How To: ATR-IR Spec-E-Chem

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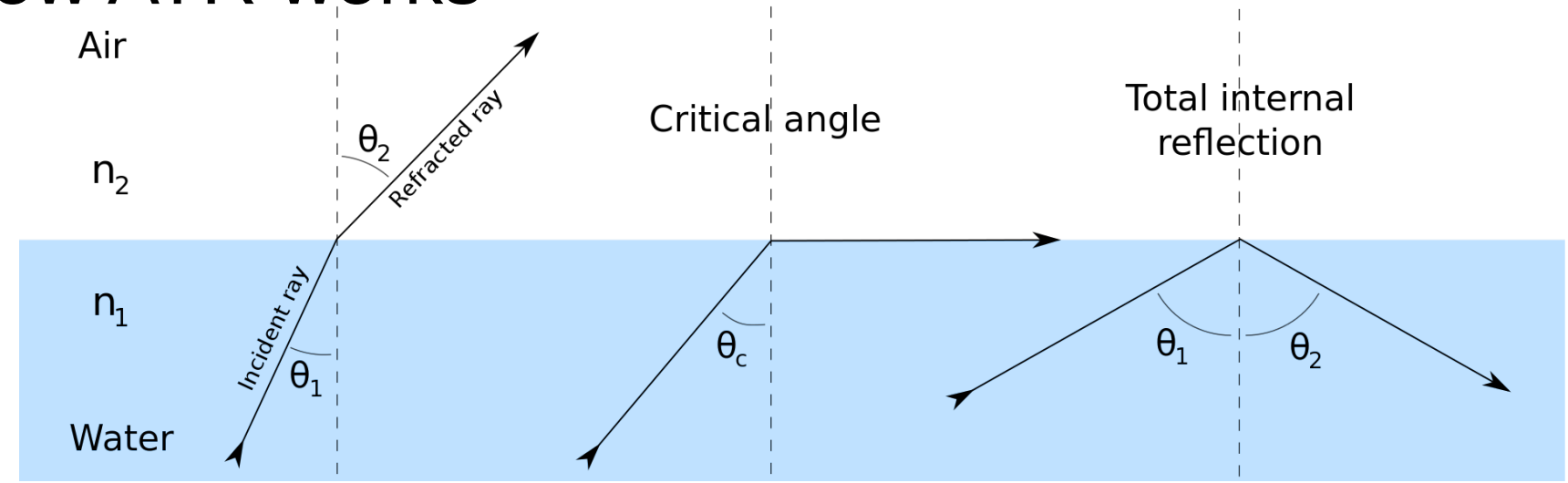
How ATR works

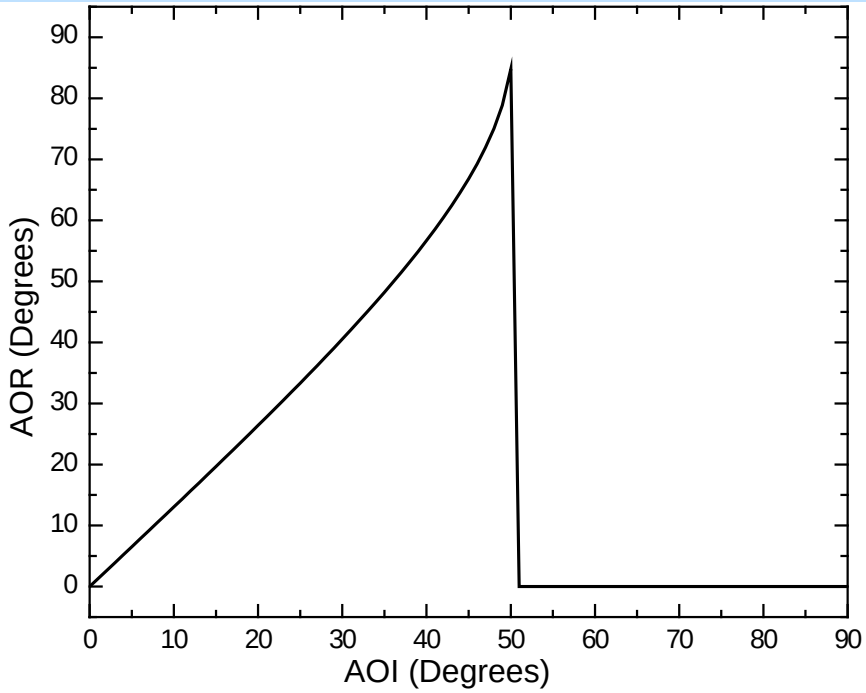


$$\eta_1 sin\theta_1 = \eta_2 sin\theta_2$$

$$\frac{\eta_1}{\eta_2} sin\theta_1 = sin\theta_2$$

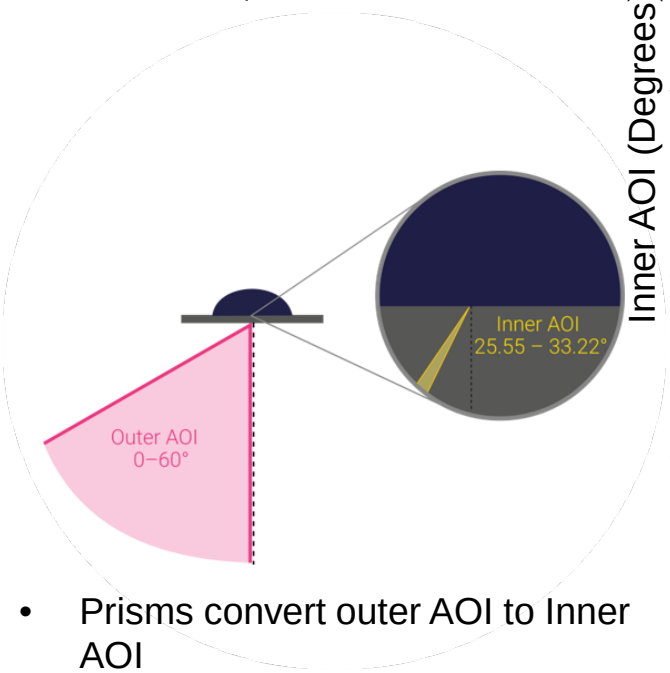
$$\theta_{crit} = \arcsin\left(\frac{\eta_2}{\eta_1} sin\theta_2\right)$$

- Snell's Law describing the critical angle for TIR
- Critical angle for Water-Air Interface Shown →

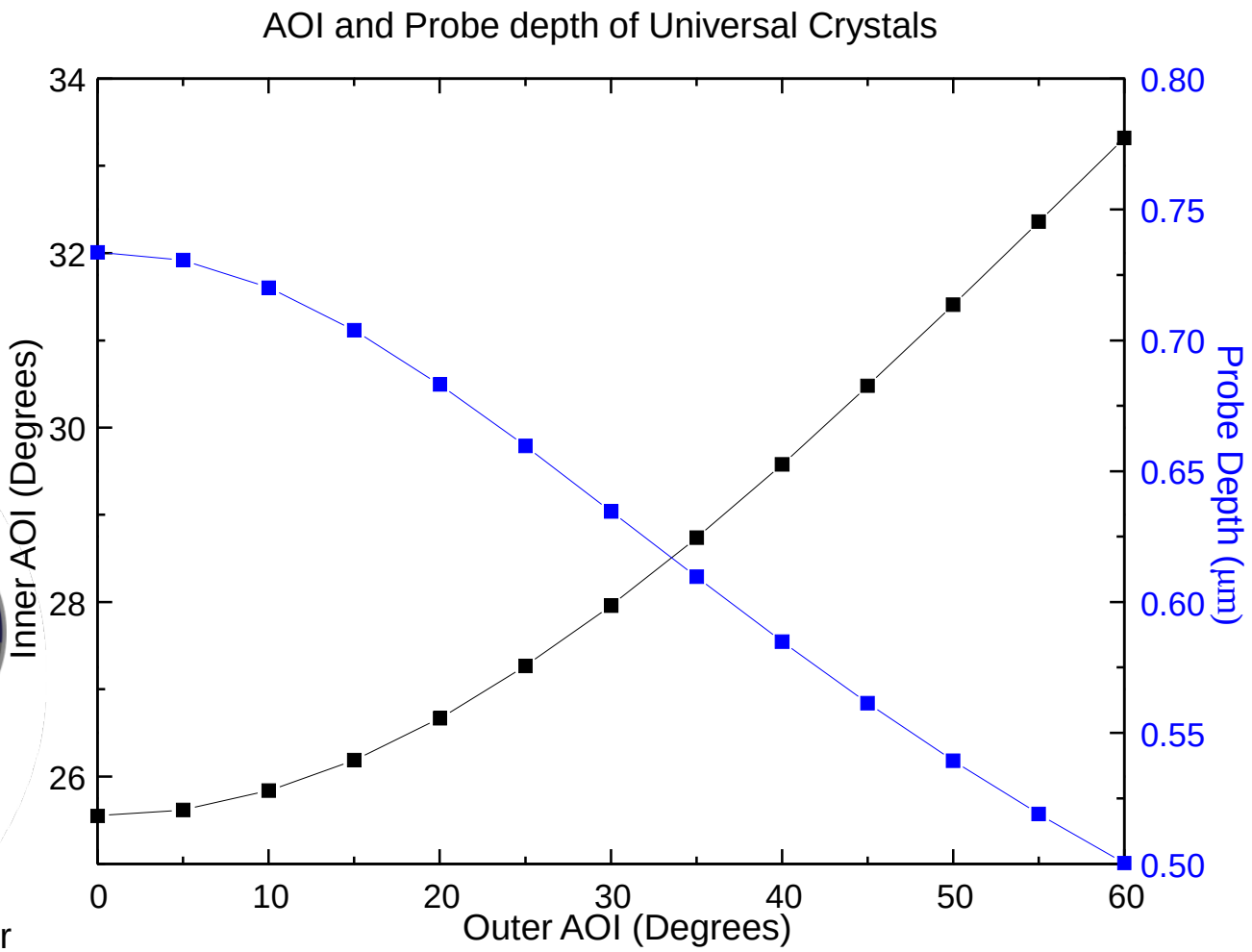


Probe depth is dependent on Angle of Incidence

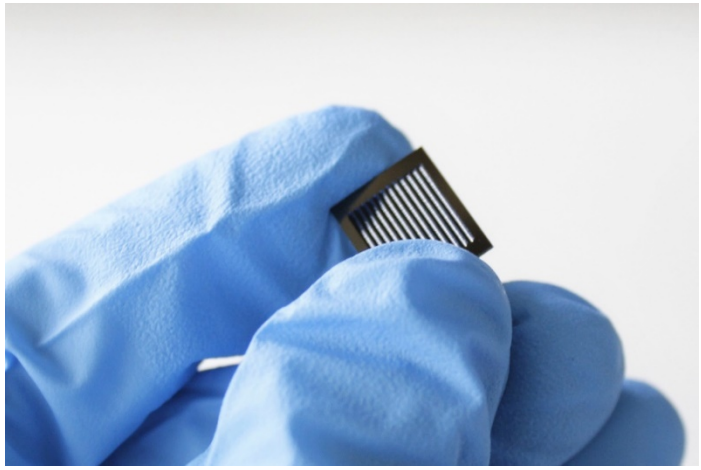
$$d_p = \frac{\frac{\lambda}{\eta_1}}{2\pi\sqrt{\sin^2\theta_i - \left(\frac{\eta_2}{\eta_1}\right)^2}}$$



- Prisms convert outer AOI to Inner AOI

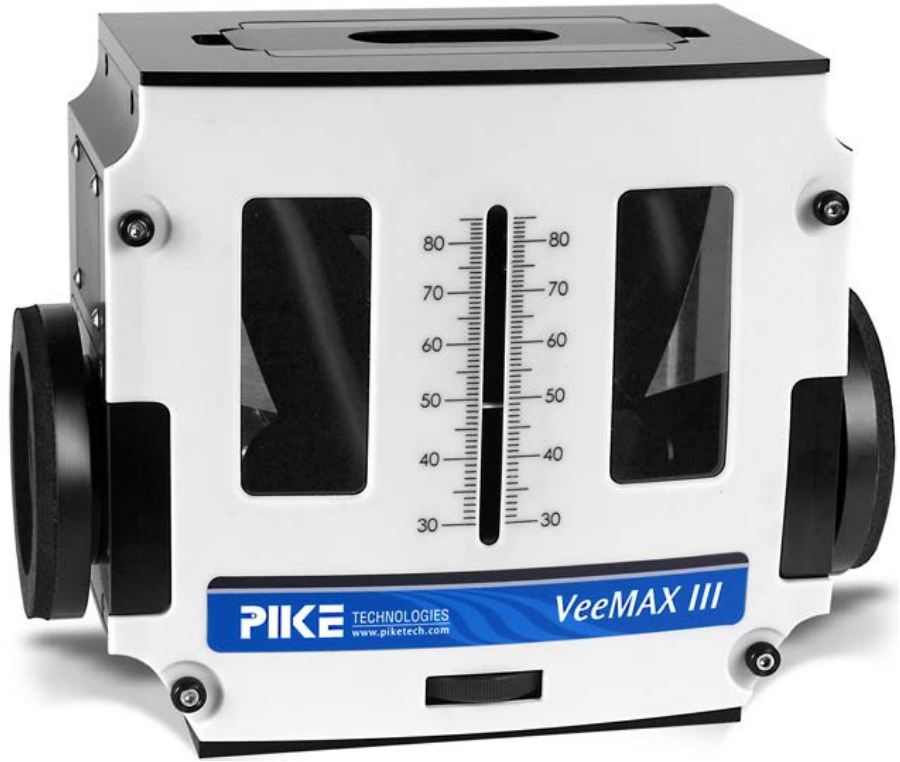


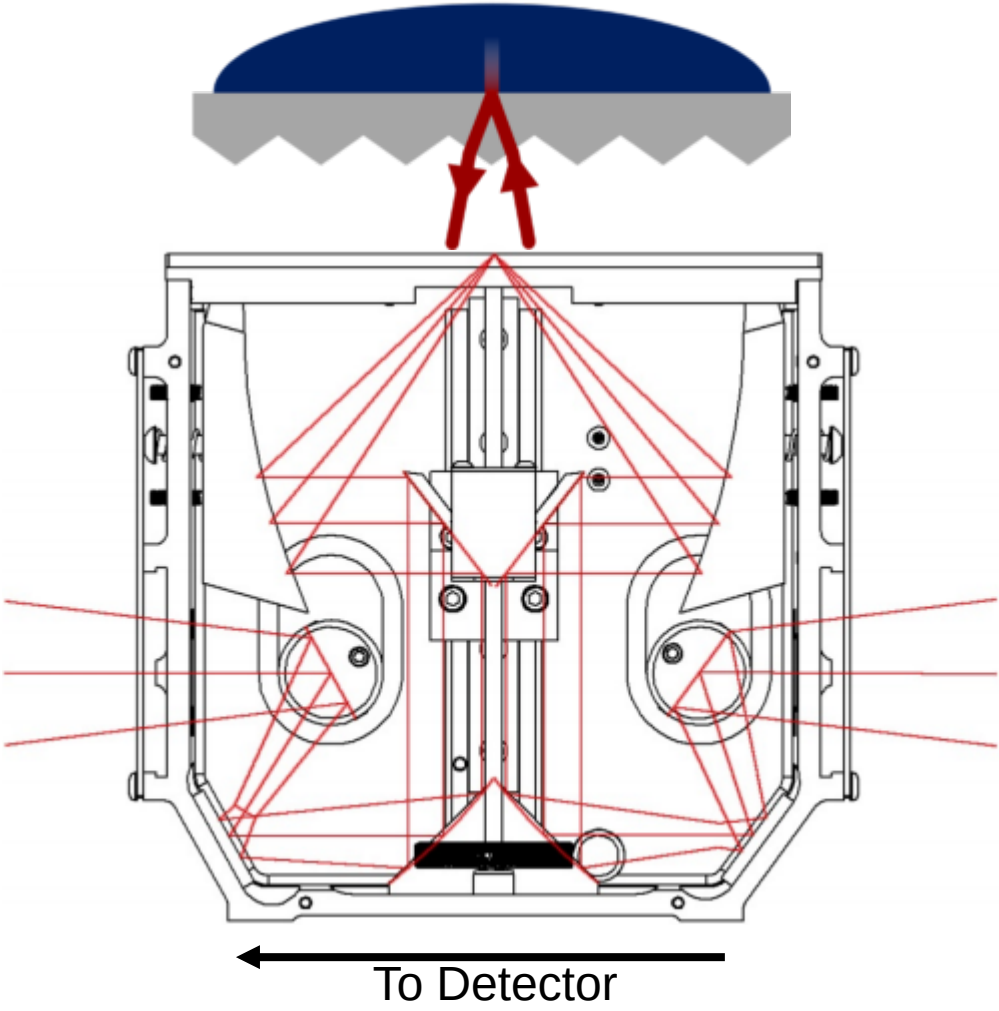
Angstrom Engineering 6-pocket E-Beam Evaporator



- The Angstrom E-beam is for thin-film evaporation in the range of 5 nm to several microns thick.
- A maximum of six different materials can be evaporated in one pump-down cycle, from the six pockets electron gun.
- The thickness of the metal is monitored in-situ by a thickness monitor.
  - Substrates up to 6 inch diameter can be attached to the substrate holder.
- The sample temperature usually does not exceed 50 °C.
- The following materials are available: Au, Pd, Ge, Ni, Pt, and Ti.

Pike VeeMax III Variable angle Specular reflectance Accessory



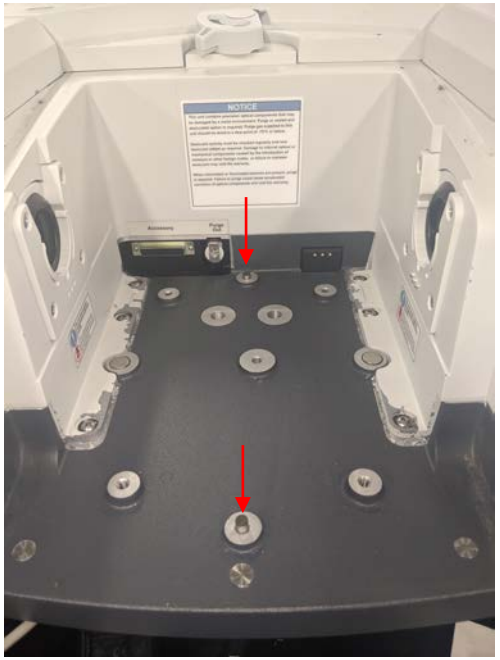


Mounting the VeeMax in the is50

Remove transmission mounting bracket:

- 4 1/8th" hex screws located in corners

Unlatch Chamber Cover



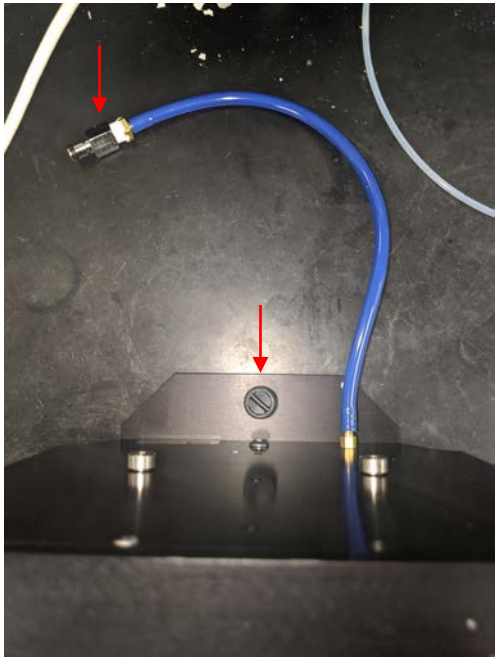
Note bracket positioning posts

Mounting the VeeMax in the is50

Install gaskets on sides of sample chamber

Install ATR bracket in sample chamber

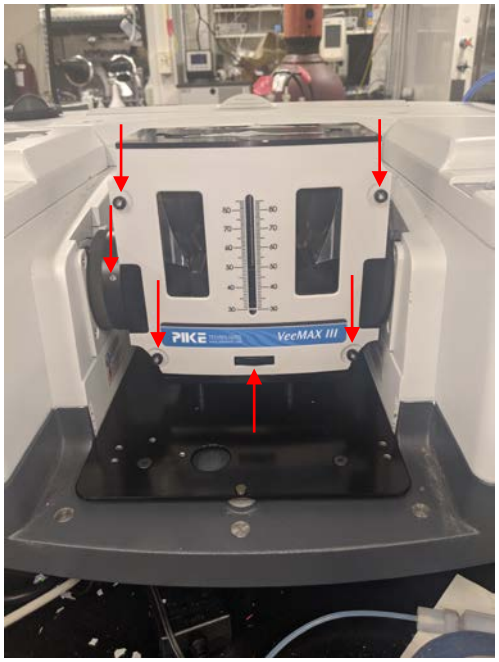
- Attach accessory connector to back of bracket
  - 7/64th" hex screw



Connect purge line to back of sample chamber

- Note mounting thumbscrew

Optimizing Signal



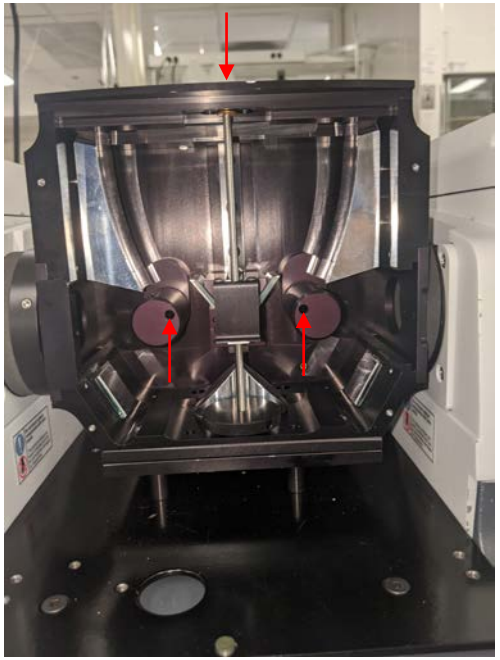
Install VeeMax and tighten thumbscrew

- Adjust gasket to seal against sides of chamber
  - 2 3/32nd" hex screws
- Set reflectance angle to 45°
- Remove faceplate
  - 4 3/32nd" hex screws

Open bench in Omnic

- Monitor peak to peak

Adjust rotation of input mirror
Adjust rotation of output mirror

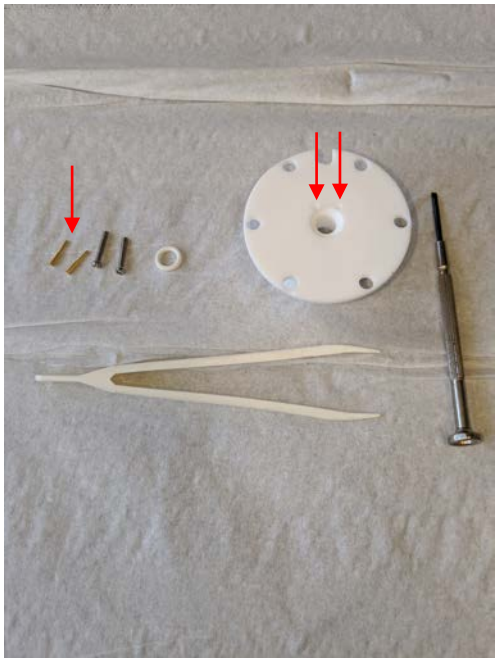


Adjust tilt of input mirror
Adjust tilt of output mirror

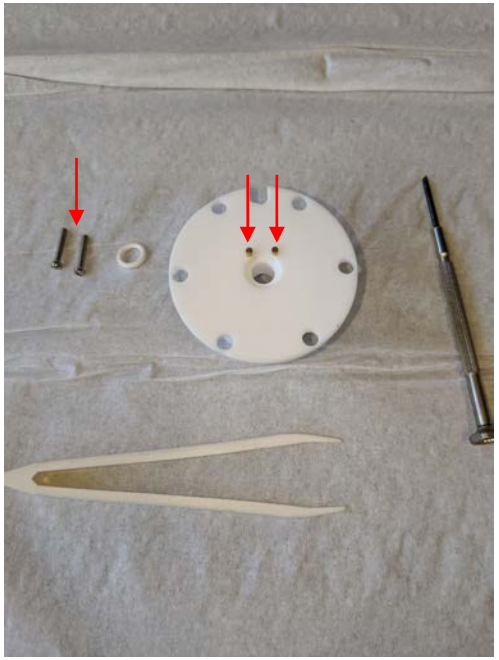
- 3/32nd" hex wrench

Iterate until peak to peak is optimized

Assembling the Spec-E-Chem cell base



Install pogo pins in bottom of Teflon cell base

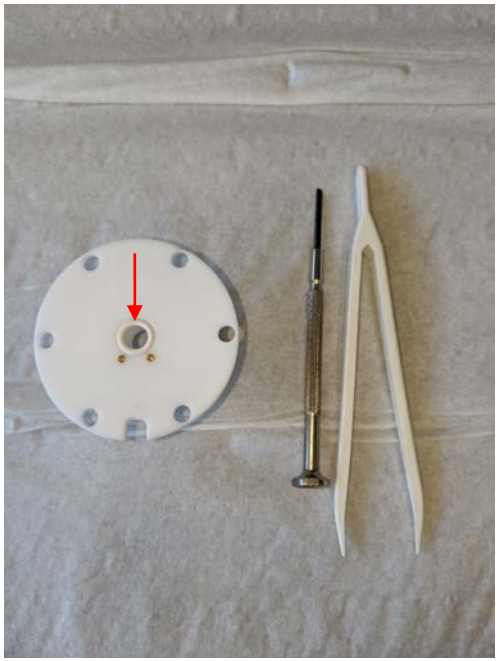


Install contact screws in side of cell body

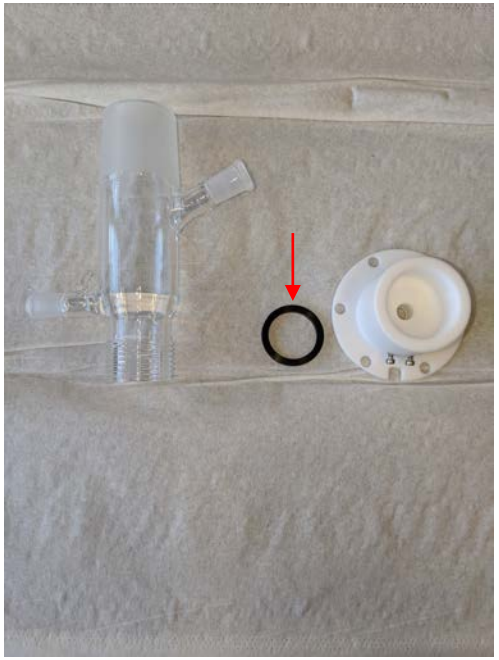
- Tighten until contact is made with pogo pin then back off ½ turn

Depress pogo pin and tighten screw to keep flush with cell bottom

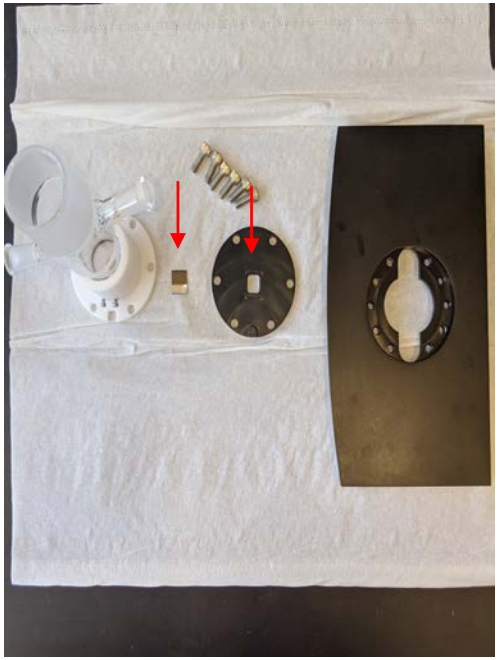
Assembling the cell body and sample



Install Teflon gasket in bottom of Teflon cell base



Install Viton gasket and cell body in base



Place ATR crystal in Delrin holder

Mounting cell to baseplate

Align crystal holder to proper orientation based on crystal grove direction

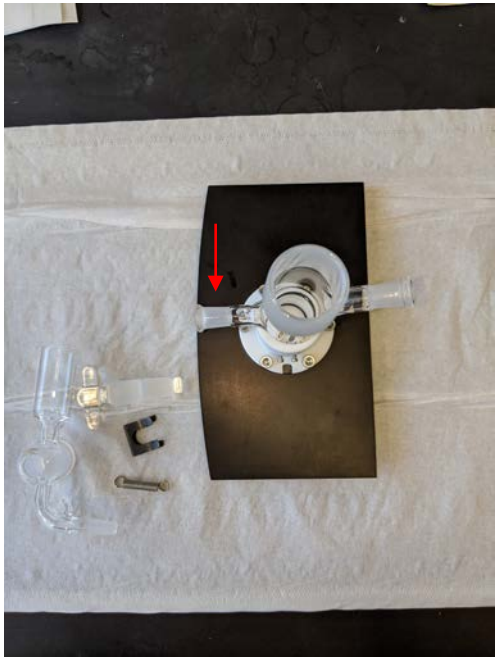
- Place Spec-E-Chem cell on base plate

DO NOT APPLY PRESSURE!!!

Tighten screws in star pattern

- 9/64th“ hex screw

DO NOT OVER TIGHTEN!!!

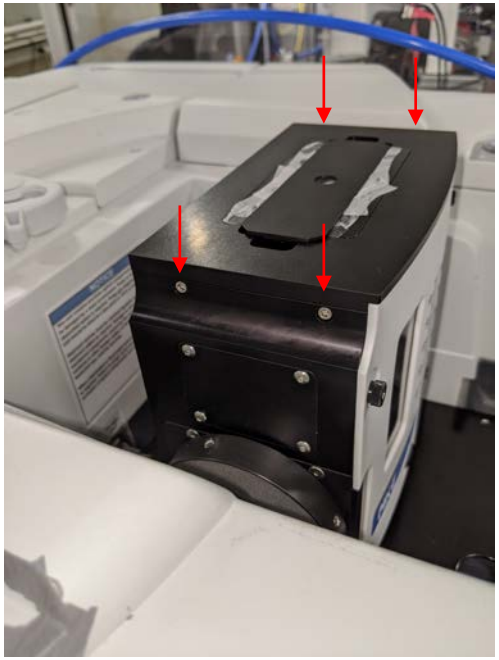


Assemble and attach reference bridge to cell

Assembling the rest of the cell

Install cap, bubbler wand, purge inlet, counter electrode holder, and exhaust bubbler

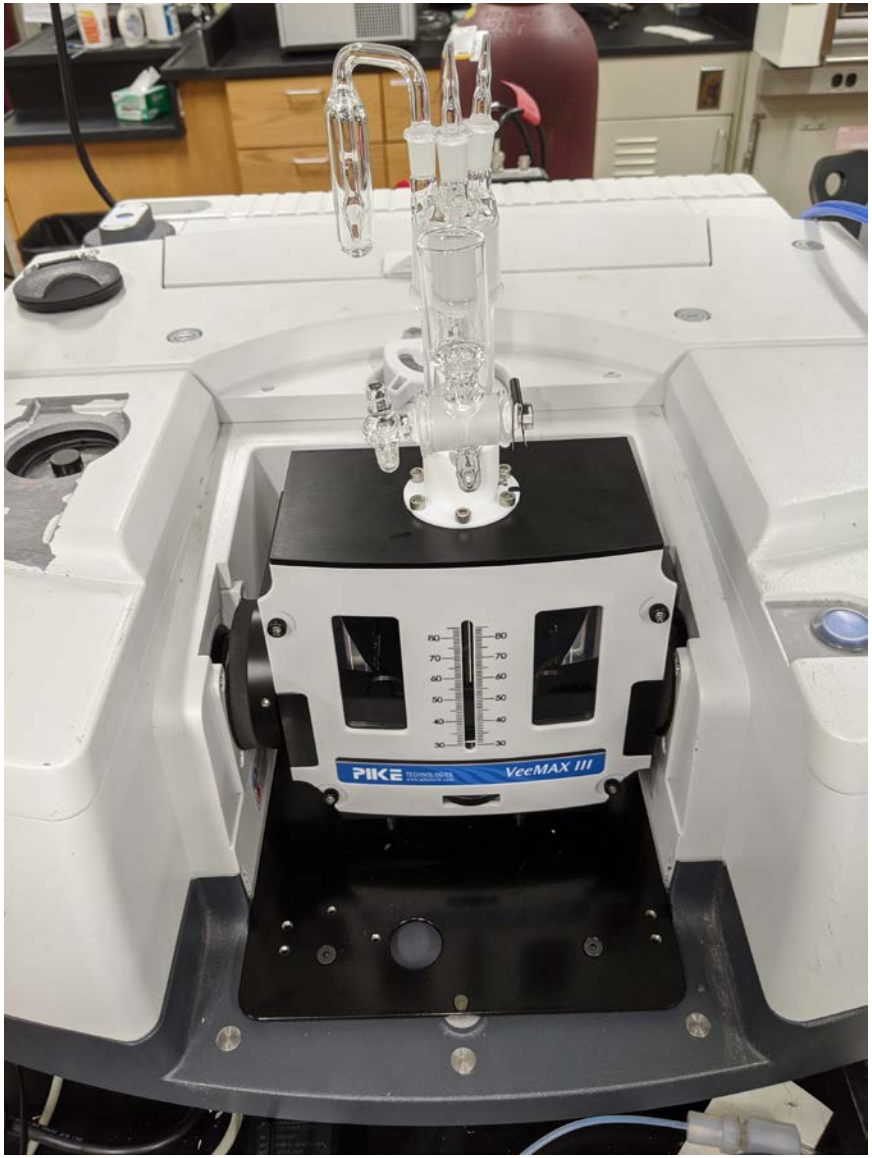
Leak test before installing on VeeMax



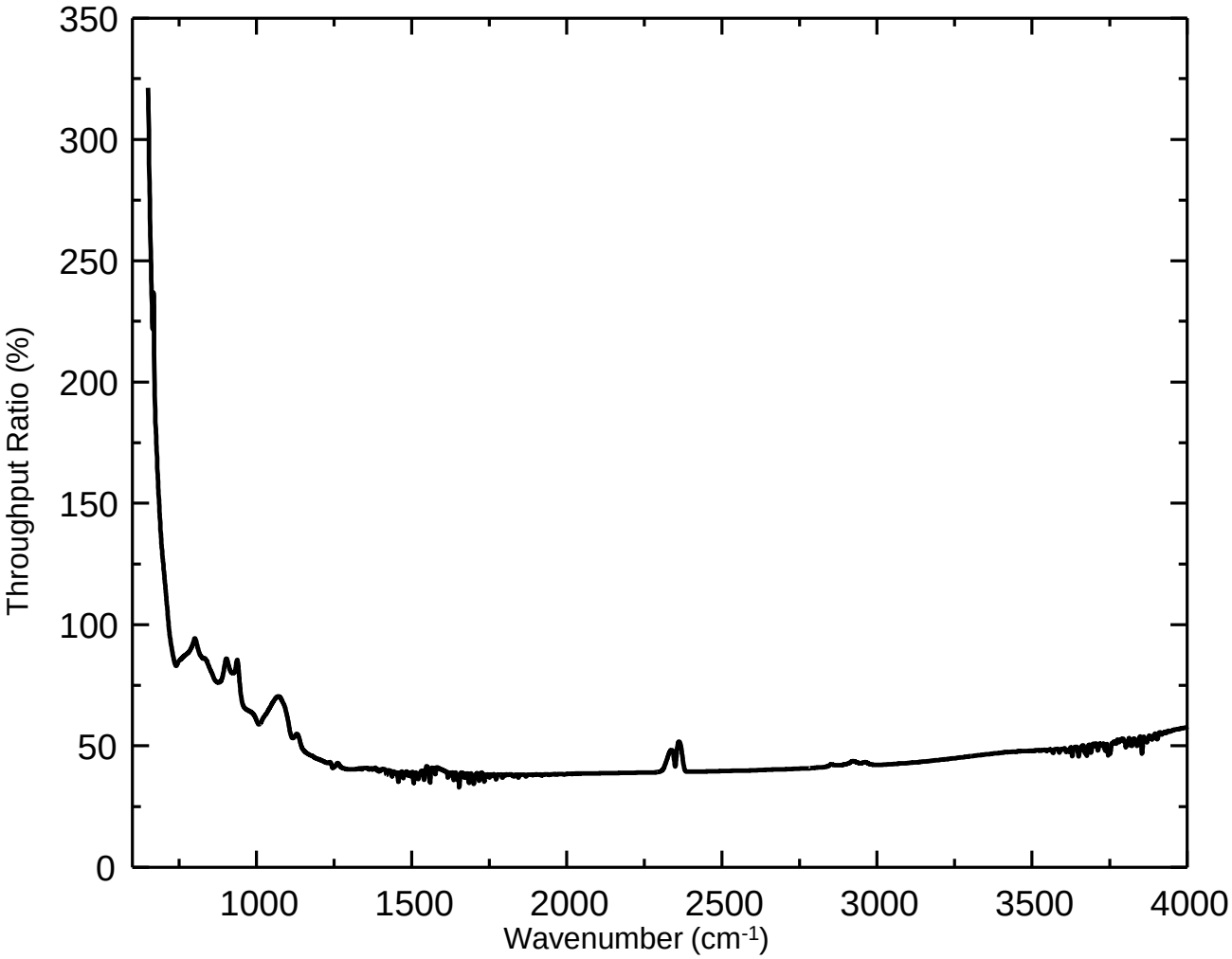
Remove reflectance baseplate and replace with Spec-E-Chem Plate

- 4 3/32nd“ hex screws

Final Assembly

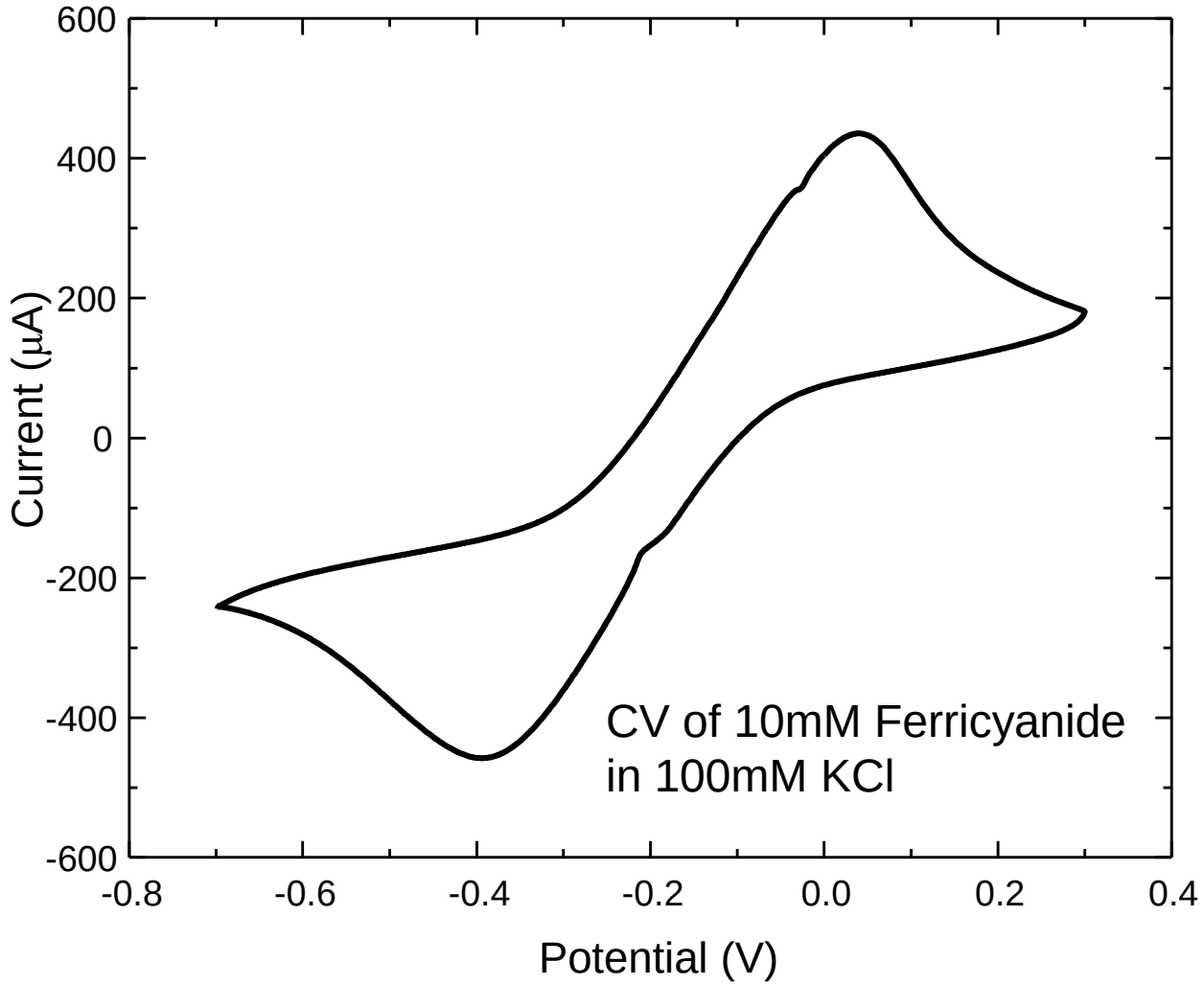


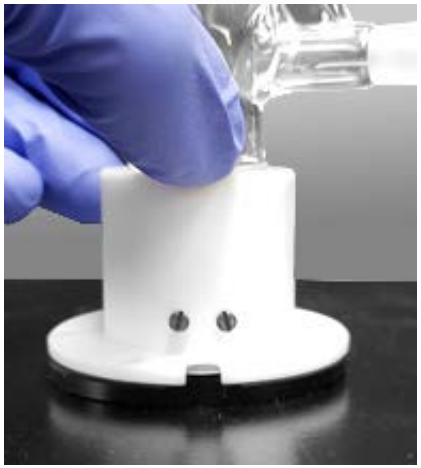
Check throughput ratio



- Collect open beam transmission spectrum
- Collect transmission spectrum with VeeMax unit in place
- Ratio and compare to Accepted values
  - 40-60%

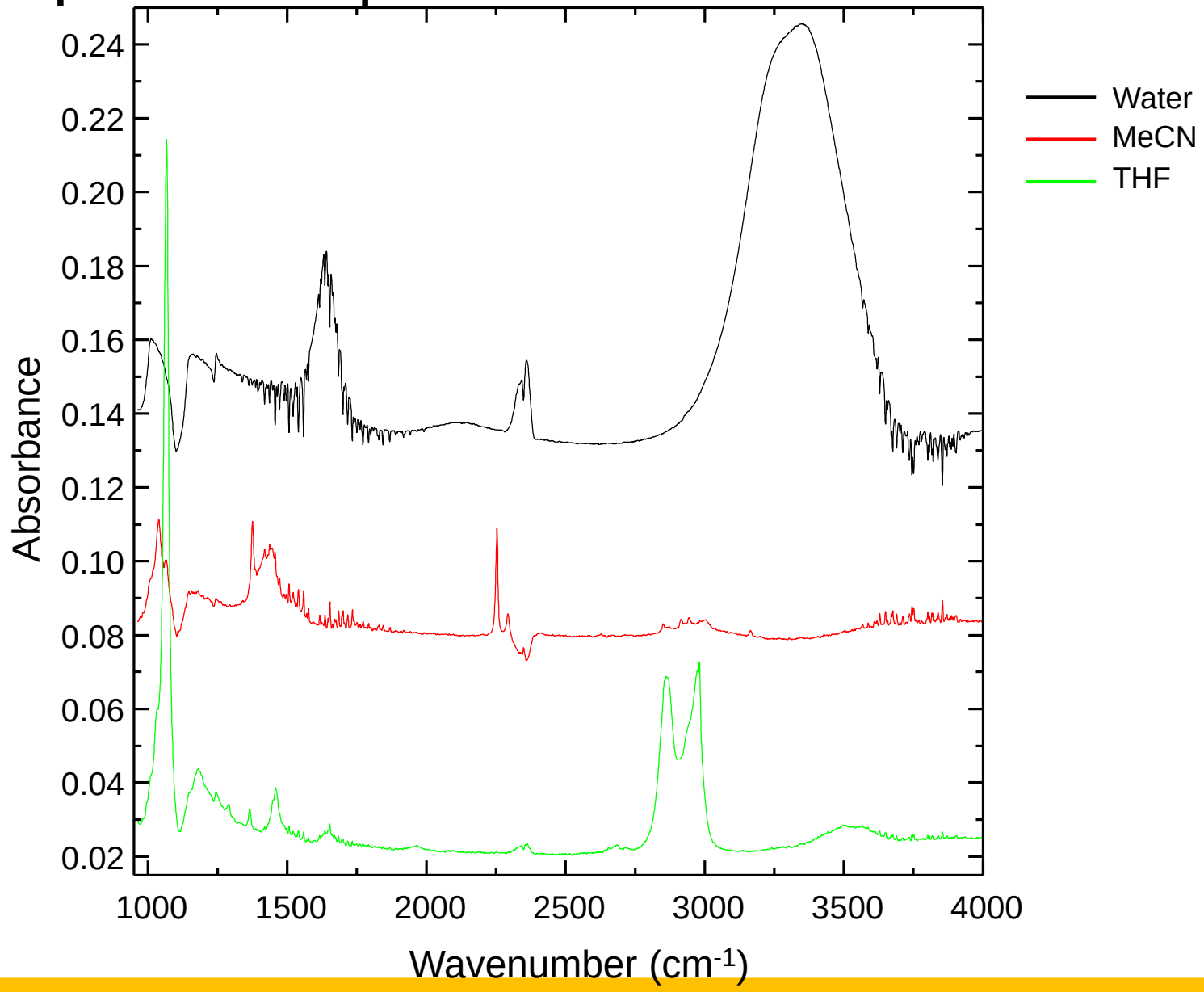
E-chem in the ATR Cell



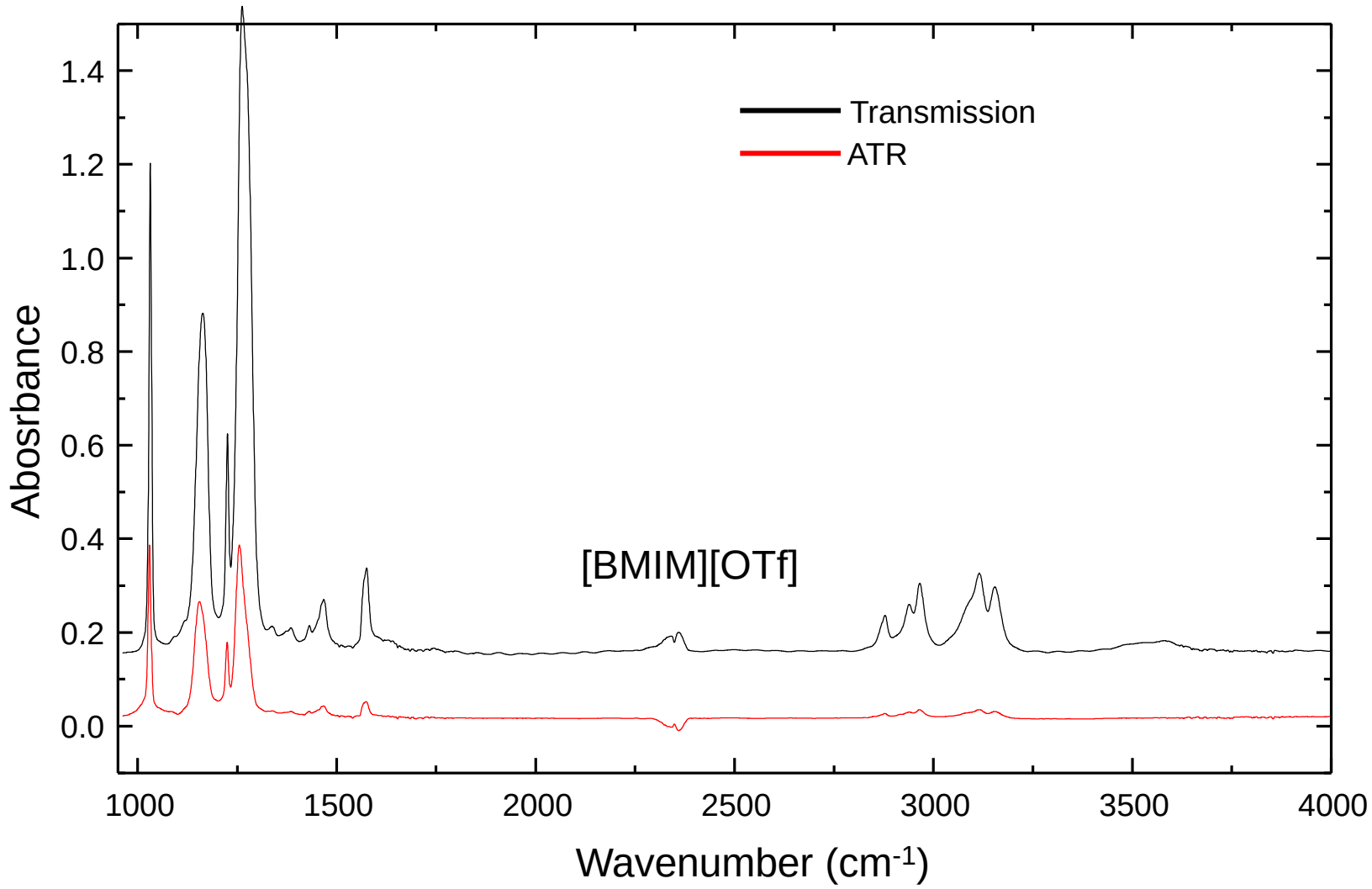


- Contact made via spring loaded Au Pins
- Turn screws ½ turn counter clockwise to make contact

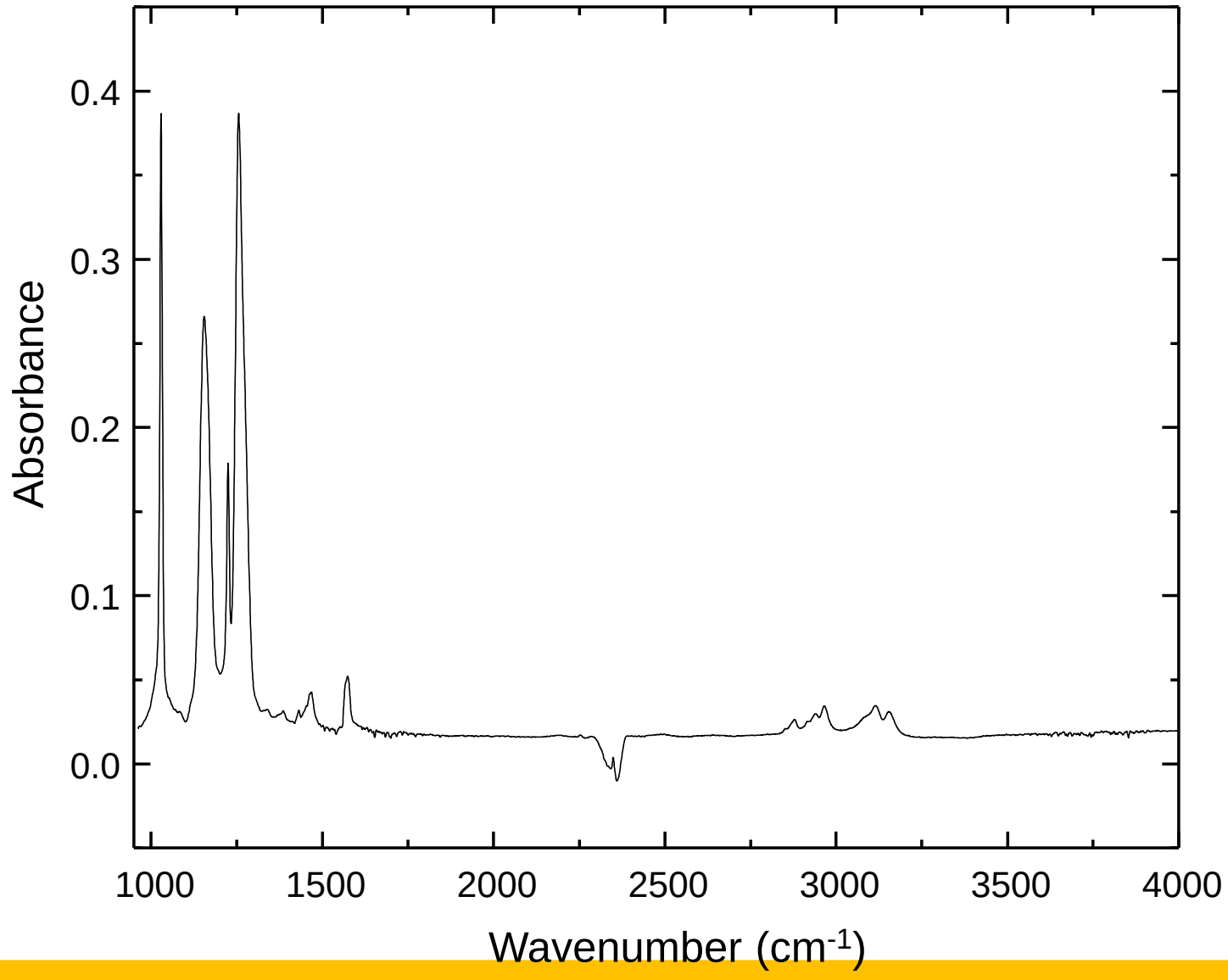
Example ATR Spectra



ATR vs Transmission on ILs



ATR on [BMIM][OTf]



Troubleshooting:

Contacts:

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- Jenni Briggs: briggs@piketech.com

Jackfish:

- Tyler Mohart: mohart@jackfishsec.com

E-beam:

- Connor Grierson: connor-Grierson@uiowa.edu

Irubis:

- Anja Mueller: anja.Mueller@irubis.com

Manuals:

Veemax III:

- https://www.piketech.com/files/user-manuals/VeeMAXIII_Manual.pdf

Jackfish:

- https://www.piketech.com/files/pdfs/PIKE_Jackfish-Spectroelectrochemical-Cell_Data_Sheet.pdf
- https://www.piketech.com/files/pdfs/PIKE_Jackfish-Spectroelectrochemical-Cell_Data_Sheet.pdf

Crystals:

- https://irubis.com/wp-content/uploads/2019/01/IRUBIS_ProductBrochure_ATRCrystals_January2019.pdf

Articles:

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https://pubs.acs.org/doi/10.1021/acsanm.8b02155

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