Yttrium Speciation in an Inductively Coupled Plasma Utilizing Acousto-Optic Tunable Filter Hyperspectral Imaging (AOTF-HSI)

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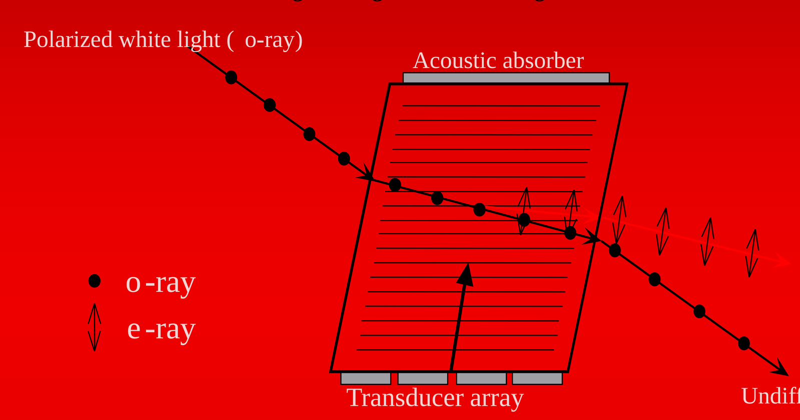
Abstract

- Visualize several species of multiple elements
  - How/where they reside/interact throughout the plasma
  - Increase knowledge of plasma chemistry
  - Allow for the ability to understand observed results found using other ICP instruments
- Observe yttrium atom and ion lines as well as YO molecular bands
- CCD camera used to take analytical and background images of the plasma
  - Obtain spatially resolved emission images in real time
- Known Y(I), Y(II), and YO bands optimized using a 1M monochromator
  - Found using AOTF-HSI system
- Results indicate that this system can be used for yttrium speciation
  - Future work would involve additional elemental speciation

1. Acousto-Optic Tunable Filter

Acts as a a narrow bandpass transmission filter
Tunable
Variable wavelength

- **High frequency variable stress is applied to an optically transparent medium**
  - **Perturbation produces periodic variations in the medium**
    - **These packets of energy are known as phonons**
  - **Light is directed through the medium**
    - **Photons of the light interact with the phonons in the crystal medium diffracting the light**
    - **Diffracted and 0-order light emerges at different angles with non-collinear devices**



- **The central wavelength of transmitted light is described by the equation:**

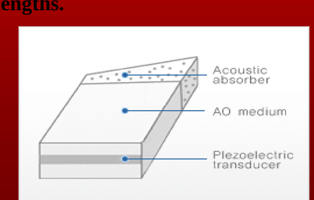
$$\lambda_d = \frac{v\Delta n}{f}\sqrt{\sin^4\theta_i + \sin^2 2\theta_i}$$

- **Diffraction can occur for radiation in which the phase is not perfectly matched with that of the phonon**
- **Radiation of wavelengths slightly greater and less than λ will be transmitted**
- **The bandpass is the wavelength range of the diffracted radiation band**
  - **Bandpass Δλ is defined as the full width at half-maximum intensity of the diffracted light described by**

$$\Delta\lambda = \frac{0.9\lambda_o^2}{\Delta n L \sin^2\theta_i}$$

assuming all other terms to be approximately constant, Δλ is proportional to the square of the diffracted wavelength
  - **This relationship indicates that better resolution occurs at shorter wavelengths.**

- IntraAction Corporation AOTF Specifications
  - Spectral range: 400-700 nm
  - Spectral resolution: 0.5 nm @ 400 nm and 2.5 nm @ 700 nm
  - Optical aperture: 3 x 3 mm
  - RF frequency range: 75-150 MHz
  - Drive power range: 0-10 W



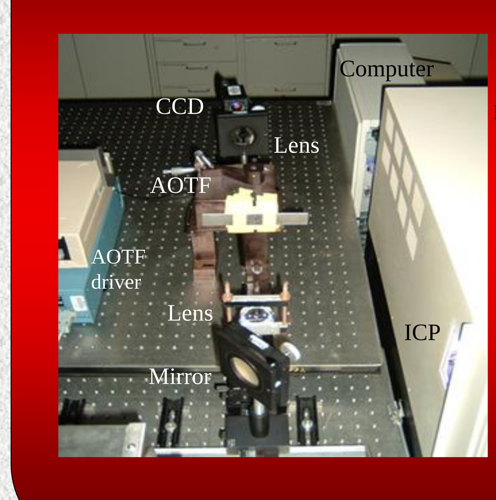
2. Hyperspectral Imaging

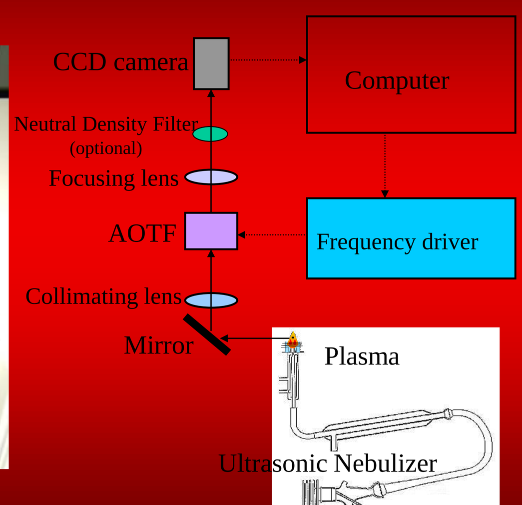
- Mode of data acquisition belonging to chemical imaging
  - Hyperspectral imaging collects the same picture on many bands of the light spectrum to generate a "datacube"
    - Which can reveal objects and information that more limited scanners cannot pick up.
- Advantages
  - Non-invasively analyze proximal or distal objects
  - Highly detailed spectral information in a short period of time
    - Software Dependent
  - Many potential applications
- Addition of AOTF makes HSI more accessible
  - Solid-state design
  - Rapid wavelength accessibility

http://www.imaginghardware.com/Products/Cameras/Images/P98069.jpg

Sony XCD-X710
CCD black-and-white video camera module
Solid state image sensor
1024 x 768 pixels
Sensitivity= 4 *lux*

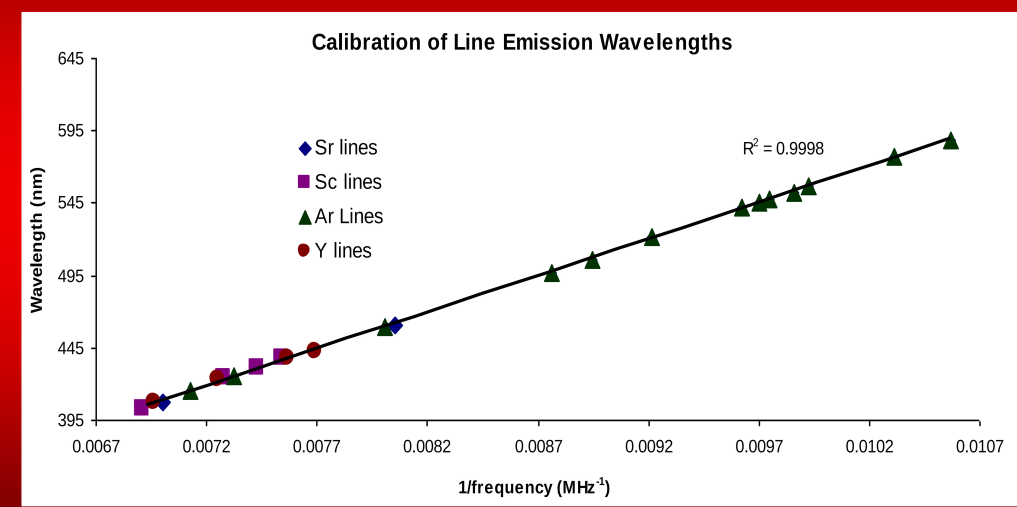
3. ICP-AOTF-HSI System



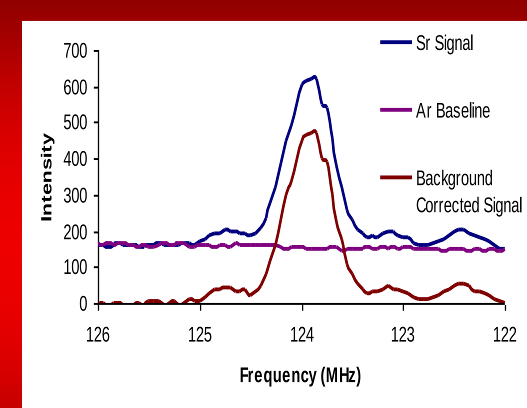


4. Wavelength / Frequency Calibration

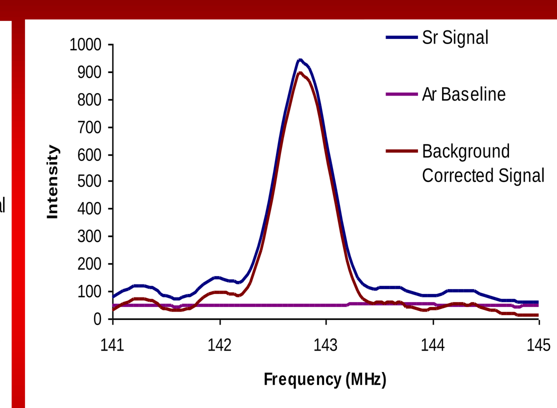
Compare frequencies of emission lines with the known emission wavelengths
The wavelength of light undergoing AOTF diffraction (λd) corresponds to the applied frequency
Calibration aided with the use of Ar, Sc, Sr, and Y emission lines.



5. Sr Atom and Ion Spectral Emission Lines



Sr atom line [50ppm Sr(I) at 460.73 nm]

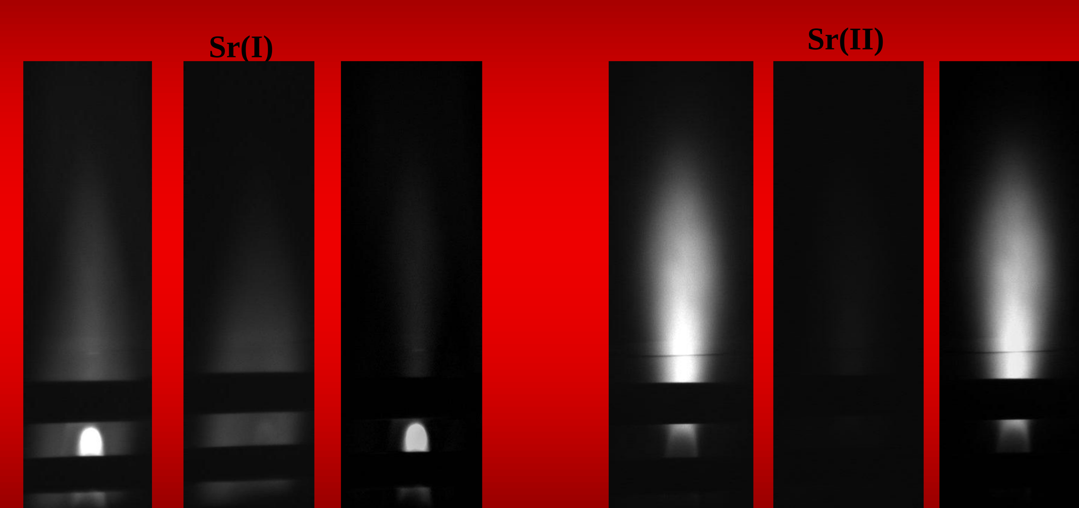


Sr ion line [50ppm Sr(II) at 407.77 nm]

Examples of off-line background correction are shown for Sr(I) and Sr(II). The "observation pixels" were in the ICP's analytical region.

6. Sr Atom and Ion Emission Images

True analyte intensity distributions were obtained by background correction of online and offline plasma images of a 50ppm Sr solution

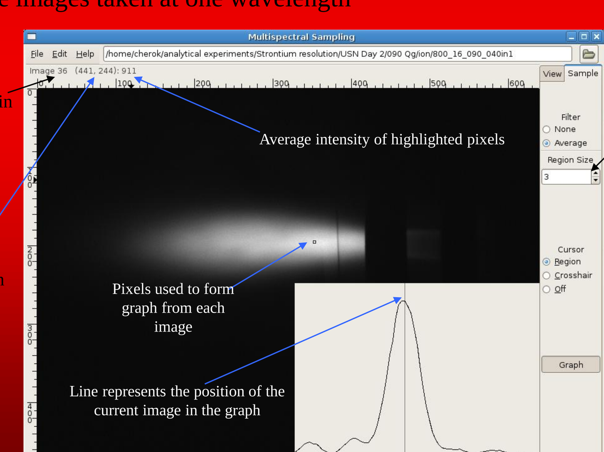


Sr(I): 124.0 MHz (460.73 nm) **Online image** | 123.26 MHz **Offline image** | **Background Corrected image**

Sr(II): 142.8 MHz (407.771 nm) **Online image** | 141.5 MHz **Offline image** | **Background Corrected image**

7. System Software

- Visualize plasma while scanning wavelengths in real time
- Automation of frequency change with image gathering
- Frequency range 75-150 MHz with steps as small as 0.01 MHz
  - Each final image is a average of three images
  - Images can be taken at multiple resolutions and screen sizes
- Gathered images analyzed at one pixel throughout all images
  - Images can be analyzed by averaging multiple pixels throughout all images (frequencies/wavelengths)
  - Pixel data can be exported as a graph, image, or region
  - Data can then be manipulated using additional programs
- Multiple images taken at one wavelength

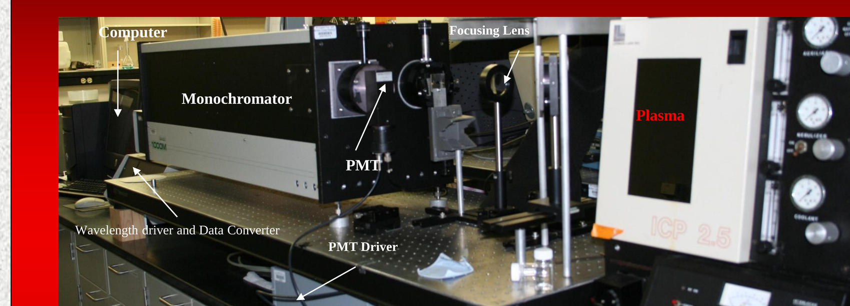


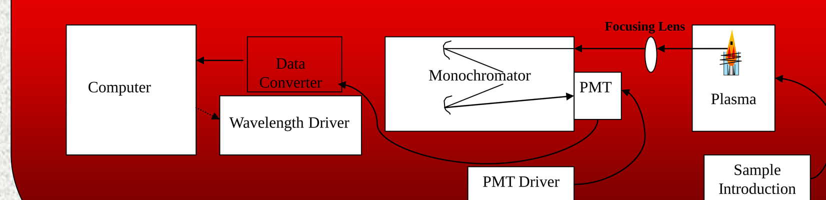
Graph Specifications
Frequency range of graph 141-145 MHz
Related to 403.8-413.7 nm
Selected image @ 142.85 MHz
Related to 407.77nm

8. Yttrium Speciation Study

- Background
  - Necessary to understand plasma chemistry
    - Understand observed results using other ICP techniques
  - Helpful to look at images of emission lines
    - Ion, atom, and molecular bands
    - Visualize interactions/residences
    - Visualize effects of changes in plasma parameters
- Study
  - Correctly Identify Y(I) and Y(II) emission lines, and YO emission bands
  - Collect data for these three Y emissions using the AOTF-HSI system
    - Determine emission trends while changing the plasma rf power
      - 0.88 kW-1.12 kW with a 0.04 kW step
    - Determine emission trends with the addition of H_2
      - Addition of 5% H_2
      - YO+H→Y+OH
  - Compare data to that of a 1M monochromator
    - Use same conditions

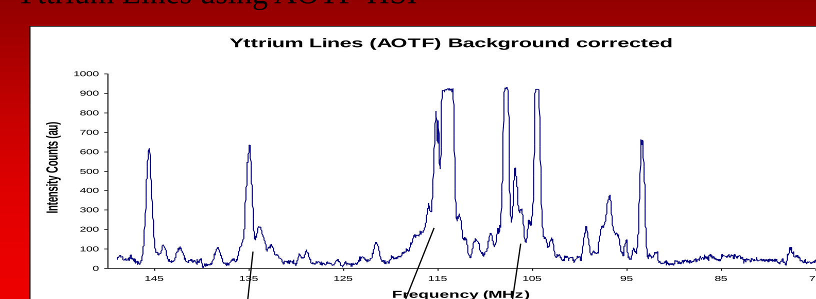
9. ICP-Monochromator System



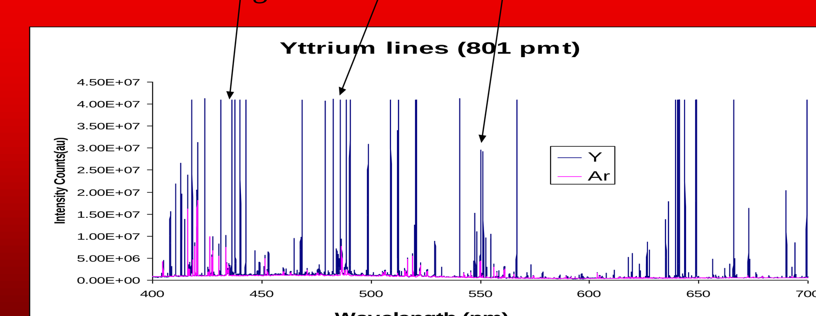


10. Yttrium Emission Line Identification

- Yttrium Lines using AOTF-HSI

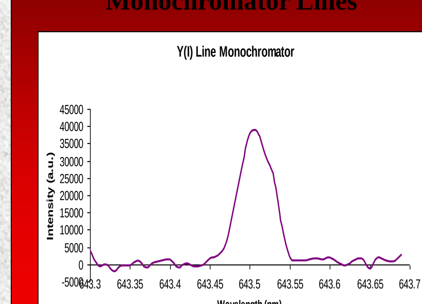
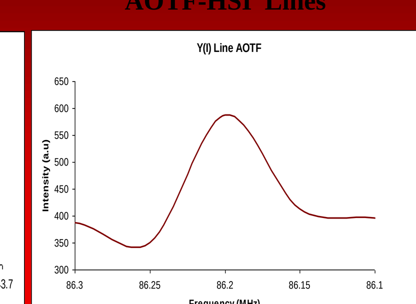


- Yttrium Lines using 1M monochromator

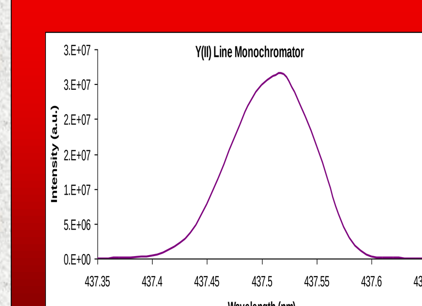
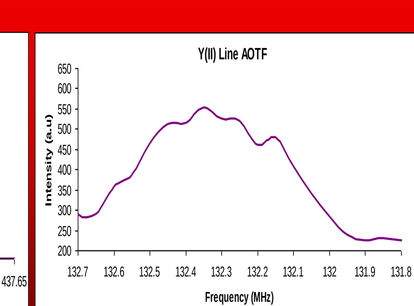
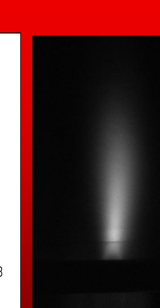


11. Yttrium Atom and Ion Emission Lines

Monochromator Lines | **AOTF-HSI Lines** | **Background Corrected Images**

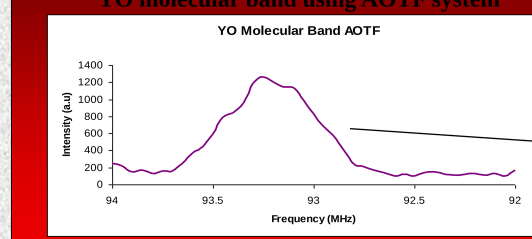

Y(I) 643.5 nm

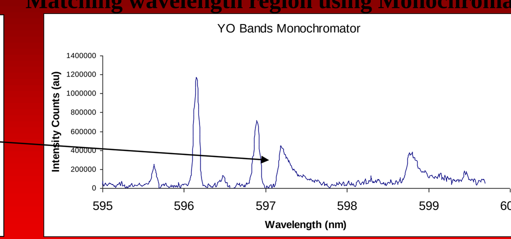
Y(II) 437.494nm

12. YO Emission Band Identification

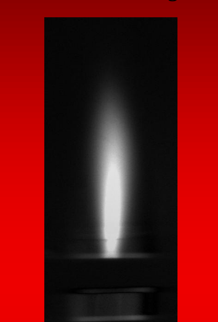
YO molecular band using AOTF system



Matching wavelength region using Monochromator

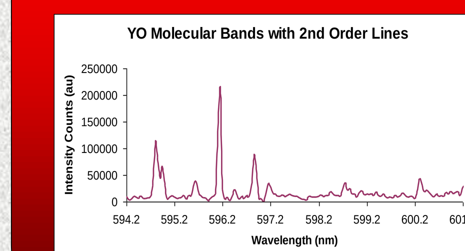


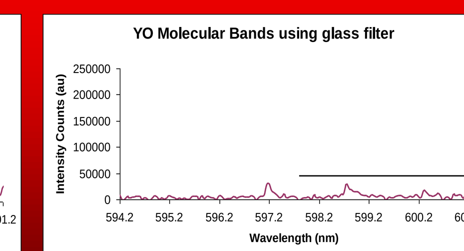
Background Corrected Image

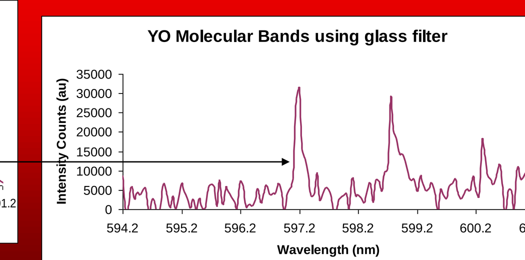


YO 597.2 nm

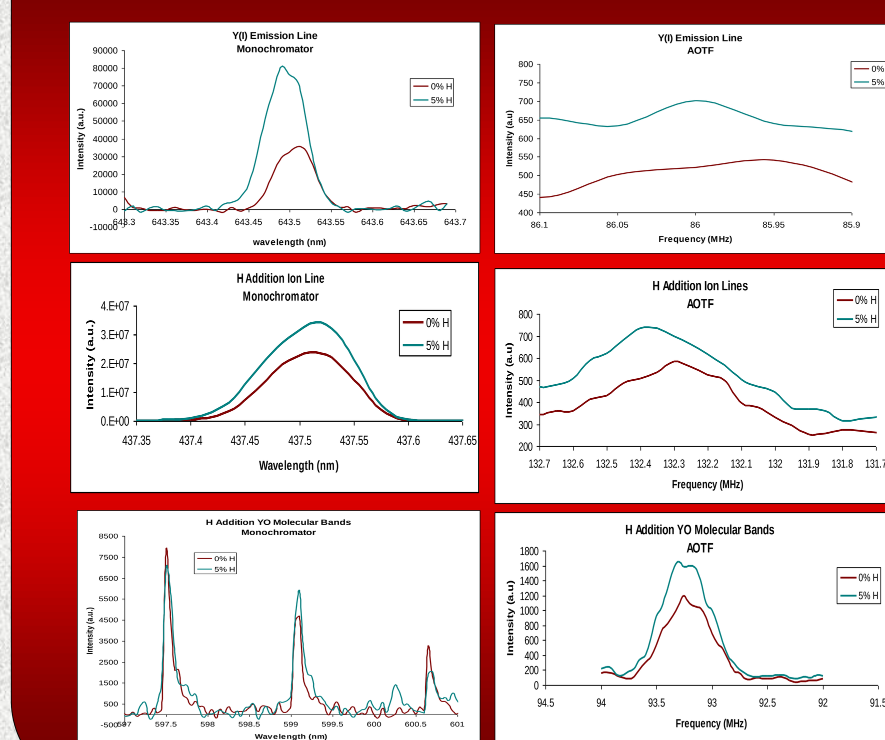
- Emission Lines between 595 to 597 nm were assumed to be 2nd order lines
- AOTF is opaque to wavelengths below 400nm; These 2nd order lines would not appear in resulting spectra
- A glass slide was placed in front of the monochromator entrance slit to subtract out emission lines below the visible region







13. Hydrogen Addition

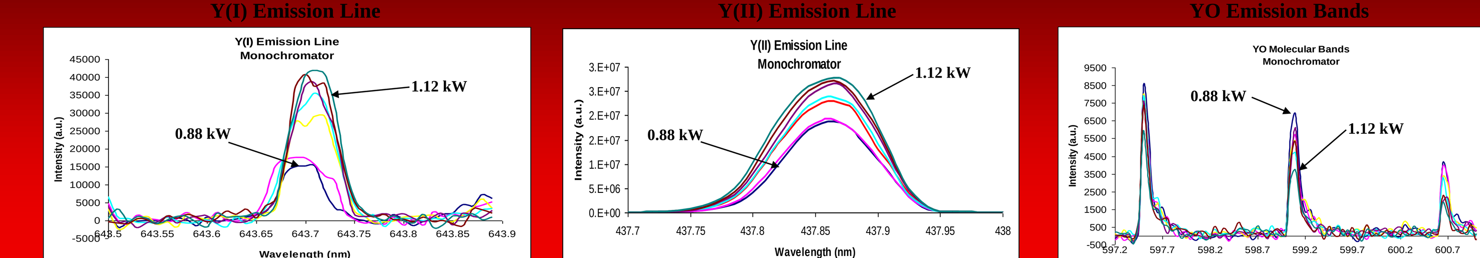


Background Corrected Images
0%H **5%H**

14. RF Power Study

Y(I) Emission Line | **Y(II) Emission Line** | **YO Emission Bands**

Monochromator Emission Lines



Background Corrected Images

kW 0.88 0.92 0.96 1.00 1.04 1.08 1.12 | 0.88 0.92 0.96 1.00 1.04 1.08 1.12 | 0.88 0.92 0.96 1.00 1.04 1.08 1.12

15. Conclusions / Future Work

- **Conclusions**
  - This ICP-AOTF-HSI system is capable of elemental speciation
  - Y(I), Y(II), and YO hydrogen addition trends found with the AOTF-HSI system are similar to those found with the monochromator
  - Y(I), Y(II) plasma rf power trends are similar with both the AOTF and monochromator system
  - Plasma rf power YO trends are very different between the AOTF and monchromator system
    - Probably a spatial issue
    - Need to separate and differentiate plasma heights
- **Future Work**
  - Focus on plasma chemistry and characterization
    - Determine trends in different regions of the plasma
  - Attempt speciation experiments with other elements
  - Compare speciation capabilities with multiple nebulizers

A Special Thanks To

Northern Illinois University: Department of Chemistry and Biochemistry