NIR-DIODE LASER BASED IN-SITU MEASUREMENT OF MOLECULAR OXYGEN IN FULL-SCALE FIRE SUPPRESSION TESTS

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ABSTRACT

The accurate measurement of oxygen is essential to evaluate the effectiveness of tire suppressant agents and to model the dynamics of the suppression process. We employed tunable diode laser absorption spectroscopy (TDLAS) at 760nm to detect molecular oxygen. This technique has sensitivity to oxygen, minimal interference from other species, and capability of in-situ operation. Measurements were carried out at the NRL Chesapeake Bay Detachment in a 28 m'test compartment under three scenarios: water mist only, unsuppressed fire, and fire suppressed by water mist. This paper will discuss the diode lasers properties, techniques to confine the probe region, and the basic measurement principles of TDLAS based in-situ oxygen sensors under the condition of large, rapid obscuration changes. Finally we present the first results of laboratory measurements and field tests.

INTRODUCTION

The use of water to extinguish a fire is not a new idea. Water is a powerful and convenient agent to suppress fire due to its high evaporation enthalpy, zero-toxicity, availability, and safe handling. Water, in the form of fine mists (droplets $<100\ \mu m$ in diameter), has the potential to replace halons. The effectiveness of water to suppress fire depends very much on the form in which it is used. Studies by the Navy [1, 2] have shown that water is most effective in suppressing fire if it is applied in the form of fine droplets. The optimum droplet size is between $10\mu m$ and $30\ \mu m$ in diameter. Limitations in its use include a lack of mist delivery systems that provide appropriately sized water mist in this size range in sufficient quantities and how to transport the mist to the fire.

Current studies at the Chesapeake Bay Detachment (CBD) of the Naval Research Laboratory are investigating the effectiveness of water mist versus gaseous suppression systems with regard to cost, space, and weight. For these studies two steel compartments representing flammable liquid storage rooms onboard Navy ships have been built: one with a volume of $28\ m^3$ ($1000\ ft^3$) and a larger one with $298\ m^3$ ($10{,}500\ ft^3$) [3]. Performance tests of various fire suppression systems, including water mist, and an evaluation of how to implement these systems are being carried out. To accomplish these goals, a fairly comprehensive data set is needed including the concentrations of fuel and oxygen.

The O_2 measurement in the presence of both liquid and vaporized water is not trivial. Extractive techniques are not capable of determining the actual oxygen concentration because the ratio between the two phases of water in the sample is not obtained. The decomposition products of fluorohydrocarbon agents attack ZrO_2 in-situ probes and lead to erroneous O_2 concentrations. Also, oxygen has no mid-IR absorption and cannot be detected by conventional IR-spectroscopy. Tunable diode laser absorption spectroscopy (TDLAS) can detect oxygen concentrations in-situ under these conditions as we show in this study.

TDLAS

Tunable diode laser absorption spectroscopy is the combination of in-situ absorption techniques with tunable near infrared diode lasers (NIR-DL). One main advantage of direct TDLAS is that the absorption signal can be easily evaluated in such a way that it is in principle insensitive to the

degree of optical obscuration. TDLAS is well suited for the investigation of gas samples with high particle loads. If the DC-signal is evaluated, the gas can be analysed in-situ, e.g., directly in the probe volume. completely avoiding any gas sampling, which is a common source of errors.

In contrast to many other lasers, NIR-DLs are inexpensive, compact, rugged, and relatively simple to operate at room temperature. They are particularly suitable for weight and space restricted applications. The lifetime of a diode is above 10^5h. NIR-DL output wavelengths can range from 635 to 1650 nm, although some wavelengths are not readily available, including those around 760 nm. Another disadvantage is the typically low emission power of 5 to 10 mW. Only for certain wavelengths (e.g., water at 810 nm) are there available diode lasers with 100 mW output. However, because of their fast tunability, high spectral resolution (10^{-3} nm), and spectral power density, NIR-DL offer the opportunity for nonintrusive, chemical sensors with high sensitivity, specificity. and a fast time response. TDLAS has been used in many applications to measure reliably the in-situ gas concentrations of species like CH_4 [4], H_2O [5], and O_2 [6] in challenging environments. Simultaneous multi-species detection and optical temperature measurement by two line absorption techniques has been demonstrated in power plants and waste incinerators [7, 8, 9].

THIS STUDY

An NIR-DL based spectrometer has been assembled to measure in-situ oxygen concentration inside a 28-m^3 compartment which simulates a typical flammable liquid storeroom (FLSR I) located at the NRL Chesapeake Beach Detachment (CBD) facility. Initial tests as presented here have been carried out characterizing the optical boundary conditions, including the overall transmission of the measurement path, and the variation of the oxygen concentration. This has been done for three different test scenarios important to fire suppression research: water mist only, fire only, and water mist suppressed fire. Water mist was generated using a single dual-fluid, high-pressure nozzle (HI-FOG, Marioff Oy). Test results show the potential of TDLAS as a tool for the in-situ determination of O_2 concentrations in these difficult environments.

BACKGROUND

ABSORPTION SPECTROSCOPY

The measurement principle of absorption spectroscopy is based upon a spectrally resolved measurement of the losses of the laser beam propagating through the measurement volume. These losses are recovered by wavelength tuning of the laser and are described by Lambert Beer's law:

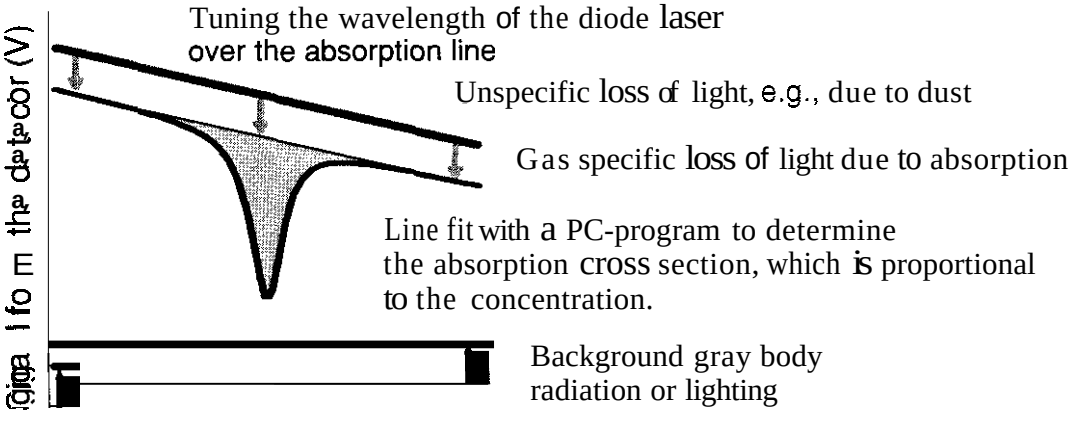
$$I(\lambda, T)=I_0(\lambda) \cdot \exp(-S(T) \cdot g(\lambda-\lambda_0) \cdot N \cdot L) = I_0(\lambda) \cdot \exp(-\alpha_m) \qquad (1)$$

where $I_0(\lambda)$ is the initial laser intensity, $I(\lambda)$ is the measured intensity after passage through an absorbing medium of thickness L characterized by S(T), the temperature dependent line strength, N the number density of absorbers, and $g(\lambda, \lambda_0)$ describing the shape of an absorption line, centred at wavelength λ_0.

In a gas cell, NIR-DL-based O_2 detection is possible with a sensitivity of 8 ppmV perm of absorption path [10]. In real world environments, sensitivity is reduced because molecular absorption is usually orders of magnitude smaller than the disturbances that interfere with the

signal. Wavelength unspecific absorption leads to time dependent variations in the optical transmission of the measurement path. The NIR oxygen transition at 760 nm is quite weak, and an absorption path of more than a meter is typically required to achieve a reasonable detection limit. The poor visibility environment of a fire test chamber produces variations in background absorbances 100 times larger than the molecular absorption signal of oxygen. Scattering, refractive index gradients, thermal lensing, mechanical vibrations, and deformation of the alignment will occur to some extent and affect the beam profile, the pointing stability, and will lead to light loss. The detector signal can be increased by background radiation from the fire. Problems can be caused by electromagnetic pickup, high voltage/current switching, ground loops, and thermal drift in the laser power supply, detector or signal processing electronics. Techniques have been developed to compensate for or avoid most of these problems.

The ability to follow transmission fluctuations by high-speed wavelength tuning achieved by simple modulation of the laser current is one of the major advantages of diode lasers. Repetition rates in the kHz range allow acquisition of an absorption profile in less than a millisecond, which is sufficiently fast to "freeze" all transmission variations within an absorption scan, making correction by division with a background transmission signal possible. Background radiation can be minimized by the use of narrowband optical filters. Additional noise reduction is achieved by averaging consecutive absorption profiles and extracting the absorption signal, i.e., the area underneath the absorption line, by a fitting algorithm. This absorption cross section is proportional to the concentration of the absorber, in this case the oxygen molecules (Figure 1). If the temperature change in the measurement region is large (>ca. 30 **K)** the temperature dependence of the observed absorption line has to be considered. It is therefore important to select a suitable rotational line and to know its properties.



O_2 SPECTRUM

The spectrum of molecular oxygen shows several well defined bands (A, B, y, δ) in the near infrared that belong to vibrational suhtransitions of the $b^1\Sigma^+_g \leftarrow X^3\Sigma^-_g$ electronic transition. The region of strongest absorption is found in the A-band ($0 \leftarrow 0$) that extends from 759 to 770 nm. The spectrum consists of quite regularly spaced, rotationally resolved lines (FWHM **3** GHz at 300 **K,** latm), which belong to magnetic dipole transitions with a typical line strength of about

$2 \cdot 10^{-4}$ cm^{-1} atm^{-1}. Due to its overlap with the emission region of AlGaAs diode lasers. the A-band was one of the first spectral regions to be used for quantitative detection with diode lasers [11]. Inexpensive Fabry-Perot-type lasers require a tedious selection procedure for coincidences with absorption lines due to their discontinuous tuning behaviour. Continuously tunable, distributed feedback diode lasers (DFB-DL) are more suitable for obtaining spectra. For O_2, all lines within the R-branch of the A-hand are generally accessible using a DFB-DL. Temperature dependence of the line strengths can he minimized by appropriate choice of the absorption line.

Comparison of the TDLAS measured spectrum of oxygen in air using a DFB-DL and a multipass cell of the Herriot type [12] with a Hitran calculation are shown in Figure *2*. The absorptions were calculated with a LabVIEW® (National Instruments Corp.) program using the 1996 HITRAN database (Ontar Corp.) with good agreement to the measurement. The long path length (1.5 m) Herriot cell provided sufficient absorption and a better signal to noise ratio.

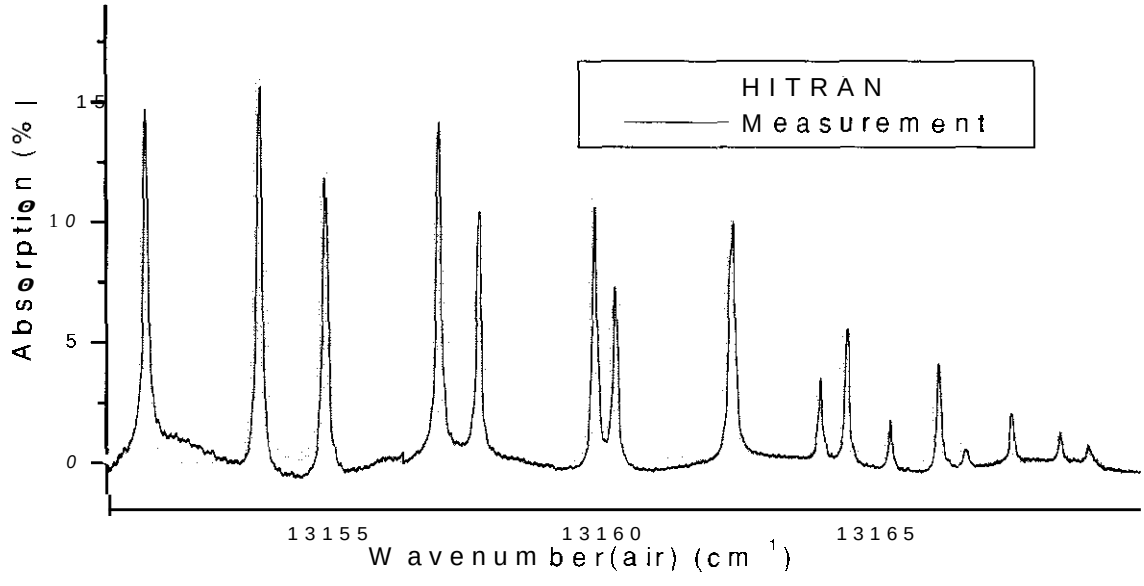


Figure 2. Spectrum of O_2 in air measured in a Herriot cell with TDLAS and calculation using the HITRAN database for a 1.5-m absorption path length at 296 K.

HERRIOT CELL

A Herriot cell consists of two concave mirrors. The laser beam enters through an off-axis hole in one mirror and is reflected several times between the mirrors resulting in an elliptical spot pattern. It leaves through the same hole with a slightly different angle. This optical multipass arrangement provides long effective path lengths in a small volume. To determine the optimum path and base lengths and minimal interferences for the Herriot cell, different spot patterns were calculated [13]. Since for our application the number of spots will be low compared to other applications of the Herriot cell, e.g., in trace gas analysis, fringes due to beam overlap are more easily avoided. A White cell. very useful for incoherent light sources, generates interferences because of overlapping beams in the optical layout that disturb a higli-resolution absorption measurement. The Herriot setup is surprisingly stable and has been used to successfully measure

concentrations of SO_2 with mid infrared diode lasers in the harsh environment of an aircraft exhaust [14].

DIODE LASERS

Two types of lasers were used for the present experiments: Fabry-Perot diode lasers (FP-DL), (Mitsubishi ML4405) and distributed feedback diode lasers (DFB-DL), (Sensors Unlimited, SU 761-CD, optical power up to 10 mW). Both types can be tuned slowly by temperature over several nanometers or rapidly by current over some tenth of a nm. Fabry-Perot lasers designed to emit in a single mode are more easily produced than DFB-DL; however, they have the disadvantage of showing mode hops, i.e., they have gaps in their spectral emission and can only be used to measure absorption lines that lie completely in one of the continuously tunable sections.

The larger graph in Figure 3 shows the emitted wavelength of an FP-DL in nm versus time (a.u.) while being modulated with two different amplitudes. The emitted wavelength was measured with a Burleigh wavemeter (WA-1500) and saved to a computer via RS232. The laser shows mode hops, i.e., sudden changes in the emitted wavelength, at the same positions when modulated too hard whereas the laser power (not shown) oscillates in a triangular function without showing any sign of the mode hops. The emitted wavelength versus the injection current (mode map) is shown in the smaller graph.

Unlike FP-DL lasers, which need to be selected for the individual absorption line to be detected, DFB-DL lasers are tunable over a wide spectral range without showing any mode hops. DFB-DL can be rapidly (>10^5 GHz/s) tuned over 30 to 100 GHz without mode hops and *are* generally preferable.

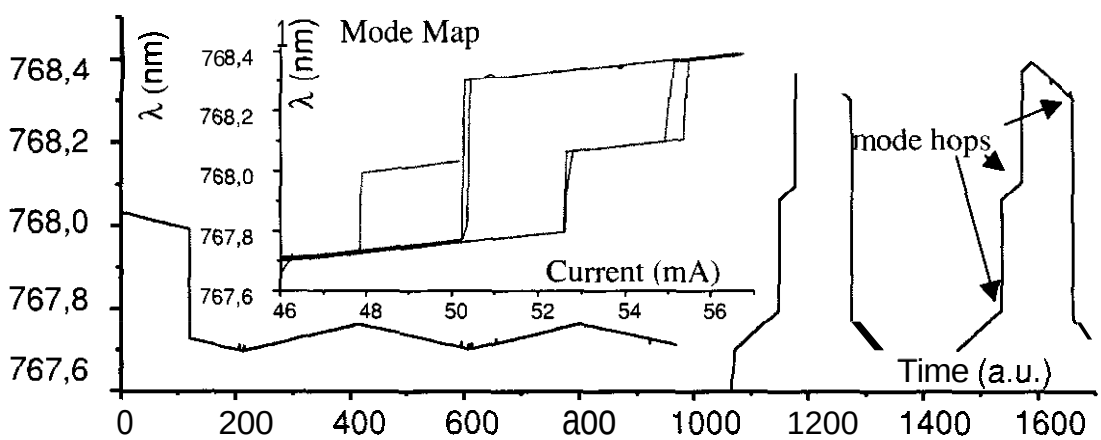


Figure **3.** Current modulation of a Fabry-Perot diode laser (Mitsubishi ML4405) showing mode hops.

EXPERIMENTAL APPARATUS AND RESEARCH APPROACH

TESTS AT FLSR I

A simple setup with few optical components was chosen for all tests to minimize the intensity of interferences that increases with the number of transits through optical surfaces which the laser

beam passes. The laser and detector as well as some of the electronics were located inside the test compartment. The first tests were designed to determine the amount of obscuration and frequency components for unsuppressed fire and water suppressed fire. For these tests we employed a FP-DL in order to protect the more expensive DFB-DL. The beam of a FP-DL (Mitsubishi ML4405) driven by a laser diode controller (ILX LDC3714B) was directed from one side of the compartment to a mirror at the other side and reflected back to a silicon photodiode next to the diode laser. **A** narrowband interference filter was used to block light from the fire and background light. The pathlength was varied according to the expected differences in the conditions during the tests. For the water mist test, a DFB-DL (SU 761-CD) replaced the FP-DL.

To protect the sensitive electronic equipment from the water mist. droplets, condensation, soot and heat, three nitrogen purged boxes were built, one each for the diode laser, the laser driver, and the photodiode detector with interference filter, amplifier, and power supply. The signal generator **(SRS** DS345), which provides the 1kHz modulation signal for the laser driver, was located outside of the compartment, close to the laser controller. Detector signals were amplified and digitized with 1750 samples per scan at 2.5 Msample/s by a four channel 14 Bit A/D (Datel 416P). The data collection computer was inside the MCR (mobile control room) located 10 m from the test compartment. Successive absorption profiles could be averaged to increase resolution and saved to disk for evaluation after the tests.

DATA REDUCTION AND EVALUATION

The signal consisted of the modulated laser intensity upon which any absorption due to oxygen was superimposed. The data evaluation mathematically separates those two features using a linear line fit, reducing the digitized absorption line profile to only a few parameters. The absorption profiles were saved in a binary format during the tests and were later read by a LabVIEW program. An offset of the signal was subtracted at very high amplification when the dark current of the diode needed to be corrected. The position of the offset was verified during the experiment by modulating the diode laser over a current range partially below the threshold. Thus the laser diode is practically turned off on every cycle. **A** Lorentzian line shape function was fitted to the data assuming a quadratic background polynomial. The asymmetry of the line shape caused by the amplitude modulation of the laser was taken into account by the program without changing the actual absorption cross section. The line drift of the laser was followed by automatically optimizing the center of the line fit position within a manually applied window. Too large of a window led to erroneous fits because the true line position would not always be found by the automated fit. The use of a reference cell would guarantee detection of the line center. This capability is planned. The line width was determined automatically for different times of the test, but kept constant for the line fit for the different sections of the test to keep the number of free parameters low. It was neither observed nor expected that the line width would change during a short time scale. The line width was adjusted manually when necessary.

The transmission independent line strength was derived from the fitted absorption line after dividing the transmission dependent absorption cross section by the fitted background. The line position, the linewidth, the three coefficients for the background, and the mean square error were saved, as well as the time of the measurement, reducing about 1000 samples of each scan to 8 values. The line strength that resulted from the line fit was set to the initial value of 20.9% oxygen. The transmission was derived from the first coefficient of the background. The unified

transmission (in % after a path of Im) was calculated from the absorption coefficient of a broad banded absorber (e.g., mist in the absorption path) assuming the Lambert Beer law.

HERRIOT CELL

For the first version of the Herriot cell, two 50-mm diameter aluminum coated concave mirrors with radius of curvature of 600 mm were used. Gold-coated mirrors will be used in future cells since aluminum has a lower reflectance. A 5-mm hole was cut in one of the mirrors 5 mm from the mirror rim using 400 μ SiC paste inside a stainless steel tube "drill bit" and a drill press. The mirrors were placed in kinematic holders. The solid mirror holder was movable along a rail to allow different separations of the mirrors (base length) and thus different patterns and path-lengths. The Herriot cell provided a 1.47-m long absorption path while maintaining a base length of only 0.35 m. The oxygen spectrum shown in Figure 2 was taken with this cell. For this spectrum, the DFB-DL was tuned by temperature varying from 285 to 306 K. We are in the process of preparing the Herriot cell for the field tests.

RESULTS AND DISCUSSION

FIRE TEST (NO SUPPRESSION)

A methanol/heptane 0.09-m^2 (1 x 1-ft) pan Fire was used for the fire test in FLSR I. There was enough fuel to bum for about 6 min. No fire suppression was used for this test, and O_2 depletion did not cause extinguishment. The absorption path was located 0.85 m above the floor and 1 to 1.5 m from the fire. TDLAS results using the FP-DL are shown in Figure 4. As this figure shows, FP-DL can be used to successfully detect O_2 concentration even though the number of absorption lines that can be observed is limited.

Data was recorded at three absorption profiles per second (scans/s) with no averages. After the extraction of the O_2 concentration, five adjacent concentration values were averaged by a moving linear average. A tiny pressure peak is visible at the time of ignition since the data have not been corrected for pressure. An almost linear drop in oxygen concentration down to 12.6% was deected after ignition. The data acquisition program stopped during the test for 6 min due to an incorrect configuration setting and had to be restarted. Shortly after the hiatus began the fire burned out as recorded by a monitor video camera. Since the compartment was closed, the O_2 concentration stayed at 12.7% (±0.25%) once the fire was out until the ventilation was started. The increase in oxygen concentration back to its original value can be seen in Figure 4. The signal noise is higher than at the beginning of the test due to smoke inside the compartment and vibrations from the ventilation fans. Fan vibrations tended to move the laser beam from its optimal position on the photodiode.

The gas temperature during the test at the measurement location changed from 290 to 328 K (maximum) and dropped back to 298 K at the end of the test. The transmission for the **4.3** m path changed during the test from 100%down to **1.64%** when the fire burned out. To compare this value to the results from tests involving different absorption lengths the transmission value is converted to a standard distance of one meter. The minimum transmission for the unsuppressed fire test was 38% (1 m).s

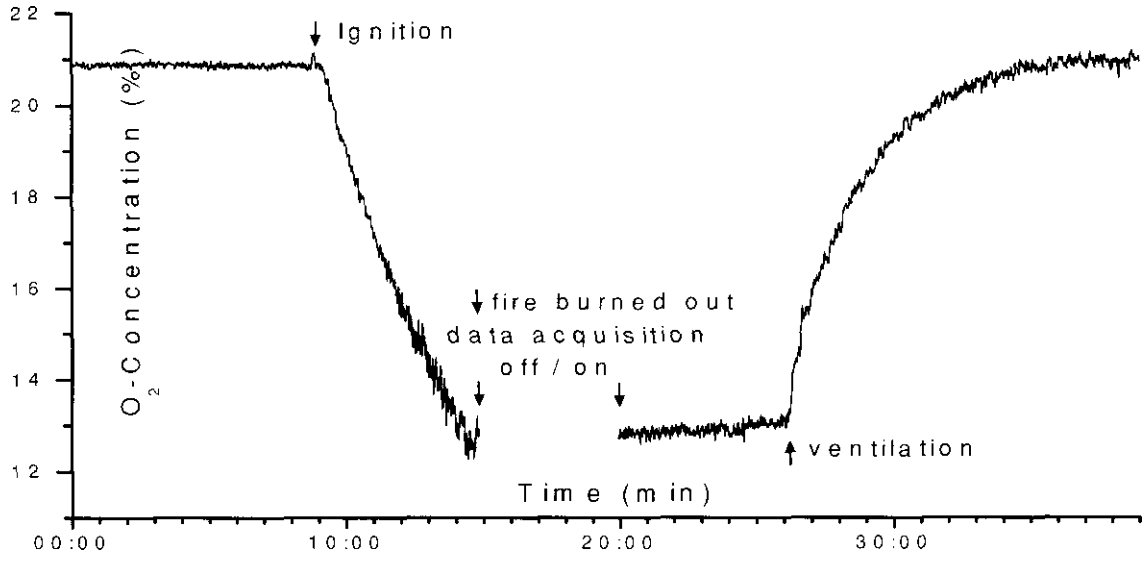


Figure 4. TDLAS measured O_2 concentration during a fire test with a 4.3-m absorption path length.

WATER MIST (NO FIRE)

Water mist was generated inside FLSR I with a nitrogen propelled, dual-fluid, single nozzle system (HI-FOG, Marioff Oy). Pressurized nitrogen forces both nitrogen and water through the nozzle resulting in a very fine mist. The measured (VisiSizer, Oxford Laser, Inc.) mean droplet diameter in the room at a height of 1 m was 10 µm and the Sauter mean was 20 µm. The mist system is started manually and stops when the water and nitrogen run out.

Unlike the fire test results, the laser beam obscuration by the water mist is much larger. To respond to this. the test was repeated with improved dynamic range, faster data acquisition, and higher averaging (100 individual absorption profiles averaged, giving a time resolution of 1.6 Hz). The transmission, shown in Figure 5, changes through four decades. After the start of the water discharge, the relative transmission drops to 0.02% (0.6%[1 m] ± 0.1% [1 m]). The transmission increases to almost 100% following opening the door, as indicated in Figure 5. The noise is reduced after the ventilation fans stop (and thus the vibrations); the nitrogen discharge is turned off (thus ending the mist production).

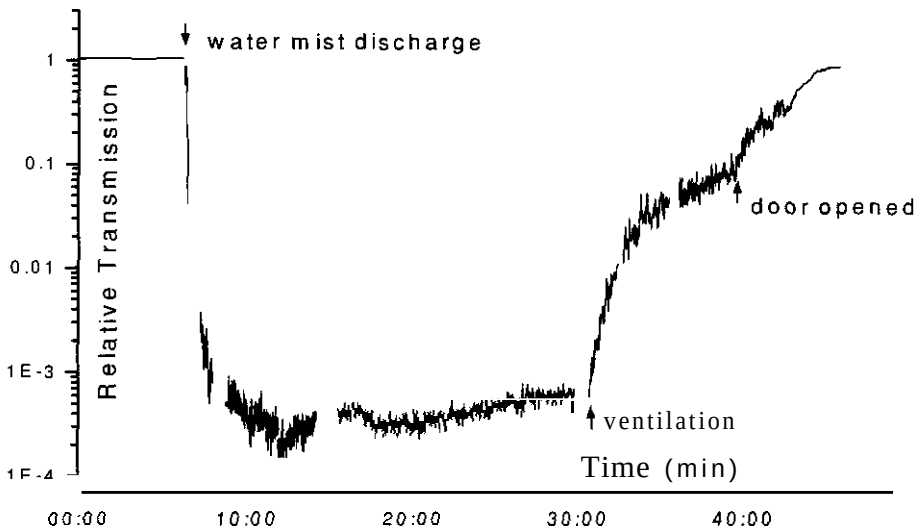


Figure 5. Measured obscuration at 760 nm by water mist for **a** 1.65-m absorption path during a water mist discharge in FLSR **I.**

The concentration profile of O_2 during the discharge is shown in Figure 6. The evaluation of the oxygen concentration shows some gaps because the amplification of the photodiode signal was manually changed attempting to optimize the dynamic range. This will be automated in the future. Increased digitization noise can be noted in some regions due to a less than optimized detector amplifier gain setting. It appears that the oxygen concentration is about 2% lower during the water discharge. This is expected from the displacement of O_2 by N_2 (discharged with the water mist) and evaporated water drops. Obscuration and increased noise made it difficult to see small changes under the conditions of this test. However, these test results are very promising. In spite of the high obscuration. it was possible to extract the oxygen concentration for all conditions throughout the test.

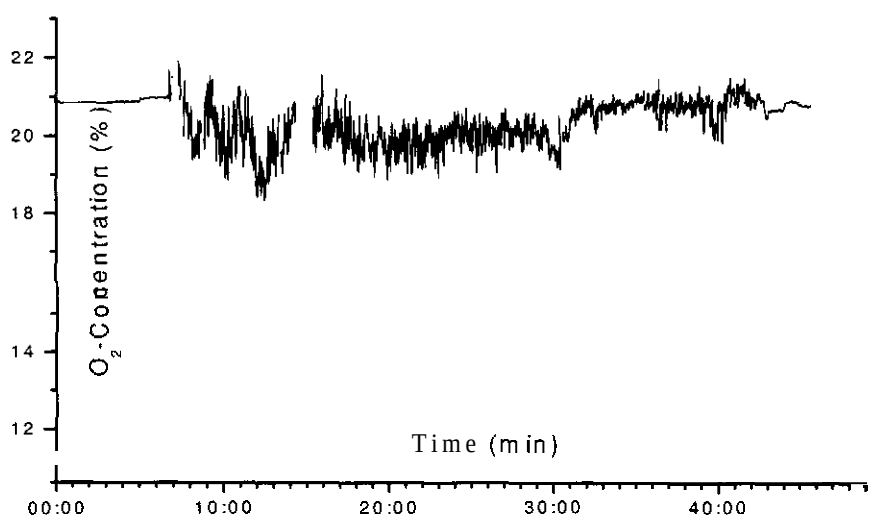


Figure *6.* NIR-DL measured oxygen concentration in a 1.65-m absorption path during water mist as described in Figure *5.*

WATER MIST SUPPRESSED FIRE TEST

O_2 concentrations for the suppression of a methanol/heptane pan fire with water mist wcre measured at one detector amplifier setting and with (1–100) averages. During this test the laser light went directly into the photodiode giving *a* pathlength of 1m.

The relative transmission shown in Figure 7 starts at 100% (2 min before water discharge) and drops during the fire to about 74% (1 m) when water mist discharge began. At this time the number of averages was increased. During the fire suppression the transmission was about 48%. The fire was extinguished before it ran out of fuel. The transmission dropped further to –0.6% (1 m) (±0.1% [1 m]) after the fire was successfully suppressed (5:30 min after start of water discharge). The water discharge lasted approximately 10 min. When the ventilation was started (15 min past water discharge), the transmission came back up. The O_2 concentration initially dropped to 17% (±1%), at the time of the water discharge, then rose back to 21% after the ventilation was turned on.

CONCLUSIONS AND FUTURE WORK

We have successfully performed the first TDLAS-based in-situ detection of oxygen concentrations at 760 nm in the NRL flammable liquid store room (FLSR I) at Chesapeake Bay Detachment. The test conditions included methanol/heptane pan fire, discharge of fine water mist, and pan fire suppressed by water mist. Despite high obscuration during the injection of water mist, these tests show that it is possible to measure in-situ absolute oxygen concentrations with a time resolution of 1.6 **Hz** using TDLAS throughout an entire water mist suppressed fire test. The measured concentration profiles ranged between 12 and 21% for the unsuppressed fire test and 17 and 21% for the suppressed fire test, and 18 to 21% for the water mist discharge. The transmission during the tests varies over several decades. During water mist discharge, as little as 0.02% of the emitted laser light reached the detector.

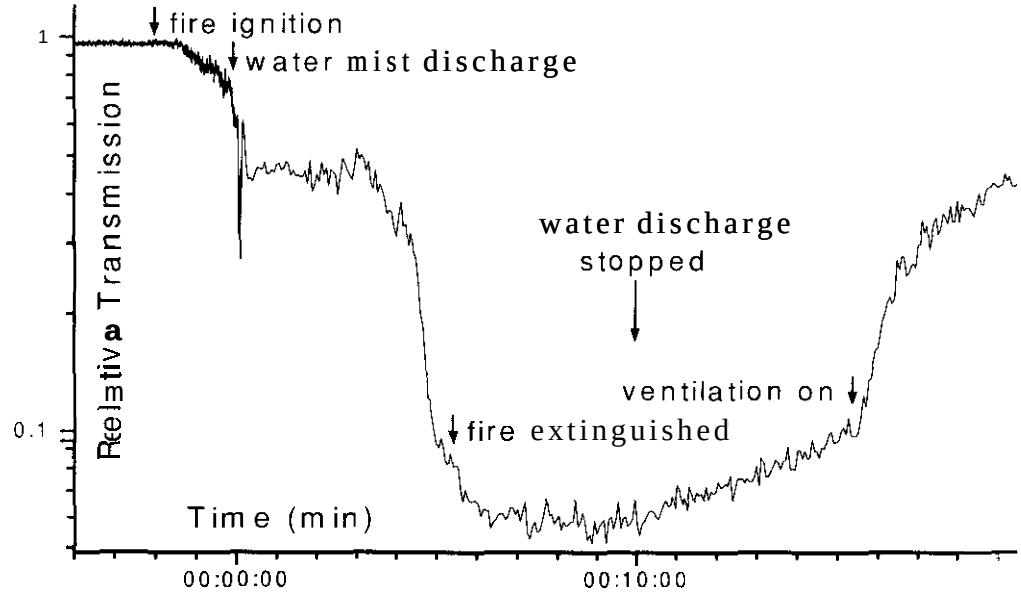


Figure 7. NIR-DL measured relative transmission using a 1.O-m absorption path length during a water mist suppressed fire.

Based on the present results, it would not be advisable to use fiber optics for more remote location of the laser and other components. We have shown that these items can be successfully protected from the hostile environment. The use of fibers would cause additional light loss and possibly create another source for interferences. It might be attractive to reconsider this option following further noise reduction improvements in the experiment.

Tests are in progress using a Herriot optical setup with reduced physical dimensions. Long path lengths achievable with the Herriot cell are desirable in order to get higher absorption and spatial resolution. However, light loss due to scattering will increase significantly with the longer pathlength. At the same time more background noise is expected. For the tests using fine water mist, an optimal path length should be between 1 and 2 m. The pathlength can be extended to between 4 and 10 m for the fire test without suppression.

Future work will include automating the detector amplifier gain settings. Also, the maximum speed of the data acquisition and storage to disk will be increased from the current 40 scans/s for no averages and 2.5 scans/s for 100 averages by optimizing the LabVIEW data collection program. These modifications will reduce noise and improve the signal quality.

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