Development of a Modular Process Flow Cell for UV-Vis Monitoring of LCLS Samples

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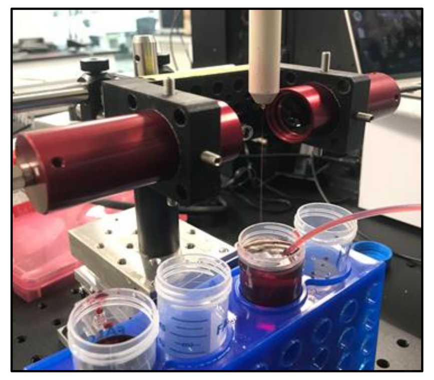
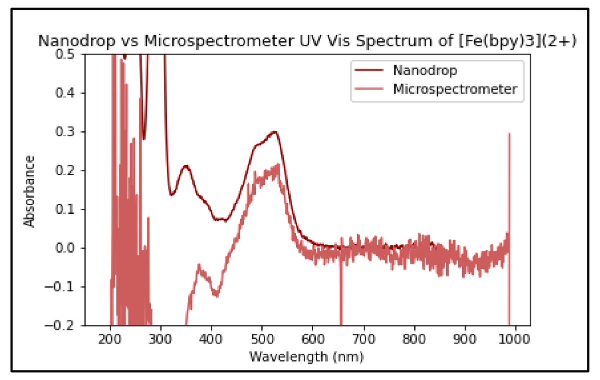
Introduction

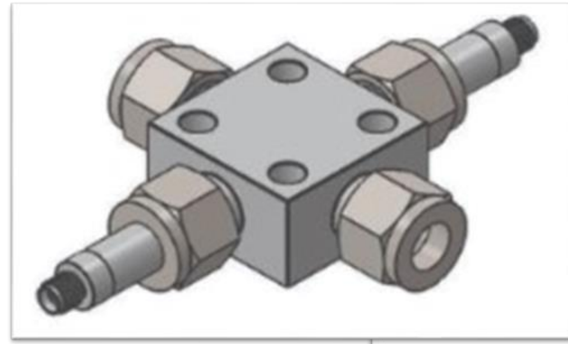
- Couple liquid jet sample delivery system with micro focused spectrometer in a way such that high liquid pressures can be withstood
- Liquid jet user experiments encounter problems with sample decay, light sensitivity, or damaged by oxygen exposure that are difficult to track during the experiment
- Enable users to characterize their samples in real time before interacting with the beam, continually monitoring sample integrity

Last Year: Challenges & Inspiration

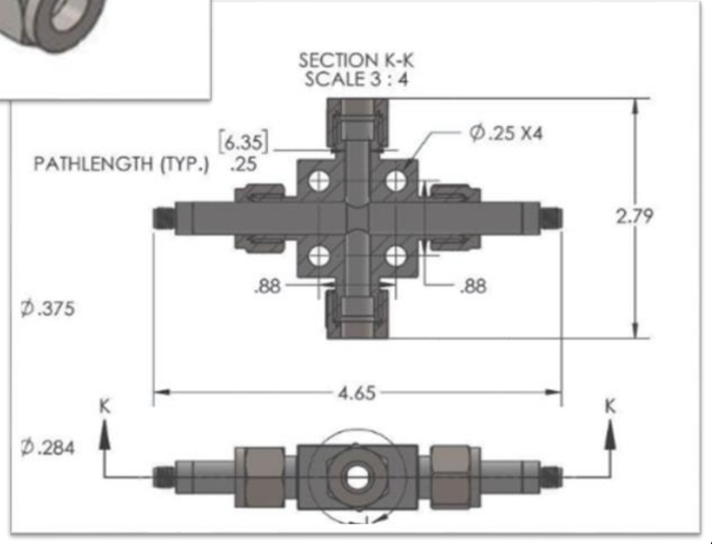




Issues with last year's setup: Difficult to align, losing intensity of beam, unreliable for beam sizes < 50 microns, bulky

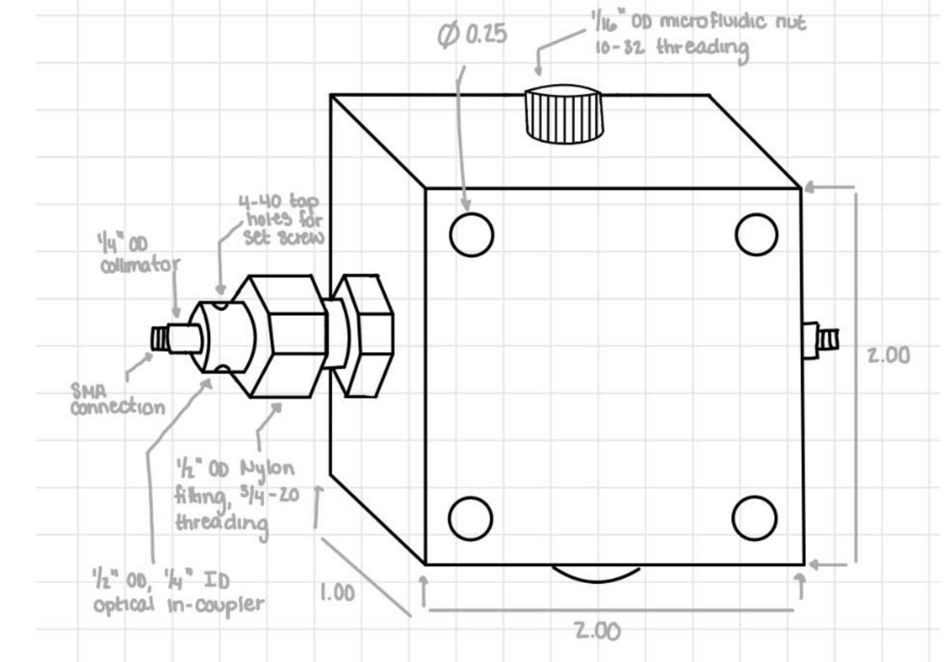


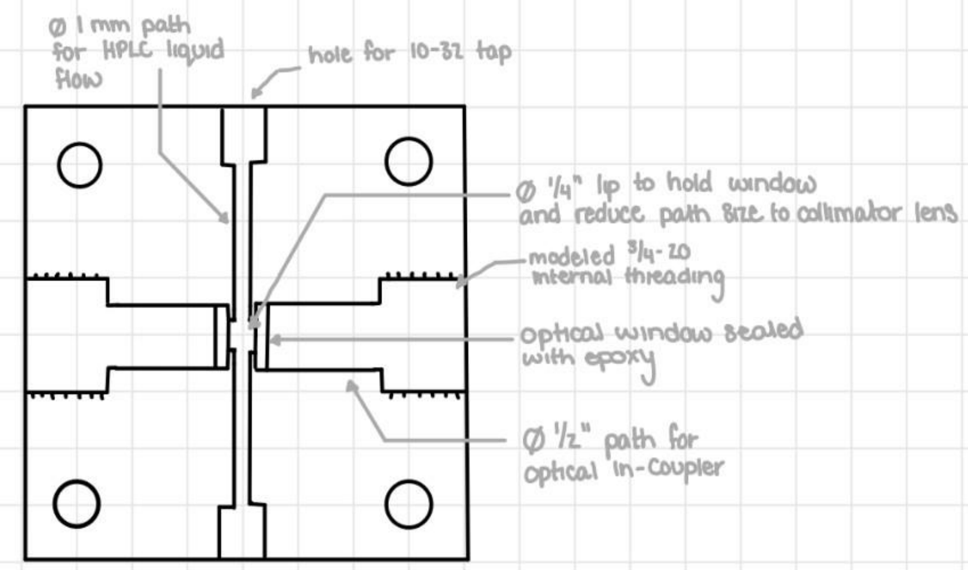
Took inspiration from manufactured absorbance cross flow cell designs.



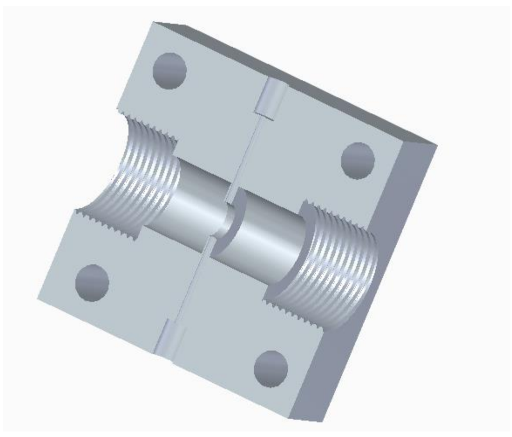
Necessary features:
- Watertight
- Able to withstand high pressures
- Couple with ¼" collimator
- Adaptable for many user and staff experiments

CAD Design



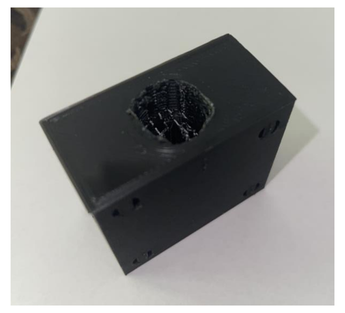


Solutions to various challenges:
- Watertight: thin layers, sealing with epoxy, using compression fittings
- High pressures: PEEK material amenable to high pressure, compatible fittings
- Coupling ¼" collimator: optical in coupler to fit collimator into nylon fitting, held with set screws
- Adaptability: PEEK material which has high solvent-compatibility, compression fittings to adjust path length with ease

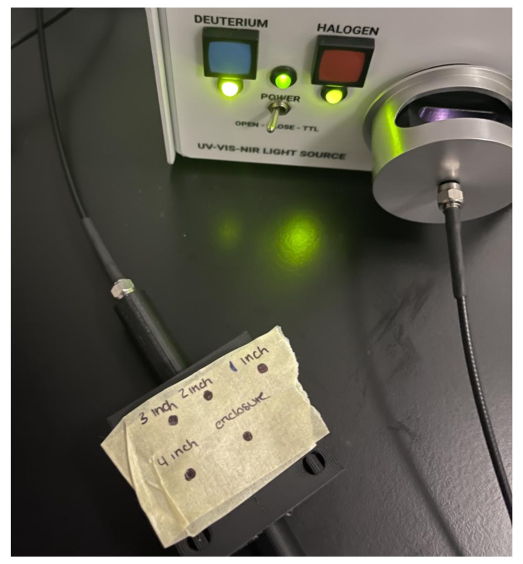


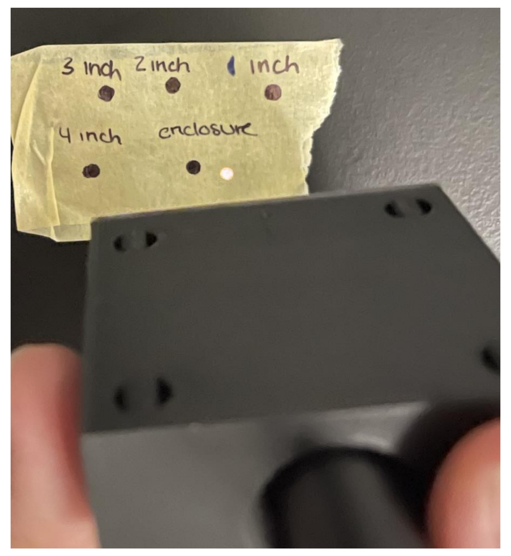
Printing & Adjustment

- Print prototypes in tough PLA with Ultimaker, with goal of using Funmat Pro to print final model in PEEK
- Multiple iterations of prototype



Tested spot size from collimator at multiple distances to ensure a constant beam size would be maintained to interact with sample and optimize beam intensity





Goals for continuing this project:
- Complete a successful PEEK print with PVA supports & post-process
- Assemble flow cell with connections and optical windows
- Using Beer's law, verify variable pathlengths by measuring UV-Vis spectra of known concentrations of iron tris bipyridine, take samples of spectra by connecting HPLC and spectrometer to flow cell and compare this to a Nanodrop spectrum.

Aside: Batlle with the Ultimaker S5 in the shared characterization lab
- Goal to print support material in PVA (polyvinyl alcohol), a water-soluble support material design for high-precision projects
- Issues to printing with PVA included very old and brittle fillament, extruder jammed with fillament, debris in printer nozzle, broken tension on feeder knurl, and overheating PVA, now fixed!

Conclusions

- Compact, mobile, and affordable design that would ensure accuracy and precision, this flow cell has the potential to be a helpful and accessible resource to user groups and staff in both online and offline experimentation who wish to verify the state of their sample
- Capable of adapting to various experimental design, with high solvent-compatibility, amenable to high-pressure, adjustable to collimator size and jet size
- Rapid prototyping and 3D printing ensures an easily modifiable design

Further Considerations

- Cell could be modified to introduce a strong light source to "pre-pump" then monitor a sample before the X-ray interaction point; could optically access molecular configurations that don't have long-term experimental stability but are interesting on ultra-fast timescales
- Other diagnostics like pH sensor, temperature probe, conductivity sensor, flow sensor, etc. could have their ports integrated to create a complete diagnostic package for samples

Acknowledgments

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