“-CARS and LIF Studies of Non-Equilibrium Nsec Pulsed Molecular Plasmas”

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A Bit of Motivation

Brief Introduction to Coherent Anti-Stokes Raman Scattering (CARS) and OH and Two Photon LIF.

Measurements in Non-Equilibrium Nsec Pulsed Plasmas.
   i. H₂/Air PAC Model Validation and Non-Thermal Ignition
   ii. Vibrational Energy Dynamics.
   iii. NO, N, and O Measurements.

Some Initial Thomson Scattering and Psec E-field Measurements.

Conclusions and Acknowledgements.
Motivation

Low T ($T_g \sim 300–800$ K, $T_e \sim 1–2$ eV), High P ($\sim 0.1 – 1$ Bar) Air and “Air Like” NEQ plasmas have shown promise for a variety of applications including aerodynamic flow control, plasma combustion, and plasma medicine (jets).

NS Surface DBD - 45 m/sec (thermal interaction)
Little et al, AIAA J., 2012

Effect of $O_2 (a^1 \Delta_g)$ on flame propagation speed
(T. Ombrello, AFRL – WPAFB)

But Understanding of Underlying Kinetics / Energy Transfer is Often Lacking.
The CARS process can be thought of as two 2-photon processes:

1.) The pump and Stokes photons interact to create a **Coherent Oscillating Polarization**, which is maximum when the energy difference between the photons is equal to a molecular resonant frequency.

2.) Probe photons (typically from the same laser as the pump) anti-Stokes Raman scatter from this polarization **Coherently, in what is known as the “Phase Matching Direction”**

\[
\begin{align*}
E_{\text{CARS}} & \propto E_{\text{pump}} E_{\text{Stokes}}^* E_{\text{probe}} \left( n_i, \text{ quantum state} - n_j, \text{ quantum state} \right) \\
I_{\text{CARS}} & \propto I_{\text{pump}} I_{\text{Stokes}} I_{\text{probe}} \left( n_i, \text{ quantum state} - n_j, \text{ quantum state} \right)^2
\end{align*}
\]
Typical Schematic for Psec CARS*  
(* Patterned after S. Roy, et al.)

**Ekspla Nd:YAG Laser**
- 10 Hz, ~150 psec pulses
- 125 mJ per pulse max @ 532 nm

**Modeless Psec Dye Laser**
- Broadband ~592-610 nm FWHM
- ~7-10% conversion efficiency
- Spectral Resolution ~0.4 cm⁻¹

**Folded Box-CARS**
- ~0.5 mm (length) x 0.1 mm (diameter) spatial resolution

**Diagram Details**
- Short-Pass Filter
- 100mm Lens
- Relay Lens Magnification System
- EMCCD
- 0.75 m Spectrometer
- Nd:YAG
- Broadband Dye Laser
- beam dump
- test section
Very Broadband Pyrrromethene Dye Mixture (* S. Tedder, et al., 2011)

95% of signal generated in ~0.5 mm
Some Typical Psec CARS Spectra

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Vibrational Q-branch

\( P = \sim 92 \text{ torr, H}_2\text{-air (} \phi = 0.4 \) \\
\( (P_{N_2} \sim 60 \text{ Torr}) \) \\
100 Shot Average \\
“High” Resolution (\( \sim 0.4 \text{ cm}^{-1} \))

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Pure Rotation

\( P = 40 \text{ Torr (} P_{O_2} \sim 6.5 \text{ Torr}), 20\% \text{ O}_2/\text{Ar} – \text{ H}_2, \phi = 0.5 \) \\
401 nsec Discharge Pulses, 200 Shot Average

\( T = 375 +/- 7 \text{ K} \)

Line Spacing: \( \sim J*(J+1)*(B_1-B_0) \) \\
\( (B_1-B_0) \sim 0.01* \) \* \( B_0 \)

Line Spacing: \( \sim 4B_0 (N_2) \) \\
\( \sim 8B_0 (O_2) \)
Psec CARS Thermometry: Precision and Accuracy (Vibrational Q-Branch)

Histograms produced from 60 fitted spectra at each temperature. Standard Deviation ~10-15 K for both conditions (within ~3-4%)

Sample 100-shot accumulation spectra, in 100 Torr air. $T_0 = 300, 500$ K
O, NO, N LIF/TALIF Experimental Schematic
(All measurements performed with single dye)

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O Calibrated Using Xe*

\[ N_O = \frac{S_O}{S_{Xe}} \cdot g_{ND} \cdot \frac{a_{21}(O)}{a_{21}(Xe)} \cdot \frac{\hat{\sigma}^{(2)}(Xe)}{\hat{\sigma}^{(2)}(O)} \cdot \left[ \frac{v_O}{v_{Xe}} \right]^2 \cdot \frac{1}{F_0(T)} \cdot N_{Xe} \]

NO Calibrated Using 100 ppm NO/N\textsubscript{2}

N Calibrated Using Kr*

Kinetic Modeling: Coupled Master Equation / 0 and 1D* Boltzmann Equation Model of Nonequilibrium Air Plasma

\[
\frac{dn_v(t)}{dt} = (\text{El. imp})_v + (VT)_v + (VV)_v + (VE)_v + (V - \text{Chem})_v
\]

El. Imp.: inelastic electron impact processes by free electrons. \( N_2 \ v = 1, 12 \) excited including processes such as:

\[
N_2(X, v) + e \rightarrow N_2(X, w) + e \quad 0 \leq v, w \leq 12
\]

VT: vibration-to-translation/rotation energy relaxation

VV: vibration-to-vibration energy exchange

VE: electronic-vibration energy transfer during collisional quenching

Exc State Chem: chemistry coupling for electronically (and formally vibrationally) enhanced reactions such as

\[
N_2^* + O \rightarrow NO + N
\]

- Rotational and translational modes are in equilibrium at a gas kinetic temperature
- Single vibrational quantum change processes dominate at low temperatures involved
- Significant body of theory and experimental validation data for the rates used
- Master equation coupled to Boltzmann equation for electron energy distribution, species concentrations equations. \( N_2 \ v = 0, 45 \) included as well as estimate of cathode fall.

(*Working on 2-D in collaboration with M. Kushner)

Nonequilibrium air plasma chemistry, excited electronic states kinetics are included
Ex: Model Validation and Non-Thermal Ignition
Plasma creation by nsec pulsed discharge: \( \sim 30 \text{ kV, } \sim 5 \text{ nsec duration, repetition rate up to } 100 \text{ kHz.} \)

Coupled pulse energy \( \sim 0.7 \text{ meV/molecule} \rightarrow \Delta T \sim 2 \text{ K/molecule/pulse.} \)

- Discharge dimensions \( 1 \text{ cm x 2 cm x 6 cm} \), right angle quartz prism 6 cm long for optical access
- \( \text{N}_2, \text{ air, H}_2\text{-air, and C}_x\text{H}_y\text{-air at } T_0 = 100-300^\circ \text{C, } P=50-500 \text{ torr, } \phi=0.03-1.2 \)
- Flow velocity \( u=40 \text{ cm/sec; residence time in the discharge } \sim 0.1 \text{ sec} \)
- Ample optical access (LIF, TALIF, CARS) for species and temperature measurements

For ns plane-to-plane DBD the specific coupled pulse energy is quite low, typically ~0.7 meV/molecule ($\Delta T \sim 2K$/pulse). For this reason the measurements are performed in repetitive “burst” mode.

- Time between individual pulses in burst: $\sim 10 - 25 \mu s$ (40 – 100 kHz).
- Time delay after last pulse in burst, $\Delta t$: $\sim 2 - 10,000 \mu s$.
- Burst repeated at 5 – 10 Hz, sufficient to purge gas in measurement volume.
Model Validation: Ignition in \( \text{H}_2\)-air* Mixtures Repetitively Pulsed NS DBD

Ignition volume

\[ \text{H}_2\text{-air, } T_0=473 \text{ K, } P=80 \text{ torr, } \nu=40 \text{ kHz} \]

Ignition (as determined by OH spontaneous emission), AFTER application of variable “burst” of ns pulses is extremely reproducible and exhibits a very well defined threshold!

(* Chemistry model of N. Popov)
Time-resolved Psec CARS Temperature of H₂-air AFTER Application of Pulse Burst
(φ=0.4 H₂-air at T₀=500 K, P=92 torr and ν=10 kHz)

Rotational T

Rotational T (and modeled OH)
Log scale

* Models quantitatively captures temperature evolution.
* When burst size reduced to 99 pulses, ignition does not occur.
* Threshold ignition temperature $T_f \sim 700$ K - BELOW $\sim 900$ K autoignition T.
* No overshoot in non-ignition cases, temperature decays to baseline $T_0=500$ K.
Modeling Predictions With and Without Plasma Generation of H and O.

With and without plasma generation of O and H, including dissociation by electron impact and by quenching of $N_2^*$.
$\text{N}_2$ Vibrational Dynamics in Air (and pure $\text{N}_2$)
Smaller (compared to plane-to-plane), bare spherical electrodes enables significantly increased power loading (~300-700 meV/molecule at P = 100 Torr (11.5 - 17 mJ coupled pulse energy) while creating diffuse, uniform plasma with large enough plasma volume to be easily probed by laser diagnostics.

\[ N_2 \quad 100 \text{ Torr} \]
Characterization of Discharge Filament Size

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ICCD Images
During (left) and 1 μsec after pulse (right).
Air, P = 100 Torr, T = 300 K

Spatial scan of “1st Level” $T_v$ by CARS

![Graph showing spatial scan of $T_v$ vs X distance]
Some Typical CARS Spectra – 100 Torr N$_2$
(Normalized to v=0)

100 laser “shot” averaged spectra as function of time after rising edge of current pulse.

Extraction of vibrational level populations by least squares curve fitting.

Vibrational (Log Intensity Scale)

$n=0$

$n=3$

$n=6$

$n=9$
Air, 100 Torr, High Loading: VDFs as Function of Time After Rising Edge of Current.

Total $N_2$ Vibrational Quanta Increase Significantly Between 1 – 10 μs!
What Does (Zero D) Model Predict?

1. Predicts electron impact vibrational excitation quite well.
2. Does NOT predict INCREASE in vibrational quanta after $\sim 1\mu s$ (V-V Transfer Only)
3. Over predicts rotational/translational temperature.

Results suggest collisional processes AFTER plasma decay which “feed” vibration of the ground electronic state. For example

\[ N_2(C) + N_2(X) \rightarrow N_2(B) + N_2(X, v) \]
\[ N_2(A) + N_2(A) \rightarrow N_2(B, C) + N_2(X, v) \]
Results in N₂ are Similar (CARS and Spontaneous Raman Scattering)

Spontaneous Raman Spectrum
N₂, P = 100 Torr, Tᵢ = 300 K
10⁵ laser “shot” (1 hour @ 30 Hz) average.
0.1 (dia) x 2.5 (length) mm spatial resolution

Intensity, counts
3x10⁴
2x10⁴
1x10⁴
594 596 598 600 602 604 606 608
Wavelength, nm

v=6
v=0
v=12

5 µs delay

ε, meV/molecule
εᵥ, meV/molecule

Vib. quanta per molecule

Energy input
Energy vibrational mode
Nᵥ quanta

Time, sec
10⁻⁸ 10⁻⁷ 10⁻⁶ 10⁻⁵ 10⁻⁴ 10⁻³ 10⁻²
0.0 0.1 0.2 0.3
An Even Earlier CARS Study*

N\textsubscript{2} Pulsed (400 nsec) “Glow” Discharge  
P = 60 Torr.

Observed Increase in Total (\(v=1,4\))  
vibrational quanta.

Attributed to  
N\textsubscript{2} (A) + N\textsubscript{2} (A) \rightarrow N\textsubscript{2} (C) + N\textsubscript{2} (X,v)

\(\downarrow\) Total Energy Stored in Vibration

NO, N, and O Measurements (By Single/Two Photon LIF in Single Filament Discharge)
NO Excitation Spectrum (Low Resolution)

Relatively T Insensitive

Intensity (a.u.)

Wavelength (Å)

P1(3,14)  P1(5,12)  P1(5,11)  P1(7,10)  P1(8,9)  P1(7,10)  P1(6,11)  P1(5,12)
P2(23)   P1(2,24)  Q12(16)  Q12(10)  P1(2,24)
Air, 100 torr, “Belke Switch” Pulser - 4 mJ/pulse
N, O, and NO predictions (Baseline 1-D model)

N₂* States in Baseline Model

N₂(C ³Π_u)

N₂(a' ¹Σ_u⁻)

N₂(B ³Π_g)

N₂(A ³Σ_u⁺)

Ground

NO Formation:
N₂⁺ + O → NO + N
k[A ³Σ] = 7 · 10⁻¹² cm³/s,
k[other states] = 5 · 10⁻¹⁰ cm³/s

O + N₂ ← NO + N

N, NO loss due to reverse Zeldovich reaction
Air, 100 torr, 4 mJ/pulse
N, O, and NO predictions (Full 1-D model)

$N_2^*$ States in Full Model

$N_2(E \ 3\Sigma_g^+)$
$N_2(C \ 3\Pi_u)$
$N_2(B' \ 3\Sigma_u^-)$
$N_2(W \ 3\Delta_u)$
$N_2(B \ 3\Pi_g)$
$N_2(A \ 3\Sigma_u^+)$

$N_2(a'' \ 1\Sigma_g^+)$
$N_2(w \ 1\Delta_u)$
$N_2(a' \ 1\Pi_g)$
$N_2(a' \ 1\Sigma_u^-)

Ground

$N_2^* + O \rightarrow NO + N$

$k[A \ 3\Sigma] = 7 \times 10^{-12} \text{ cm}^3/\text{s}$,

$k[\text{other states}] = 5 \times 10^{-10} \text{ cm}^3/\text{s}$
Vibrationally Excited N₂ Production by Reverse Zeldovich Process

Measurements of vibrationally excited molecules by Raman scattering. I
The yield of vibrationally excited nitrogen in the reaction N+NO→N₂+O

Graham Black, Robert L. Sharpless, and Tom G. Slanger

ΔH = ~11 N₂ (v) quanta
(Exothermic)

Citation: J. Chem. Phys. 58, 4792 (1973); doi: 10.1063/1.1679061

View online: http://dx.doi.org/10.1063/1.1679061

View Table of Contents: http://jcp.aip.org/resource/1/JCPSA6/v58/i11

Published by the AIP Publishing LLC.
NO in Air and H₂-Air: Summary, P = 40 Torr
High Specific Pulse Energy (∼550 meV/molecule)

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- Air
- H₂ phi = .07
- H₂ phi = .22

NO lifetime 100 X longer in fuel mixtures!
(N + OH → NO + H)
Thomson Scattering and Four wave Mixing Based E-Field Measurements
Thomson Scattering – Triple Grating Schematic*, **

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Mask (1 mm)

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Nd:Yag 2\textsuperscript{nd} Harmonic (532 nm).
30 Hz – 0.5 J/pulse.

(** Acknowledgement: U. Czarnetzki, Ruhr-University Bochum)
Initial Thomson Scattering Studies in Helium Single Filament Discharge

The Ohio State University Laboratory

Pulsed Filament Conditions:
- 125 ns pulse duration – 60 Hz rep rate.
- ~10 kV peak discharge voltage.
- ~45-55 amps peak discharge current.
- ~15–17 mJ/pulse coupled energy.

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Typical voltage, current, and coupled energy waveforms (P=100 torr).

~100% of energy coupled in 100 nsec.

N₂ Pure Rotational Raman Calibration Spectrum

![Graph showing N₂ Pure Rotational Raman Calibration Spectrum with wavelength (nm) on the x-axis and intensity (a.u.) on the y-axis.]
Some Sample Spectra (As function of time after rising edge of current pulse: Helium – 100 Torr)

(t > ~100 nsec Corresponds to “Afterglow”)
Model is centerline, whereas experiment is spatially averaged over \(\sim 2\) mm and temporally averaged over 10 nsec.
Sub-Nsec Electric Field Measurement by CARS-like Four Wave Mixing
(w. Prof. S. O’ Byrne, U. New South Wales, Australia)

Energy Level Diagram for E-Field CARS

- “E-Field CARS” is a 4 wave mixing process.
- The typical CARS probe beam is replaced by an external electric field, which is at essentially zero frequency. This creates an IR “CARS” signal at the vibrational frequency.
- The physical origin of this signal is the dipole induced by the external field.
- Phase Matching for E-Field CARS is collinear.

\[ E_{IR} = \chi_{IR} E_{Pump} E_{Stokes} E_{External} \]
\[ E_{CARS} = \chi_{CARS} E_{Pump} E_{Stokes} E_{Pump} \]
\[ \frac{E_{IR}}{E_{CARS}} = \left( \frac{\chi_{IR}}{\chi_{CARS}} \right) \star \left( \frac{E_{External}}{E_{Pump}} \right) \]
\[ E_{External} = \left( \frac{\chi_{CARS}}{\chi_{IR}} \right) \sqrt{\left( \frac{I_{IR} I_{Pump}}{I_{CARS}} \right)} \]

Stimulated Raman Shifting Cell Used to Generate $\text{H}_2$ Stokes Beam

Typical Raman Cell Conversion
25 ps Pump @ 532 nm (P= 11 Bar)

Output Energy (mJ)

0 1 2 3 4 5 6 7 8 9

0 5 10 15 20 25

Input Energy (mJ)

anti-Stoke's
Stoke's
Pump

Significant pump power lost due to backwards SBS!
E-Field CARS Temporal Resolution

- Recent work has been done to determine H$_2$ coherence decay time
  - Vibrational CARS intensity created only during 30 picosecond laser pulses
  - E-Field CARS generated until initial polarization decays
- At pressures <1 Bar, coherence decay time decreases approximately linearly with pressure

Initial Results – Diffuse Filament Point-to-Point Discharge

100 Torr Air
Preliminary 2-D Fsec H Atom Imaging in Single Filament Discharge (J. Schmidt talk)

1% H₂ / Helium, P = 100 Torr

Discharge Spontaneous Emission image

5 second average TALIF image
(1 kHz image acquisition rate, 12 μJ/pulse @ 205 nm)
Principal Conclusions

- Psec CARS rotational temperature measurements demonstrate non-thermal ignition in H₂ – air nsec repetitively pulsed plasmas.
- Temporal Evolution of VDF, determined by CARS (and spontaneous Raman), indicates collisional “feeding” of N₂ vibration - Perhaps N₂ (A) + N₂ (A) → N₂ (C) + N₂ (X,v) AND (in air) NO + N → N₂ (v) + O.
- NO formation (in air) dominated by enhanced Zeldovich O + N₂* → NO + N including complete set of N₂ excited electronic states.
- NO Loss dominated by reverse Zeldovich: NO + N → O + N₂ where it appears that significant N₂(v) is formed.
- Psec four wave mixing-based E-field measurement has been demonstrated.
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• Sukesh Roy and Waruna Kulatilaka: Psec CARS.

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• DOE/U. Mich Low Temperature Plasma Science Center.
• DOE/Spectral Energies Phase I SBIR.
• NSF: Plasma Physics Program.
Questions?
Pure Rotational CARS

- Uses planar, orthogonal polarization phase matching scheme*.
- Minimizes "non-resonant" background and facilitates separation of Probe and CARS beams, which have virtually the same wavelength.
- Enables high intensity beams with insignificant distortion of spectrum due to Stark broadening.

(*) Vestin, )
Psec Pure Rotational CARS Thermometry
(Model validation studies in O$_2$/Ar)

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H$_2$/O$_2$/Ar, P = 40 Torr, T$_0$ = 300 K \( \nu = 25 \) kHz
(Popov Chemistry Model)

401 pulses, 200 Shot Average, 95% CI = 7 K

- \( P_{O_2} \approx 6 \) Torr.
- Laser intensity results in significant Stark broadening, but it does not influence the pure rotational temperature.
- Temperature precision (95% CI) \( \approx \) 5-10 K.
- Inferred temperature agrees well with model predictions.
Why Psec CARS?

OSU Portable CARS System (~0.75 x 1.5 m)

- For...mation with chemical...r...ter
- In...ical...nt with chemical...r...ter
- In...ical...nt with chemical...r...ter

Component labels:
- Computer
- Spectrometer
- Nd:YAG
- Dye Laser
- YAG Power Supply
- Dye Pumps and Reservoirs
Some Other Similar Work in Nsec Discharges

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- \( T_v \) and \( T_{rot} \) \( \text{N}_2 \) Pin-to-Pin Discharge
- 1 Bar by CARS \((v=0-2\) Probed\)*
- (Attributed to V-V xfer)

\[
\text{N}_2 (v) + \text{N}_2 (w) \leftrightarrow \text{N}_2 (v+1) + \text{N}_2 (w-1)
\]

**Nonequilibrium Thermodynamics Laboratory**

- \( T_v \) and \( T_{rot} \) \( \text{N}_2 \) Pin-to-Plane Air
- 1 Bar by Spontaneous Raman*

![Graph 1](image1)

![Graph 2](image2)


$N_2$, 100 Torr, High Loading: VDFs as Function of Time After Rising Edge of Current.

Total Vibrational Quanta INCREASES by Factor of $\sim$ Two AFTER $1 \mu$sec
(Consistent with Devyatov)
Psec CARS Sensitivity: Mach 5 Flow Measurements Using nsec Pulser-DC Sustainer Discharge in Plenum to load $N_2$ (v)

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5 mm diameter x 7.5 mm long quartz cylinder model.

- (a) Free-stream ($P_0 \sim 1.2$ Torr, $T_0 \sim 50$ K) and behind shock ($P_0 \sim 30$ Torr, $T_0 \sim 300$ K) – 8 shot average, Plasma OFF.
- (b) Mach 5 free-stream, Pulser-sustainer discharge 10 shot average.
- (c) Behind bow shock, Pulser-sustainer discharge 10 shot average.

Sensitivity limited by EMI pick-up of CCD camera.
Model Validation: Vibrational Energy Loading

Vibration loaded by low E/n pulse reflections.

Analytical 1-D DBD Model Predictions (Adamovich)

\[ P_0 = 100 \text{ torr air}, \ T_0 = 300 \text{ K}, \ \nu = 10 \text{ kHz}. \]
Model Validation: Vibrational Energy Decay

\[ P_0 = 100 \text{ torr air}, \ T_0 = 300 \ K. \]

- Model predicts energy loading quantitatively.
- Model slightly under predicts rate of vibrational energy decay.
- Rate of V-T relaxation (by O\textsubscript{3}) likely under predicted.

\[ \text{N}_2(\nu = 1) + \text{O}_3(000) \rightarrow \text{N}_2(\nu = 0) + \text{O}_3(101) \]
NO – Summary of Results – Air, C₂H₄-Air

P = 40 Torr

Ethylene-Air Behavior is Similar

- Air
- C₂H₄ phi = 0.48

Number Density (cm⁻³)

Time After First Pulse (µsec)
Data/1-D Model Comparison

Air
\[ \Phi = 0.14 \]
\[ \Phi = 0.41 \]

Extended NO lifetime due, principally, to extended Zeldovich process:
\[ N + OH \rightarrow NO + H \]

Peak NO and NO decay rate over predicted in this case.
NonEquilibrium Thermodynamics Laboratory (NETL)

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Igor Adamovich, Bill Rich, Sergey Leonov, Jeff Sutton

Current Students
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Waruna Kulatilaka, Sukesh Roy, Jacob Schmidt (Spectral Energies)
Sean O’Byrne (U. of New South Wales)
OH LIF: Experimental Apparatus

Excite linear laser excitation of transitions in OH A-X (1,0) @ ~280 nm; Collect fluorescence from (0,0) and (1,1) bands @ ~310 - 313 nm.
OH LIF – Some Details

LIF excitation spectrum and Boltzmann plot used for temperature measurement. T Agrees Well with CARS.

EX: H₂-air, T₀=500 K, P=100 Torr, φ=0.12, ν=10 kHz, 50 pulses.

Q₁(3) excitation gives high sensitivity and modest T – dependence.

Absolute [OH] by Rayleigh scattering calibration

\[ S_{OH} = n_{OH} f_b b_{lu} E_j \left( \frac{W}{4p} \right) \]

\[ S_{Ray} = \frac{e_{Ray}}{hc} \int \int \left( \frac{I W_b}{\omega} \right) d \omega d \lambda \]

\( I \Omega \beta \) product determined from slope of \( S_{Ray} \) vs (NE) = (P/kT)E.