Development of Micro Focusing System of UV Vis Spectra of Liquid Jet Samples

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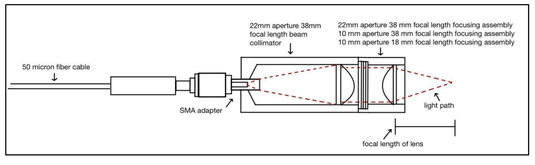
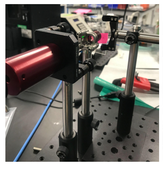
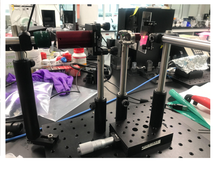
Introduction

- Develop microfocused UV Vis system to measure UV Vis spectra of small round liquid jets (<100 microns)
- Liquid jet user experiments encounter problems with sample decay, light sensitivity, or damaged by oxygen exposure that are difficult to track during the experiment
- Multiple beam diameter & optic footprint combinations to ensure the microfocused UV Vis system would be adaptable to diverse configurations at LCLS

Design

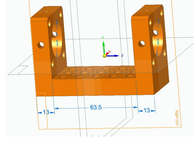


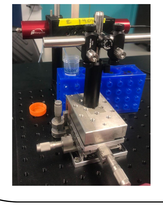
- Three pairs of different focusing assembly sizes, a pair of optic collimators, and a pair of 50 micron fiber optic cables to create smallest beam size possible

- Encountered difficulty with aligning collimator and smaller size focusing assemblies, printed adapter to join them

- Needed better way to hold both collimators and focusing assemblies in alignment with one another, designed and printed a "u-cage" to hold collimators using SolidWorks and Cura Ultimaker S5 printer

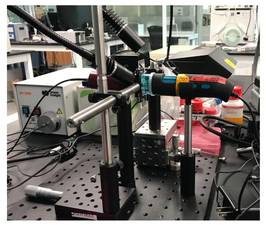




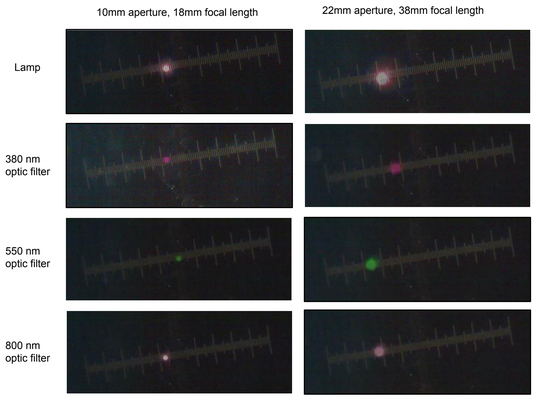
- Discovered that bringing beam into focus while maintaining alignment would be very difficult with rigid design, mounted optical lenses on a manual XYZ stage and sample on manual sliding table

Beam Size

- Quantitative measurement of spot size at focal length to ensure it would be smaller than the size of the liquid jet sample
- Used Q-Scope and a micron ruler sized 10 microns-per-tick to capture images of beam in precise focus
- Used Jupyter Notebook to analyze spot size of captured images in terms of pixel count
- Used Jupyter Notebook to analyze length of a micron in pixels based on the in-focus images of the micron ruler captured by the Q-Scope
- Translated pixels to microns for spot size

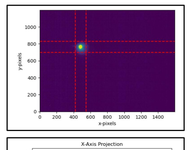
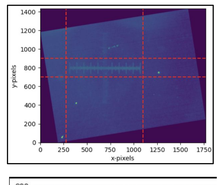
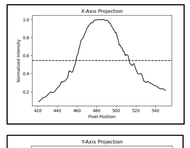


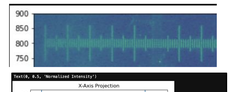
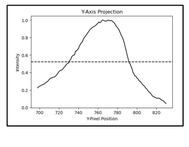
**10mm aperture, 38mm focal length refocusing assembly failed to properly refocus light into a beam that was visible or quantifiable for both high and low wavelengths

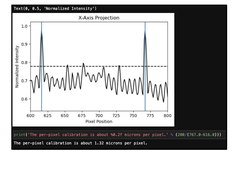


Diameter of Spot Size (pixels)	10 aperture, 18 focal length	22 aperture, 38 focal length
Lamp	33	50
380 nm	27	40
550 nm	19	36
800 nm	18	35

Diameter of Spot Size (microns)	10 aperture, 18 focal length	22 aperture, 38 focal length
Lamp	43.56	66.00
380 nm	35.64	52.80
550 nm	25.08	47.52
800 nm	23.76	46.20

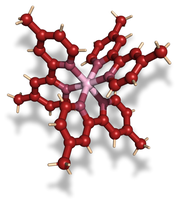




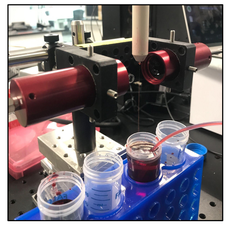





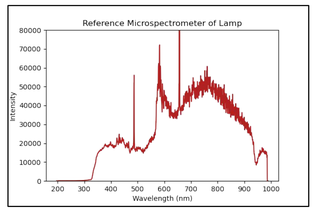
UV Vis Spectrum

- Measured UV Vis spectrum of $[Fe(bpy)_3]^{2+}$, expected to see two different metal-to-ligand charge transfer absorption bands at 522 nm and 354 nm
- Initially began with the 50 micron jet and 10mm aperture 18mm refocusing assembly but beam's transmittance was far too low to accurately measure a spectrum
- Switched to 100 micron jet and 22mm aperture 38mm focusing assembly

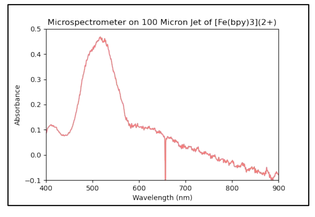




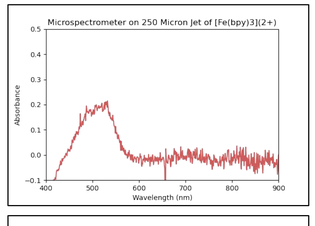
- Aimed to translate both the jet and the optical enclosure to focus the beam through the exact center of the jet at the exact focal length of the focusing assemblies
  This was more difficult with the 100 micron jet, so we switched to a 250 micron jet to obtain a more accurate spectrum



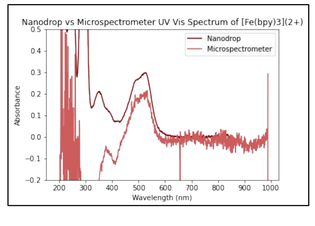
- Reference spectrum of UV Vis lamp without sample showed that very little light from lower wavelengths was being received by the spectrometer, and we expected this to impact our spectrum of $[Fe(bpy)_3]^{2+}$



- $[Fe(bpy)_3]^{2+}$ at concentration of 4.19 mmol/L (1:4 dilution of 16.75 M stock), measured in 100 micron liquid jet. Spectral peak at 522 nm clearly observable.



- $[Fe(bpy)_3]^{2+}$ at concentration of 1.34 mmol/L (4:50 dilution of 16.75 M stock), measured in 250 micron liquid jet. Spectral peak at 522 nm clearly observable



- UV Vis spectrum of $[Fe(bpy)_3]^{2+}$ at 4:50 dilution measured by Nanodrop Microvolume Spectrophotometer compared to our microspectrometer, proximity of results indicates microspectrometer is accurately reading UV Vis spectrum

Conclusions

- Microspectrometer produces meaningfully accurate results that can communicate the state of a sample
- Beam size is reliable <50 microns for the smaller focusing assembly, and reliably <60 microns for the larger focusing assembly
- Compact, mobile, and affordable (with developing accuracy and precision), this microspectrometer has the potential to be a helpful and accessible resource to user groups in both online and offline experimentation who wish to verify the state of their sample

Further Considerations

- Eliminating the adapter by obtaining properly sized collimators could further reduce the spot size to be reliably <50 microns without sacrificing intensity of the beam
- A more powerful light source, such as a commercial supercontinuum white light laser would increase intensity of beam and quality of UV Vis spectrum
- Optomechanics or computerized motors to control optics would contribute to ease of alignment and repeatability

Acknowledgments

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