Mettler-Toledo ReactlR

Instrument Care

The silicon probe is very sensitive to abrasion. Use extra care when cleaning and only wipe with Q-tips and Kim wipes.

The fiber optic cable is very brittle and can be broken by <u>dropping</u> or <u>bending</u> beyond the allowable radius. Moving the cable also changes the signal. The hood is set up so that <u>users do not need to move the probe at all.</u>

Attach a flask to the probe adaptor, then raise and lower the stir-plate as needed using the lab jack; never raise or lower the probe.

NEVER use strong acids or bases to clean and NEVER sonicate the probe.

Sample Compatibility

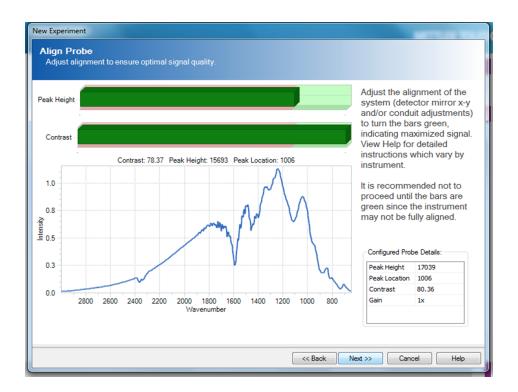
All reagents must be compatible with silicon, gold, and Alloy C22:

- o No acids, aqueous bases, and corrosive reagents.
- \circ <u>No</u> cyanide or azide ions, which will react with the gold seal.
- All reagents <u>must</u> be reasonably soluble in organic solvents or we will never be able get them off the probe.

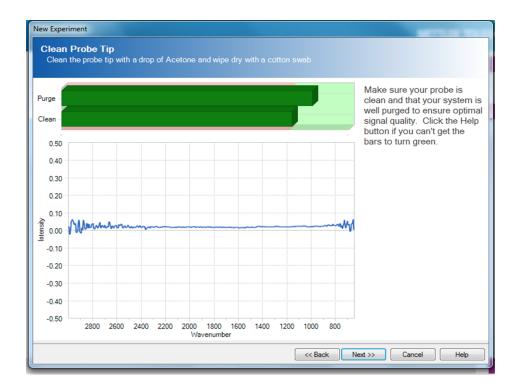
Running a Reaction

- 1. At least 20 minutes prior to starting your reaction, fill the detector with liquid nitrogen using the special funnel.
- 2. Open the ICIR program and click on "new experiment." This action will open a Wizard. Do this step <u>before</u> setting up your reaction flask.
- 3. The Wizard will prompt you to pick a sample name and template. You can use a previous experiment as a template, and modify it as needed later.
- 4. The Wizard will go through several screens:
 - a. Nothing needs to be changed on the Instrument Configuration screen.

- b. Typically we use an 8 h experiment duration and collect samples every 15 s, but these values can be changed as needed. Both parameters can also be adjusted while the experiment is running.
- c. The Wizard will ask if you want a "reference spectrum." This spectrum is an air background that will be displayed on screen during your experiment. Select "no reference spectrum needed".
- 5. The Peak Height and Contrast screen indicates how clear a signal the IR is receiving by looking at the signal for air. If both bars are green, continue with the Wizard. If either bar is red, try adding more liquid nitrogen. If you cannot get them into the green, find the group member responsible for the instrument and ask them for help.



6. The next screen checks how clean the probe is by comparing it to a signal acquired previously with a clean probe. The spectrum should appear flat with some noise at either end. If either bar is red, gently clean with DCM and acetone, using Q-tips, until they are green. If you can't get the probe clean, try soaking in warm (not boiling) THF or chloroform for several h. If it continues to be a problem, contact the group member in charge of the instrument. When they are clean and in the green, click "next."



- 7. It is now time to take a solvent background scan! Set up your reaction flask, add the reaction solvent, and cool or heat as needed. Click "Take background scan" to acquire a background, and click "finish" when it is done. The setup wizard is now complete.
- 8. To start your experiment, click on the "start" icon in the upper left of the screen.
- 9. The IR will begin acquiring scans over a range of 2500-650 cm⁻¹. To tell the program to monitor a specific peak during the reaction, click on the peak using the crosshairs and select the "add peak" icon. Then, select the peak and click "edit." Change the definition from "height to zero" to "height to single point baseline." This action ensures that your peak height is accurate even if the spectrum drifts from the baseline. Select a point in a flat region to be the baseline point. The screen will show the trend in peak height over time.



- 10. When your experiment is complete, click the "stop" icon. Remove your reaction flask from the probe, and immediately clean the probe with DCM and acetone, or an appropriate organic solvent to dissolve your materials. <u>Do not</u> allow anything to dry on the probe!
 - a. If you have to do an aqueous quench (acid, basic or neutral) or use another incompatible reagent, remove your reaction from the IR and pour into a separate flask. <u>Do not</u> expose the probe to the quenching agent!
- 11. Raw data is recorded continuously throughout the experiment. Data points for individual spectra or trends can be exported to Excel by copying as text and pasting into Excel.