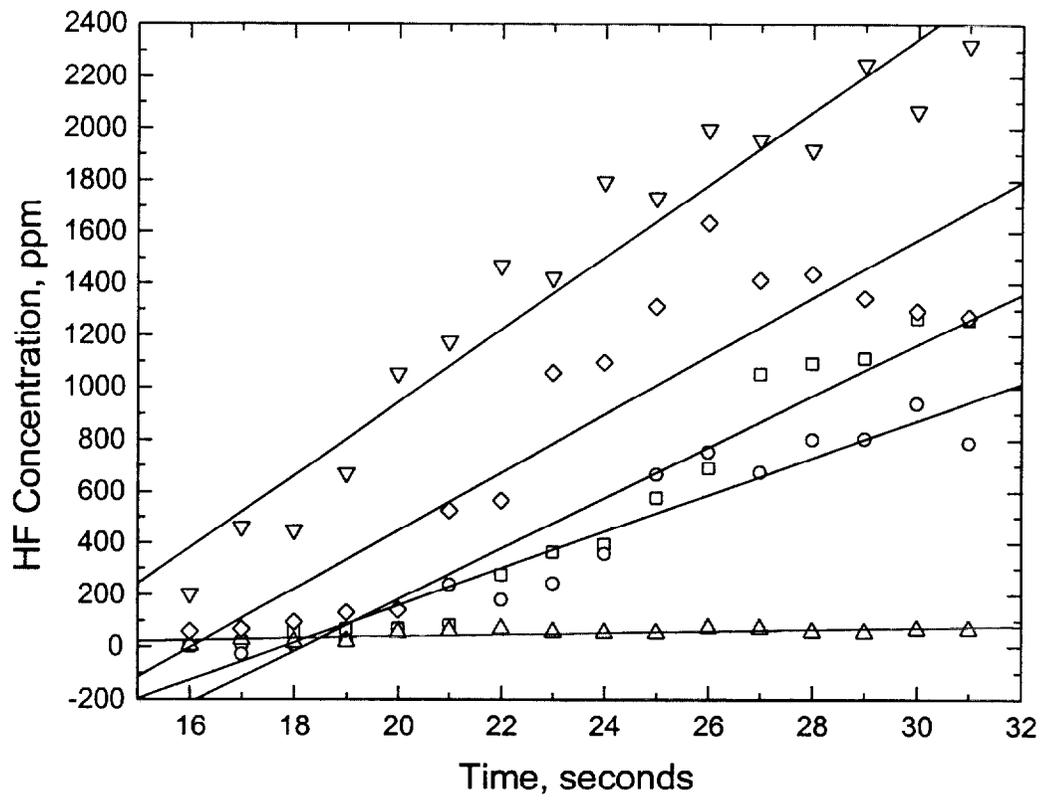


Figure 4

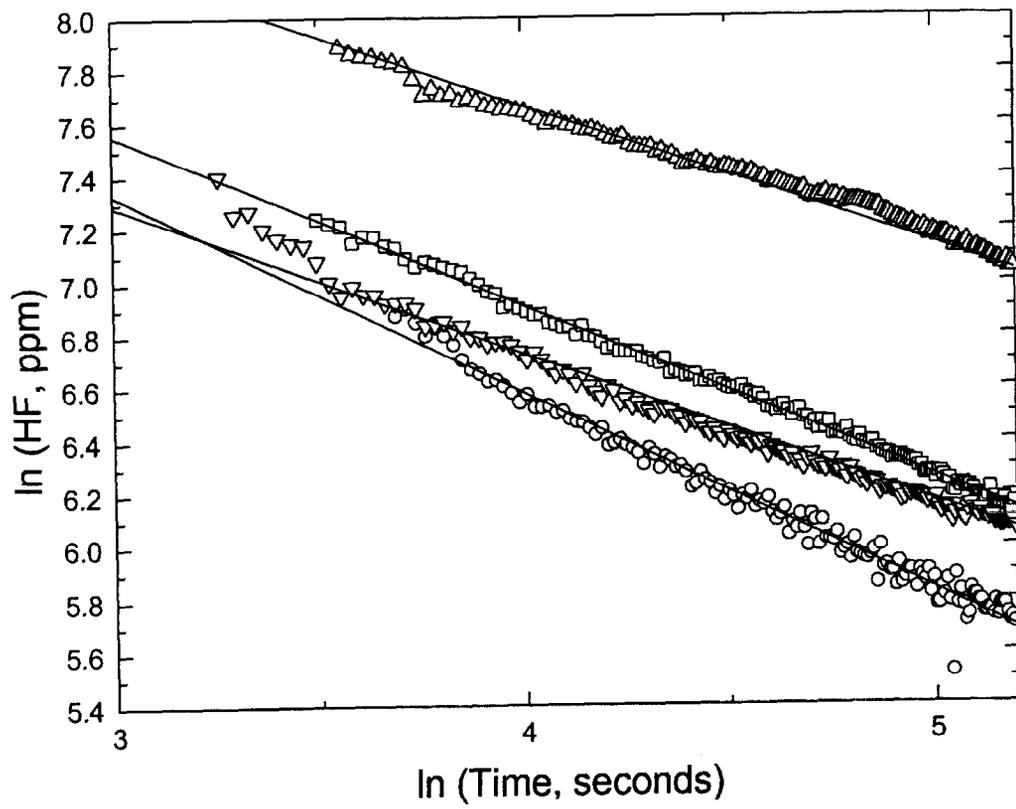


Figure 5

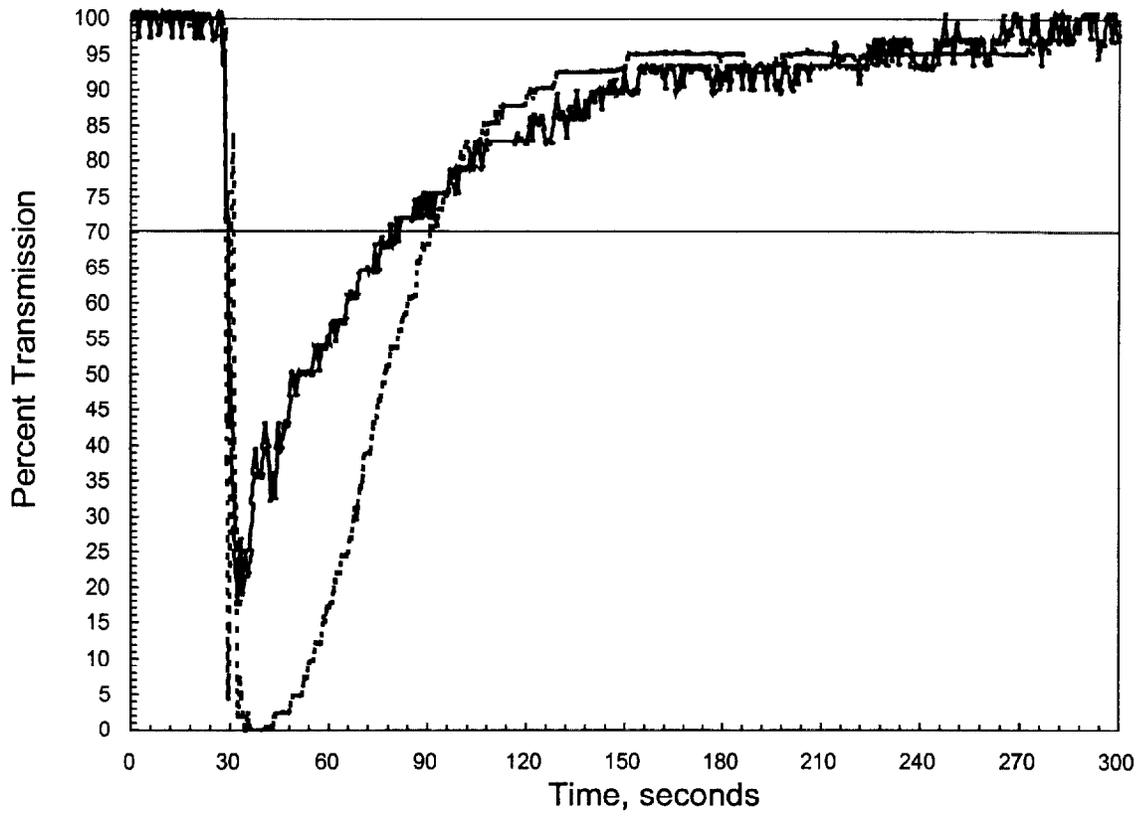


Figure 6

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