2006 HOTWC

Chapter Title:

## Instrumentation for Bench- and Large-scale Test Fixtures

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Project working title:

## 3.C - Laser-Based Instrumentation for Real-Time, In-Situ Measurements of Combustible Gases, Combustion By-Products, & Suppression Concentrations

Kevin McNesby, Erik Johnsson, George Mulholland, Reed Skaggs, Andrzej Miziolek, Edwin Lancaster, Robert Daniel, Bill Bolt, Craig Herud, Brian Kennedy





Working Description:

## OPTICAL SPECTROSCOPIC TECHNIQUES FOR ARMY APPLICATIONS

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- joint, effort at ARL (Army Research Laboratory), ATC (Aberdeen Test Center), NIST Using Optical Spectroscopy
- •(FY97-02)
- work focussed on needs pertaining to armored vehicles
- field-scale to laboratory scale environment

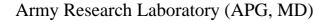




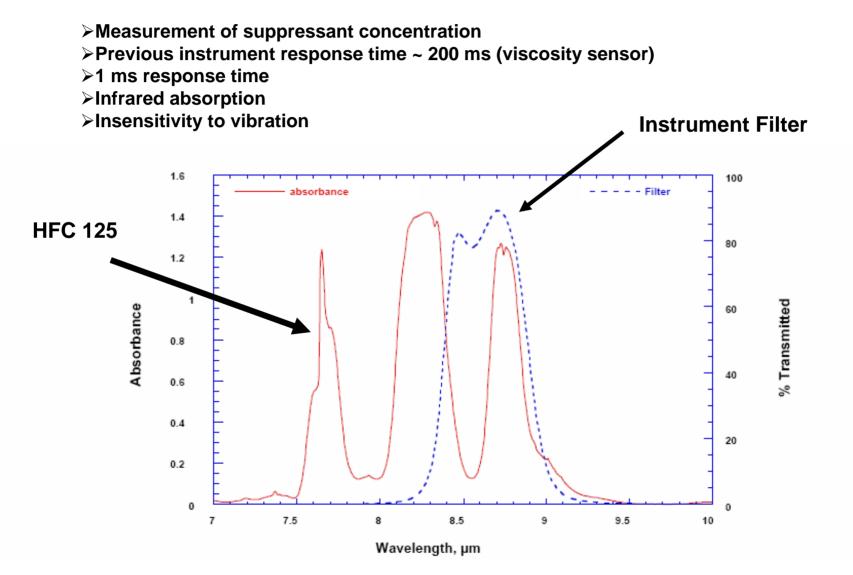
# **Talk Outline**

- Introduction Tanks
- Species (Suppressants; Toxic By-products; Fuels; Oxidizers)
- Fast Vibrational/Visible Spectroscopy
- Differential Infrared Rapid Agent Concentration Sensor (DIRRACS)
- Laser Induced Breakdown Spectroscopy for Fire and Explosive Suppressant Measurement
- Diode Laser Spectroscopy for Toxic and Combustible Gas Measurement

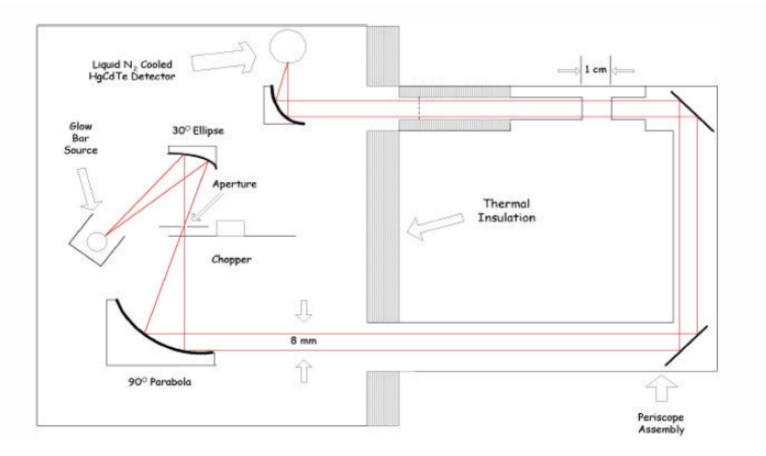




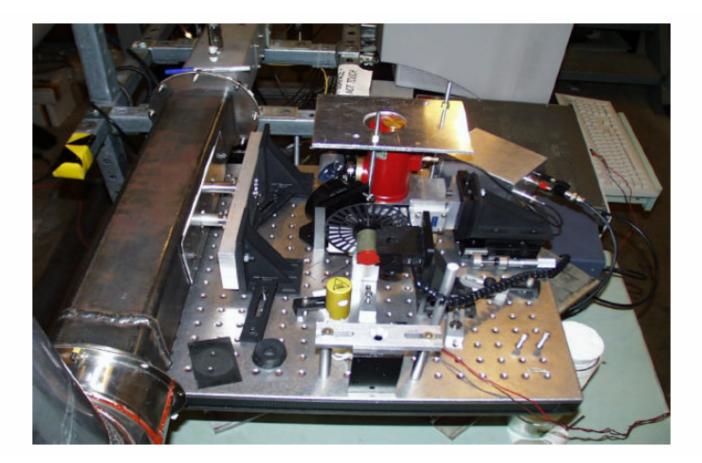
## Differential Infrared Rapid Agent Concentration Sensor (DIRRACS)



## **DIRRACS II Schematic**

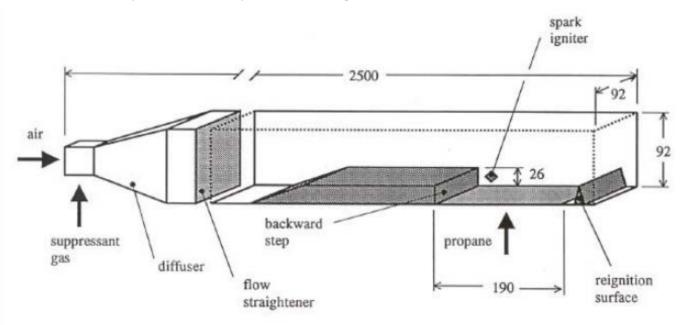


## **DIRRACS II Instrument**



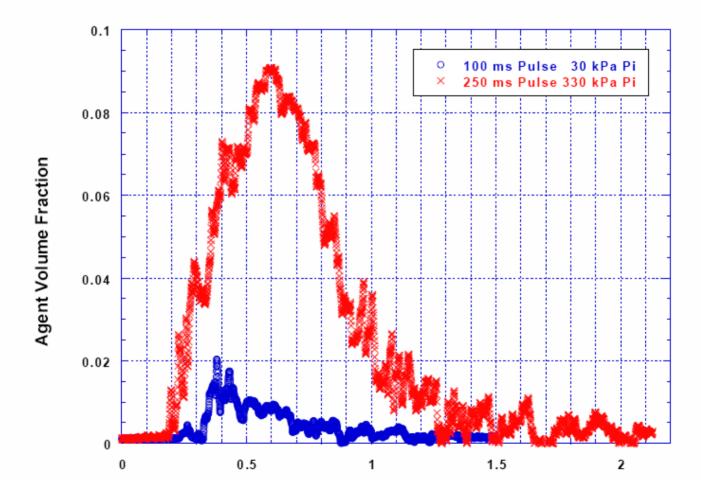
## **DIRRACS II Test Rig**

## Transient Application Recirculating Pool Fire (TARPF) Facility at NIST.



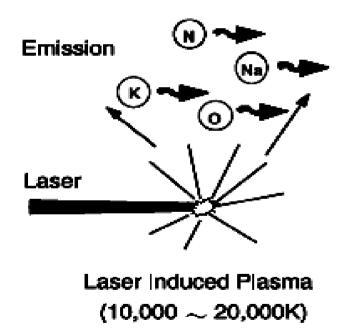
## **DIRRACS** Testing

Time Response < 10 ms Sensitivity < .005 volume fraction



HFC 125 agent volume fraction versus time for two releases of HFC-125 in the TARPF facility.

# Suppressant Gas Measurement: Laser Induced Breakdown Spectroscopy (LIBS)



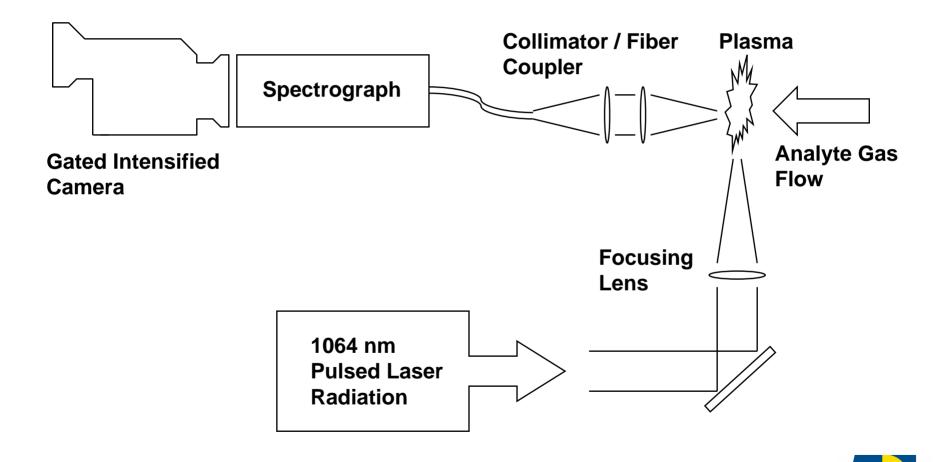
**Plasma Formation** 

- Multiphoton Absorption --> Ionization
- Absorption of Laser Radiation by Free Electrons, i.e. Inverse Brehmstrahlung
- Electron Collisions --> Ionization --> Heating --> Breakdown
- Typical Gas Temperatures, ca. 20,000 K

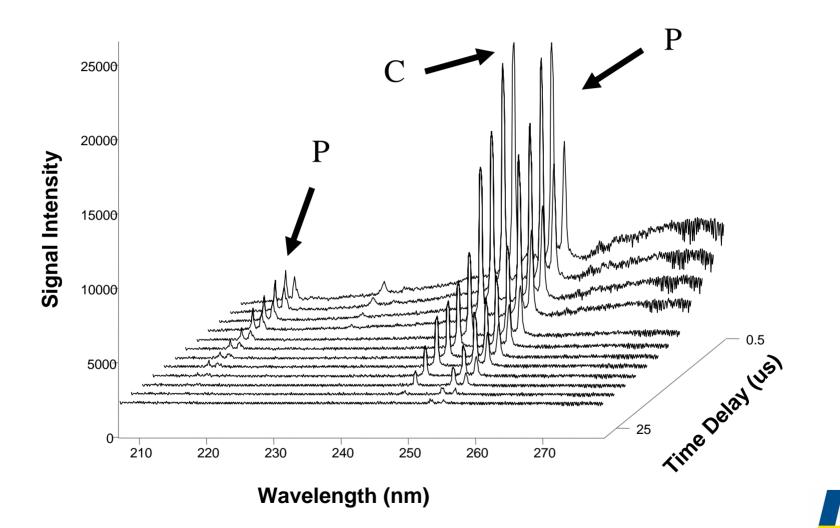
Spectrochemical Analysis based on Collection of Emission of Atomic and Molecular Constituents Usually after Plasma Continuum Radiation Decays (2-100 usec).

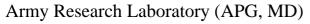


# **LIBS Instrumentation**

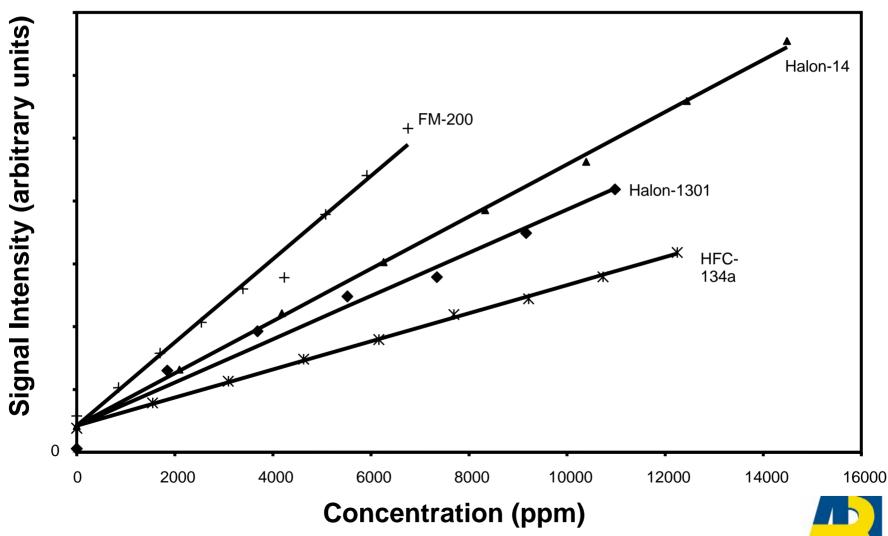


# LIBS of DMMP





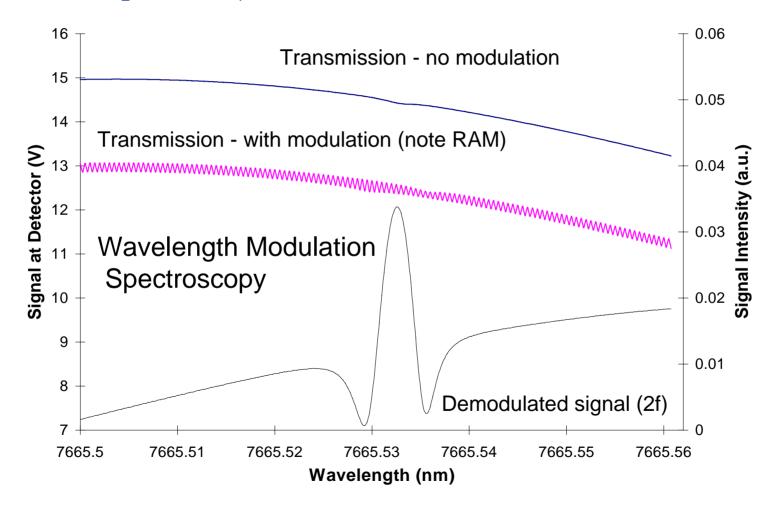
# **LIBS of Fire Suppressants**



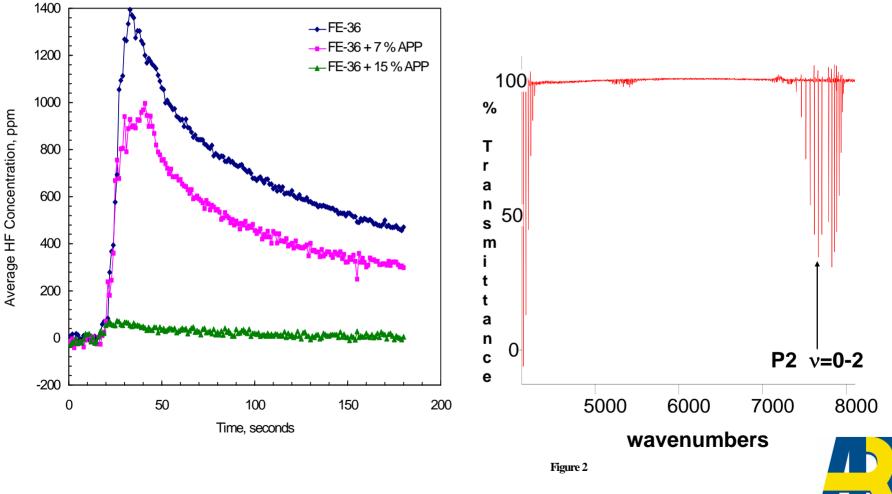
Army Research Laboratory (APG, MD)

#### Toxic and Combustible Gas Measurement: Near Infrared Tunable Diode Laser (TDL) Spectroscopy

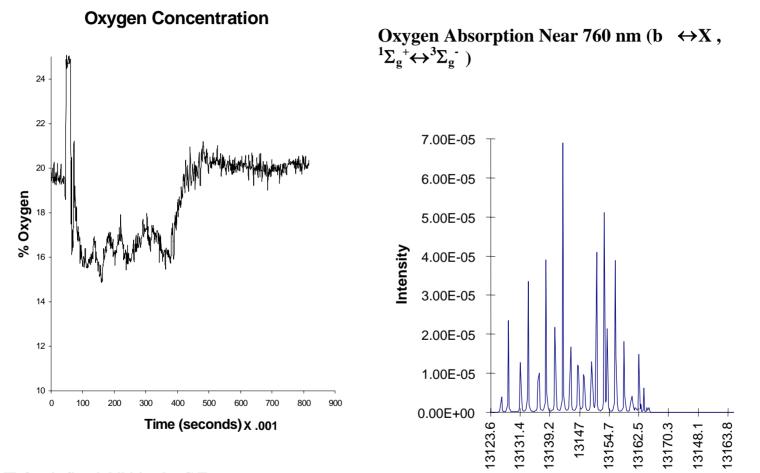
HF, O<sub>2</sub>, CO, CH<sub>4</sub>, HCI, other small molecules



# HF Gas Measurement During Suppressant Testing



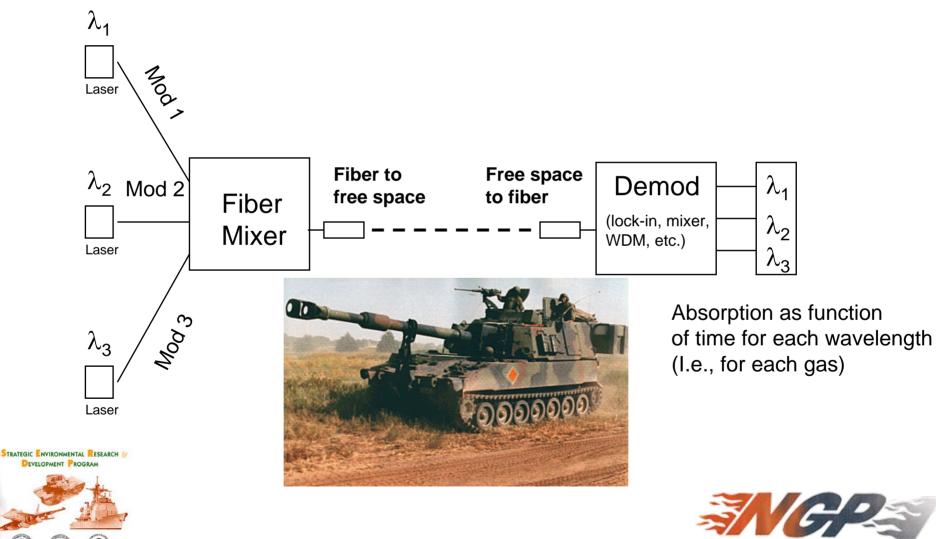
# **Oxygen Measurement Using TDL Spectroscopy**



JP-8 - air fire, inhibition by  $C_3F_8$ , crew compartment test fixture, fire extinguished 150 ms after detection.

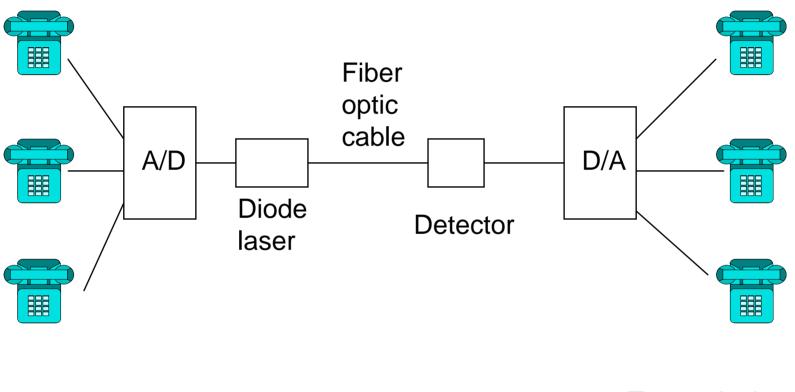
Wavenumbers (cm-1)

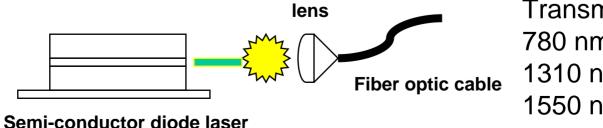
#### Multiple Species Gas Sensing Using Tunable Diode Lasers (TDL)



NEXT GENERATION FIRE SUPPRESSION TECHNOLOGY PROGRAM

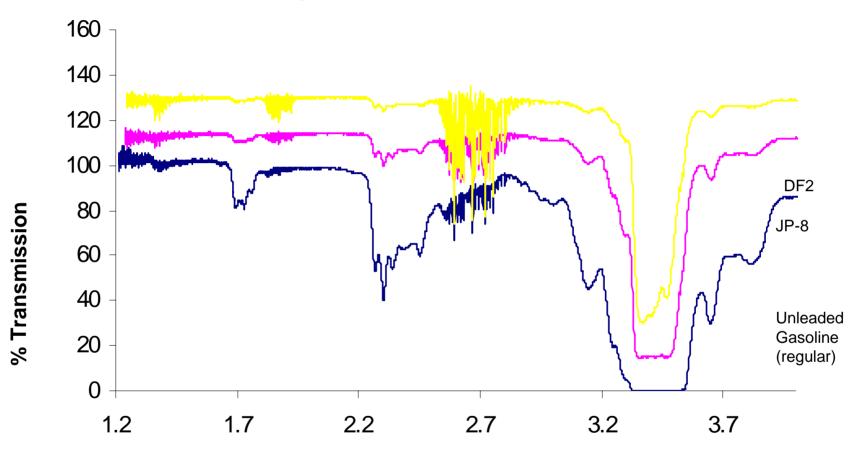
#### **Time Division Multiplexing**





Transmits best at: 780 nm 1310 nm 1550 nm

## Detection and Measurement of Middle Distillate Fuel Vapors Using Tunable Diode Lasers



#### Wavelength (micrometers)

STRATEGIC ENVIRONMENTAL RESEARCH & DEVELOPMENT PROGRAM

Spectrum of dry air saturated at 294K with vapor from unleaded gasoline, JP-8, DF2. Spectra offset for clarity.



#### Limitations of TDL Spectroscopy For Measurement of High Molecular Weight Vapors

- $\bigstar$  Absorption features are unstructured.
- Can't use traditional wavelength or frequency modulation techniques to measure big molecules (e.g., middle distillate fuels - JP-8, DF-2, etc.). Unable to scan on and off resonance with single DFB laser.

Develop a near-infrared diode laser-based sensor capable of measuring hydrocarbon fuel vapor concentrations with a time resolution
of 10 msec per measurement point; maintain S/N advantages of WMS.



#### **Measurement Technique**



uses laser diode absorption spectroscopy in the near-infrared spectrum (1.3 microns and 1.71 microns)



Emission intensity from two lasers is varied sinusoidally, with emission from first laser 180 degrees out of phase with emission from second laser.



Measurement is made in situ.

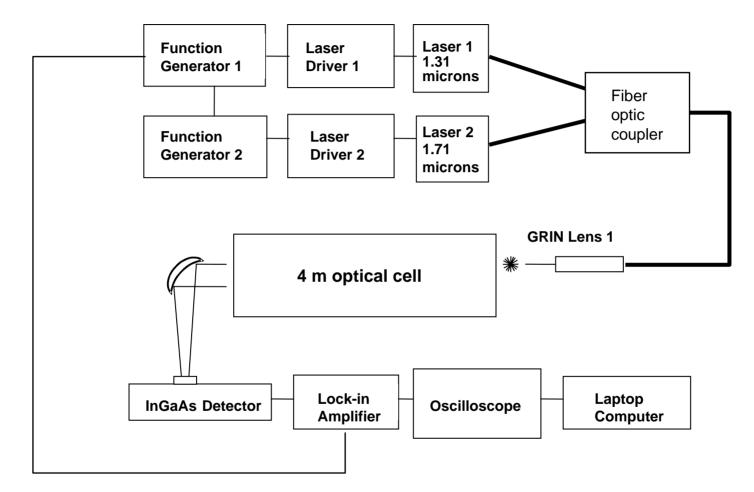


Phase sensitive detection is used to measure differential absorption at the two laser emission wavelengths.



10 msec response time

#### Laser Mixing Apparatus



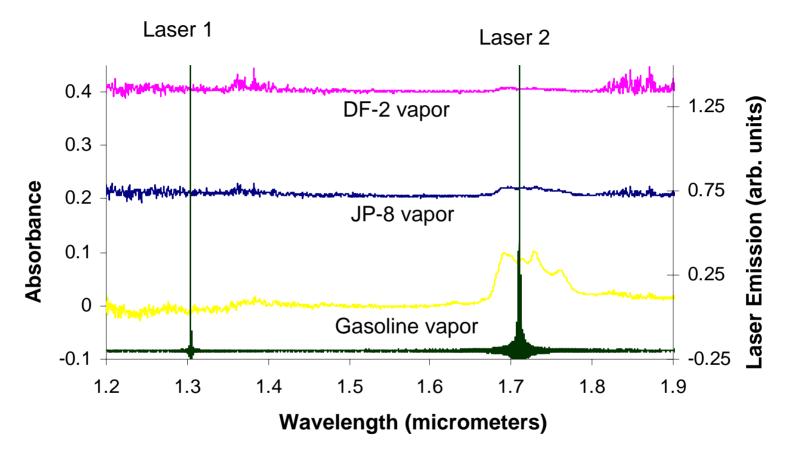


**Figure 3:** The experimental apparatus used to measure vapors from middle distillate fuels.





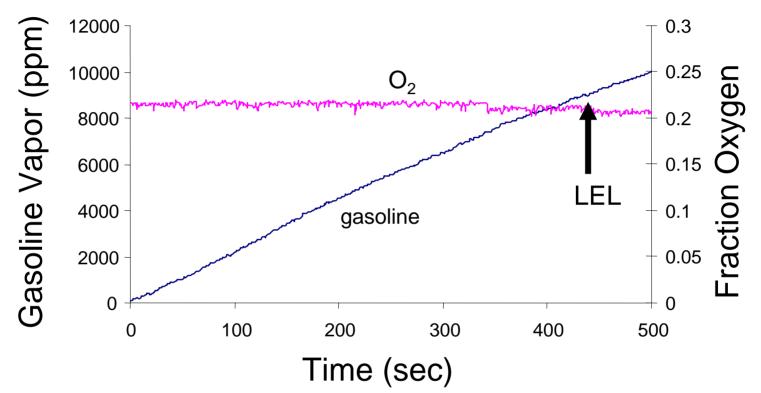
## Overlap of Fuel Absorbance Spectra and Mixed Laser Probe Beam





**Figure 4:** shows the vapor phase absorption spectrum of air saturated by vapor at 294K from JP-8, DF-2, and gasoline between wavelength values of 1.3 and 1.75 micrometers superimposed upon the emission from the optical fiber which carries the mixed wavelength probe beam.

#### Results: Approach to Lower Explosion Limit (LEL) in a Gasoline Fuel Tank at Room Temperature

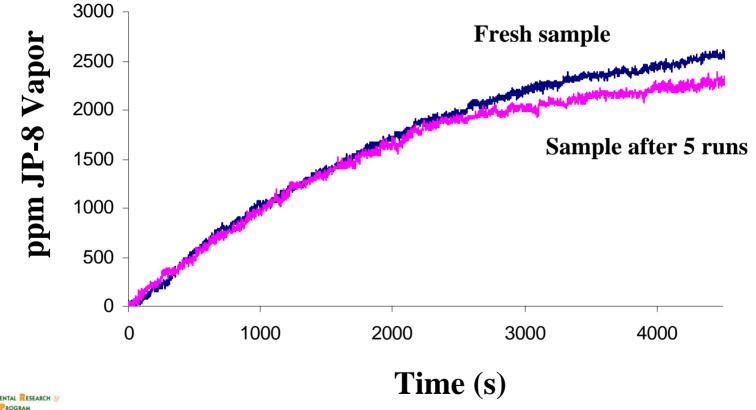


Simultaneous measurement of fuel/oxygen concentration (by volume) during displacement of contents (dry air) of a 14 liter vessel by air saturated by gasoline vapor at 294K and 1 atmosphere total pressure. Oxygen sensor courtesy of Oxigraf, Inc.





#### Results: "Aging" of JP-8 Detected Using Mixed Laser Sensor







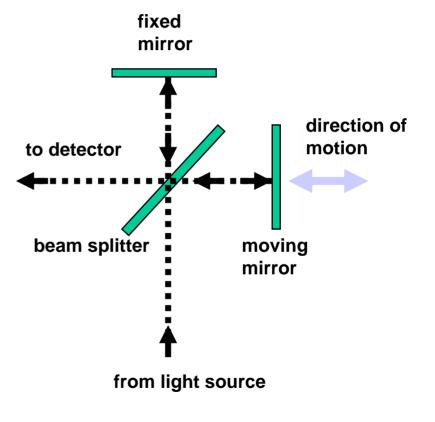
For a monochromatic source:

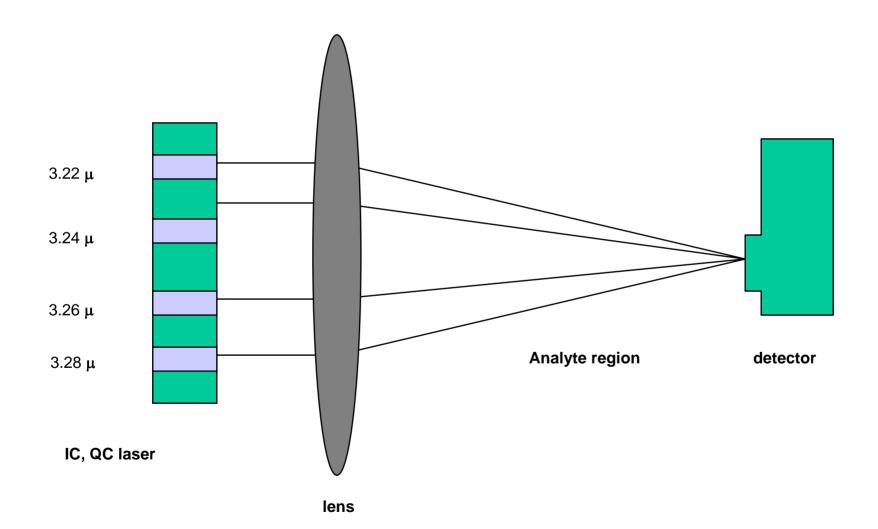
$$I(\sigma) = I(v)\cos 2\pi v \sigma$$

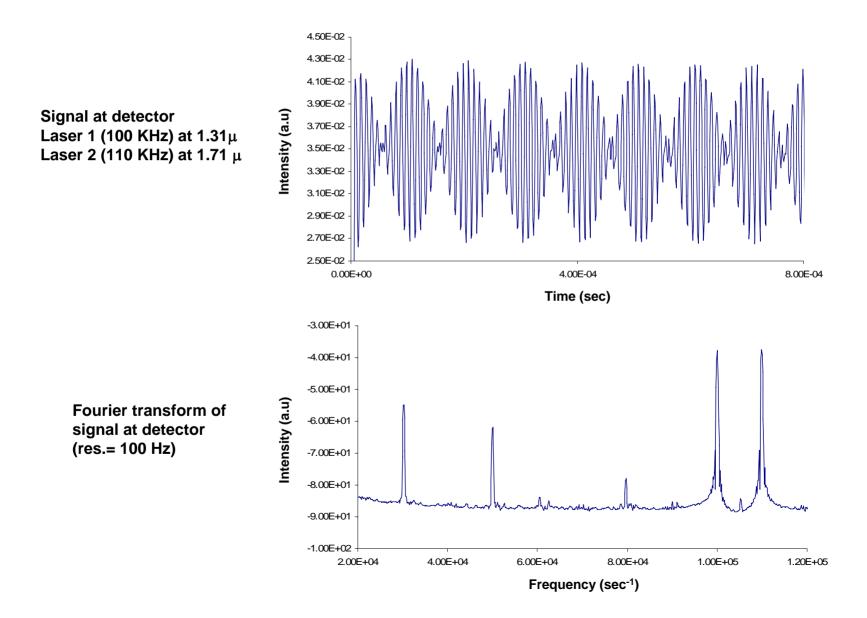
For a polychromatic source:

$$I(\sigma) = \int_{0}^{\infty} I(v) \cos 2\pi v \sigma d v$$

 $\sigma$ = mirror position  $\nu$  = light frequency (cm<sup>-1</sup>)  $I(\sigma)$  = intensity at detector  $I(\nu)$  = source intensity







1.10E+00 **Fraction Transmitted** 1.05E+00 1.00E+00 9.50E-01 9.00E-01 8.50E-01 8.00E-01 9.90E+04 1.03E+05 1.07E+05 1.11E+05 1.15E+05 9.50E+04 Frequency (sec<sup>-1</sup>) 0.09 0.08 LEL 0.07 Absorbance 0.06 0.05 0.04 0.03 0.02 0.01 -0 500 1000 1500 0 2000 2500 Time (sec)

Absorption of laser radiation at  $1.71\mu$  as air in 2m cell is displaced by air saturated with gasoline vapor.

## **Conclusions/Successes/Failures**

Project greatly enhanced knowledge of fire suppression on board combat vehicles.

>Laser diode systems fielded at ATC/ARL for HF and  $O_2$ .

≻DIRRACS II testing at WPAFB.

≻FTLS work continues.

➢JP-8 sensor vibration problems

>COF<sub>2</sub> formation documented

## **Conclusions/Successes/Failures (cont.)**

Lasers outside communication bands still very expensive

≻Mid-IR RT CW lasers still unreliable

Narrow BW, broadly tunable, fast light sources still unavailable

## **Publications**

9 open literature, 1 book chapter, 11 Gov't Tech Reports

### **Acknowledgements**



Dick Gann, NIST



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Lawrence Ash, NAWC



Oxigraph, Inc.