## SOP for ReactIR

- 1. React IR should be plugged in for 24 hours **prior to operation** to stabilize the voltage.
- Using the funnel provided, fill MCT Detector with liquid nitrogen until the LN<sub>2</sub> overflows prior to use. Cover the LN<sub>2</sub> fill using the plug to limit N<sub>2</sub> evaporation and reduce water buildup.(lasts ~24 hours).
- 3. Plug Probe into IR Max (IR Source). You are responsible for determining if the probe will withstand your reaction conditions. Check the compatibility of your reaction with diamond and gold. If either will react in your solution, do not use the ReactIR, for example:

## Do not use IR Max with strong oxidants such as piranha or nitric acid

- \* Reaction pressure should be below 60 bar.
- \* Reaction temperature limited to 180 °C
- 4. Place the probe in the reaction solution such that the tip of the probe is submerged (use stand to hold probe upright and suspended). Be sure that you can perform all additions without moving the probe because this will affect your measurements When actively taking IR spectra for your experiment do not move the probe. Make sure to adjust this prior to your experiment.
- 5. Open the software "IC IR".
- 6. Hit Quick Start

Recent	New		
MF_101.101421.4 Depriment - Samples : T8 Cultors(N)Bootments(X IR (potentients(N)TReyUsies MF S LS -273 TBA(Cu2(D)pp-np)(N3)) + PhCCH in THF Bagement - Samples : 277 Cultors(N)Bootments(X IR Speciments(V)TReyUsies MF G Exp 2021-10-13 11-44 Bootment - Samples : 80 Cultors(N)Bootments(X IR Experiments(V)TReyUsies MF G pen other files	Quick Start Clone Experiment	Quick-Start Create an experiment with default settings Clone Experiment Use settings from a previous experiment From Template Use settings from a pre-defined template	To start a new experiment, choose 'Instrument' on the left panel and configure an instrument.
	Other Documents	Other Documents Create Spectra Sets, Trend Sets, Univariate or Multivariate Models	

- 7. View IR signal
- 8. If the alignment is off, try to adjust the probe until the alignment is within the window. If this does not work, optimize the alignment by adjusting the small screw on the IR source.
- 9. Set the duration of experiment and sample interval (note: experiment can be manually stopped at any time)
- 10. Set range of collection.
- Select number of background scans (~32 -128 scans depending on reaction). This will determine the number of scans you use in your experiment; be cognizant of your reaction timescale.
- 12. Once, probe is in a fixed position, collect the background without solvent, in solvent or reaction solution.
- 13. You may now add your reaction solvent, and begin scanning. Open the notes tab and pin this first spectrum. Name this spectrum, and the solvent subtraction feature will become available in the toolbar ribbon. Select this and your solvent spectrum of choice and the software will automatically subtract this spectrum from all spectra collected.



- 14. If you are collecting the spectra of other reagents, add them sequentially, collecting a spectrum after each addition. Using the spectrum subtraction tool, you will be able to extract the spectrum of each component.
- 15. When you are finished with the experiment, thoroughly clean probe with solvent such as acetonitrile or methanol.
- 16. Label spectra accordingly in order subtract scans using data tools.
- 17. To track change in peak:
  - a. Take baseline of system.
  - b. Double click on peak of interest
  - c. Select region on interest by narrowing or expanding limits.
  - d. Subtract solvent spectra from data of interest
  - e. Apply to take second derivative of system

