

**LA-ICP-TOFMS Operating Instructions**  
**Version 1.2**  
**2/10/14**

Preparing instruments to run:

- 1) Connect the laser “out” line to the torch with a clamp if it is not already connected. The spray chamber may need to be removed to do this.
- 2) Close the lid of the ICP-TOFMS. A fan will start when the lid is closed.
- 3) Turn the chiller on and allow it to reach 15 degrees C.
- 4) Open the Helium tank. Check Helium and Argon gas levels. Replace tank(s) if pressure is 500 psi or less.
- 5) Turn laser power supply and laser on (if necessary).
- 6) Turn on the Cryo-cell (if desired) and set it to the desired temperature by using the green buttons on the front.
- 7) On the laser computer (left-hand computer), double-click the “Digilaz III” icon to start the laser software. Wait a few minutes for it to fully load. Once it is loaded, do not maximize the window. This can lead to a software crash. If the software won’t load properly, close it and turn off the laser and laser power supply. Re-boot the computer. Turn on the laser and let it warm up for a minute, turn on the laser power supply, turn on the computer, and re-start the software.
- 8) On the TOFMS computer (right-hand computer), double click the Optimass 9500 icon to start the TOFMS software.
- 9) Click the “Ready” icon in the Optimass software. This will light the torch and turn on the interface turbo pump. The green “Start” button will illuminate when the TOFMS is ready to analyze samples.
- 10) In the “Gas Management” tab of Digilaz III, set the Helium flow to 100 and click the “Set Helium Flow” icon. Increase the flow in increments of 100 up to 500. If the torch is extinguished during this process, set the flow to 0, click the “Ready” icon in the Optimass software, wait for the green “Start” button to illuminate, then slowly bring up the Helium flow again.

Tuning

Digilaz III:

- 11) Place a sample of NIST 612 glass in the laser sample cell.
- 12) In the “Light Controls” tab, set the “Upper Light Controls” to the desired intensity, either by clicking in the bar, or by using the plus and minus buttons.
- 13) Focus the sample by using the green up and down focus arrows. The larger arrows are coarse focus and the smaller arrows are fine focus.
- 14) Find the desired area to be ablated by using the blue X/Y arrows to move the sample around.
- 15) Select “Scanning” in the Method Selection drop-down menu in the General Operations tab. Draw an area to be ablated on the NIST 612 glass that will take approximately 2-3

minutes. The time of the ablation is displayed in the bottom left-hand portion of the window. Click “Start Ablation” when the Optimass 9500 parameters have been set.

#### OptiMass 9500:

- 16) Click the “Instrument” icon, which is located at the very top along the right-hand side.
- 17) Click the “ICP Tune” tab. Right-click somewhere in the open tan area, select “Open Panel Data”, and select the most recent laser ablation tune. This will re-position the torch and drop the applied power from 1200W to 900W.
- 18) Click the “Acquire” tab, and set the Acquisition time to 0.5 seconds. Set the Number of Spectra to 1. Set acquisition type to “Internal”.
- 19) Click the “ICP TimeScan” tab. Make sure mass 115 (Indium) is entered in the Time Scan window, along with any other desired masses. Under Start Acquisition, click the “Cont.” button.
- 20) Monitor the intensity of the signal from the NIST 612 glass. The min/max signal range is given for each mass. If tuning is necessary, the XYZ positioning of the torch can be modified by using the slider bars on the left side of the window. The gas flows can also be optimized by using the respective slider bars. If you want to clear the tuning field and have it start over, right-click in it and select “Clear”. Click “Off” under Start Acquisition to stop the acquisition.
- 21) Click the ICP Tune tab. Right-click in a vacant tan area, and select “Save Panel Data As...”. Enter the desired name of the tune and click Save.
- 22) Close the Instrument window.

#### Creating an experiment in Optimass 9500:

- 23) Click the green “Method” icon on the left-side of the window. Click the “New” icon directly above it to start a new method.
- 24) In the method notebook displayed, click on the “Instrument” tab on the right side. Right-click in the gray field, and select Method Properties. In the Measurement tab of the Method Properties window, select the “Transient Studies” box. Select the “Full Mass Range” radio button. Click OK.
- 25) In the “Instrument” tab, entered the desired acquisition time for the method. This may not be known until a suitable ablation area has been drawn on the desired sample. Include extra time in the acquisition time to allow for a gas blank(s) to be collected.
- 26) Click the “Description” tab. Enter the requested information, if desired.
- 27) Click “File-Save As...” from the top drop-down menu and save the method in the desired location.
- 28) Click the “Samples” icon directly beneath the Method icon. Enter “Sample” in the Measurement field, and the desired sample name in the Label field. Click “File-Save As...” from the top drop-down menu and save the sample information in the desired location.
- 29) Click the “Results” icon, located beneath the “Analysis” icon. Click the “New” icon to create a new Results file. DO NOT click “Results-Clear Results” from the top drop-down menu. This will permanently delete any files in the currently loaded Results file. If you do click “Results-Clear Results”, a pop-up window appears asking if you are sure

you want to continue. Click No and the results will not be deleted. Click “File-Save As...” from the top drop-down menu and save the Results file in the desired location.

#### Collecting data:

- 30) Make sure the desired method, sample information, and results files are loaded in the Optimass 9500 software.
- 31) Place the sample in the laser sample cell, focus, and find the desired ablation area.
- 32) Wait a minimum of 30 seconds before ablating to allow the sample cell to purge. If the cryo-cell is being used, wait about 5 minutes before ablating to allow the sample time to cool.
- 33) Draw the desired ablation area(s) on the sample.
- 34) Make sure the acquisition time in the Optimass 9500 method is long enough to collect all of the ablation data plus some extra time for a gas blank. If not, correct this and re-save the method.
- 35) Open the “Acquire” tab, and set the desired number of spectra (typically 4 per second of acquisition time). The acquisition time can also be manually entered, but this is automatically downloaded from the method when the analysis starts. Also, VERY IMPORTANT: set the acquisition type to “External”.
- 36) Click the green “Start Analysis” button at the very top center of the Optimass 9500 window. Wait 5 seconds, and click “Start Ablation” in Digilaz III.
- 37) When the TOFMS is acquiring data, you should hear a light “clicking” sound from the computer. The spectrum in the Results window will not appear until the sample has finished acquiring.
- 38) More than one sample data file can be stored in a Results file, although this is not recommended. The Results window will eventually crash when too many data files are stored in it. It is recommended to create a new Results file for every sample.

#### Processing data:

- 39) Highlight the desired sample to be processed in the top window of the Results file.
- 40) Expand the transients window in the Results file so that a specific mass can be selected. To do this, place the cursor in the window, left-click and hold the mouse, and draw a box with the red cross-hairs across the window to expand it. This can be done multiple times to further zoom in. To unzoom, double left-click the mouse in the window.
- 41) Click on (or just beneath) the desired mass in the window. The graph at the very bottom left of the Results window (Time vs. counts at the specified mass) should update.
- 42) To set integration areas, region marks must be added. To do this, locate the desired spot for the region mark on the graph with the green cross-hairs, right-click the mouse, and choose “Add region mark”. Do this for as many regions as desired. A set of region marks can be saved by right-clicking in the window, selecting “Save Regions As...”, and saving the regions to a specific filename. These can then be called up in the future by right-clicking in the window, selecting “Load Regions...”, and selecting the proper filename.

- 43) When regions are created, the right-side of the Results window displays them. The start and stop time of each region is listed, as well as the number of counts in the region. This data can be copied and pasted into Excel for further processing.

Shut down:

- 44) Click the “Standby” icon at the top center of the Optimass window. This will extinguish the torch, turn off the interface pump, close the slide valve, and place the instrument in a standby state.
- 45) After the torch is out and the interface turbo speed reads 0, turn off the chiller and lift the lid of the ICP-TOFMS.
- 46) Turn off the Cryo-cell if it was on.
- 47) In the “Gas Management” tab of Digilaz III, set the Helium flow to 0 and click the “Activate Helium Flow” icon. A click will be heard as the Helium is turned off.
- 48) In the “Light Controls” tab of Digilaz III, set everything to 0. Close the Digilaz III software.
- 49) Close the Helium tank.
- 50) Leave the Instrument Status window open.