# **PHI nanoTOF II Operation Notes**

**DO NOT touch anything that goes into the chamber with bare hand.** Wear a glove and use clean tools.

ALWAYS INSPECT THE SAMPLE FROM THE SIDE TO ENSURE IT DOES NOT EXCESS THE HEIGHT AND BOTTOM OF THE FRAME.

Read "sample platen" file for how to use them correctly.

If turn off ELECTRONICS POWER to reset system, need to <u>wait for at least 2 min</u> for capacitors inside Power Protection Unit to discharge before turn it back on. Otherwise, DSI/MS2 will go into shutdown status and cannot be recovered.

Use **TOF** session  $\rightarrow$  **Data Manager** page  $\rightarrow$  **Directory** tab (or right click on Acquisition Start button for **Data Manager** page) to navigate to your own folder in **Z** drive. Never save files in **C** drive.

All the files can be removed without prior notice so make your own copy.

**Do not save/overwrite referencing settings.** Add your name in front the setting name before save your own settings.

For each gun, *Load* required setting before put it to *Ready* state.

Unless during sample exchange, always leave the stage to the *Main Chamber*. **DO NOT LEAVE THE STAGE IN** *INTRO*.

When finished, double check the bottom status bar for instrument at *ZERO* and LMIG, GCIB, E-Neut and Gas at *Off* state. For C60, it should be either at *Idle* or *Off* state. Right-click on the status of guns allows quick changing their state.

### **GENERAL OPERATION**

- In normal condition, FIELD OK should lid on SC20 field canceling system and the total field < 0.1 mG. If the field was tripped in any axis and failed to reset automatically, put it to standby then back on from the remote or press reset on the controller.
- In normal condition, vibration isolator should enable with yellow light lid. No red light should be flashing.
- For analysis position, following items can be used to check/tune the Z height.
  - The Chamber image should be at best focus and should be at a close proximity of the Photo image.
  - ➢ SED image should be at best focus.
  - With 0 Raster Size and Detector Scanning off, the spot in *Detector* image should be in the box region.
  - With DEM off and maximum Raster Size, *Total Ion Image* should have centered circular FOV and the FOV of *SED Image* should just touch the right-edge.
- With default marker size of 50, the box marker on *Chamber* image and *Detector* image is  $\sim$ 250 µm and **Scan Size** 4, respectively.
- For sample navigation, click on live *SED* or *Chamber* Image will move stage to center the click position. It is also possible to drag to cross marker on *Photo* image or right click on

*Photo* image to drive to click. Note that the behavior of *Photo* image is different from live images.

- **Be careful not to click on image window and their control during acquisition as** sample will move and parameter will change.
- It may be possible to use C60 blanker/analyzer setting for LMIG work, but peaks lower than set m/z may appear. On the other hand, if mistakenly use LMIG setting for C60, low mass region will be cut.
- Verify the **Raster Pattern** is *Scatter* and **Detector Scan Mode** is *Fast* in **Spectrum**, **Phased** and **Mosaic** tabs in **TOF** session → **Properties**. The **Raster Pattern** in the **Properties** tab of **Image Tool** should be *Flyback* and **Sputter Tool** should be *4-Fold*.
- During first few cycle of automatic operation (especially depth profile), check the actual running parameters in **Hardware** session in each phase to make sure desire parameter is used.
  - ➢ Right-click on TOF session → Spectrum / Profile / Map page → Application Log (Acquisition) can also reveal what system is doing in each step.
- In case of software restart, do not turn off electronics unless LMIG is at off state already. Right-click on the instrument status bar to put the system to *RECOVERED-SETTING* can quickly load parameters used before normal shutdown of software. This setting is not meant for normal startup so never use it unless during software restart.

#### A. Sample loading

- 1. Choose sample platen: back-load, front-load, hot/cold and heating.
  - Only standard back-load platen is available for public use and it should be kept inside main chamber when not in use. The size of specimen is limited to be smaller than 2" in diameter and 10 mm in thickness.
  - ➤ With the built-on spacer on the top surface of back-load platen, the sample surface should not extrude and touch anything when put the platen up-side-down. When the sample is load correctly, the sample will have the correct Z-height at stage Z position of 3 mm (2 mm from extractor lens).
- 2. Inspect the sample platen from its side and ensure samples are lower than the frame and are flat with respect to the opening of covering metal plate.
- 3. In System session, drag stage icon to *Intro* and then right-click to icon to *Backfill Intro*.
- 4. Put the sample platen into intro chamber (Faraday cup should be at opposite side of the main chamber and never be touched) and secure 4 screws with 2.5 mm hex driver using uniform force. Do not tighten the screw, just snag it.
- Create Sample by right-click stage icon or Acquisition Start button allows quick selection of platen type and filename/directory (identical to TOF session → Data Manager page → Properties).
- 6. *Take Intro Photo* by right-click stage icon in *Intro* area or press *Photo* in **System** session  $\rightarrow$  **Camera** page before closing the intro chamber.
- 7. Ensure the intro cover is at correct position and does not move freely, then *Pump Intro* by right click on the stage icon at **System** session.

➢ When *Pump Intro* is finished, the V4 will open automatically and its icon turns to green.

8. At **System** session, drag the stage icon to main chamber to complete the sample loading. Use the side-view camera to observe the sample transfer. Always prepare to stop the transfer process if the stage is going to hit lens inside. The HV Enable should become green in the **System** session when transfer is complete.

- To see the *Chamber* image, turn up *Illumination Power* using the slide next to *Chamber* image or from Hardware session → Analyzer page → Properties → Spectrometer tab if necessary. The value is usually <1.</li>
  - > If the image does not show correctly, enter 1 to the magnification factor.

### **B.** General operation

- In TOF session → Data Manager page → Properties → Directory tab, navigate to your own data folder at Z drive. Use Filename tab to define filename and sample-related comments if necessary.
- 2. Right-click on the instrument status bar or use **Automation** menubar to *STARTUP* the hardware. This will set the system to +ion mode and put the system to *C60-STANDBY* with parameters for C60 and Analyzer.
- 3. Startup required guns and wait for stable emission.
  - i. LMIG (not open for public use)
    - NEVER TURN OFF ELECTRONIC POWER WHEN EMITTER IS ON.
    - Right-click on the instrument status bar to put the system to *LMIG-STANDBY* to load parameters for LMIG and Analyzer.
      - C60 and LMIG require different Analyzer setting (blanker timing) so respective setting is required. It may be possible to use C60 setting for LMIG operation, but not the other way around.
    - Select required setting in the pull-down list then press *Load* in Hardware session  $\rightarrow$ LMIG page  $\rightarrow$  LMIG tab.
    - Verify **Beam Energy** to be 30 kV, then select *Ready* mode to trigger the automatic **Start Emitter** function. The *Emission* should be stabilized at ~0.5  $\mu$ A when finished. Setup timing in **Hardware** menubar  $\rightarrow$  **Sequencer**  $\rightarrow$  **LMIG** tab according to following table to select desired particle (default Bi<sup>+</sup>) and set buncher voltage if bunched setting is loaded.

	Bi <sup>+</sup> (24%)	Bi <sub>3</sub> <sup>++</sup> (12%)	Bi <sub>2</sub> <sup>+</sup> (18%)	Bi <sub>5</sub> <sup>++</sup> (7%)	Bi <sub>3</sub> <sup>+</sup> (27%)
Buncher Start Time (µs)	4.8100	4.8080	4.7350	4.7130	4.6930
Buncher (V)	800	725	690	570	565
Quad Blanker Start Time (µs)	3.3250	2.9400	2.6350	2.3780	2.1350
Quad Blanker Duration ( $\mu$ s)	0.1500	0.1500	0.1500	0.1500	0.1500
Quad Blanker Amplitude (V)	30	30	30	30	30
LMIG Blanker Start Time (µs)	5.000	5.000	5.000	5.000	5.000
LMIG Blanker Duration (µs)	1.000	1.000	1.000	1.000	1.000

• Open V7 for operation and monitor the **Suppressor** frequently. The ideal **Suppressor** is between -1500~-1000 V.

- For short idle, set **Emission** to lock at 0.3  $\mu$ A.
- When finished, select Standby mode to trigger Shutdown Emitter then turn it Off.
- Do not start/shutdown the LMIG frequently. The firmware should allow LMIG to keep running during software restart.
- During operation, if the *Emission* is too high or **Suppressor** reaches 0 V, set the *Emission* to 1.5  $\mu$ A for a few minute then set back to 0.5  $\mu$ A may help to stabilize the **Suppressor**. If necessary, lower **Heater** by 0.02 A.
- If the *Emission* is too low or **Suppressor** reaches -2000 V, unlock *Emission* then set **Extractor** to a more negative value to get 1.5  $\mu$ A *Emission* for a few minute than slowly set it back. If necessary, increase **Heater** by 0.02 A.

- ➢ It may be necessary to fine-tune the Heater during long acquisition to keep the Suppressor stable at around −1500 ~ −1000 V.
- > Note that change Heater may require slight tuning of Beam Steering.
- During short operation, if the *Emission* disappears, *unlock* the Emission and set Suppressor to -2000 V. If the *Emission* does not recover after 10 min, slowly increase the Extractor up to -9000 V in a step of 100 V/min. When *Emission* appears, decrease the Extractor while maintaining the *Emission* <3 μA until back to its original value then *lock* Emission.
  - If the *Emission* cannot be recovered, set to *Standby* mode to **Shutdown Emitter** then set to *Ready* mode to **Start Emitter** again if necessary.
- ii. C<sub>60</sub> (open for public use)
  - Check and verify V4 and C<sub>60</sub> manual isolation valve is open.
  - From *off*, set *Idle* mode in **Hardware** session → C60 page → C60 tab and wait for temperature to stabilize at set value (~200 °C).
  - From *Idle*, set *Standby* mode and wait for temperature to stabilize at set value (~410 °C).
    - During above heat up process, V4 must remain open and *Intro* pressure at the level of lower 10<sup>-3</sup> Pa. Do not preform sample exchange during this process.
  - Select required setting in the pull-down list then press *Load*.
  - C60 and LMIG require different Analyzer setting (blanker timing) so respective setting is required. It may be possible to use C60 setting for LMIG operation, but not the other way around.
  - Verify timing in Hardware menubar  $\rightarrow$  Sequencer  $\rightarrow$  C60 tab according following value.

Buncher Start Time (µs)	2.3100
Buncher (V)	300
Pulser1 Start Time (µs)	1.0000
Pulser1 Duration (µs)	0.1000
Pulser1 Voltage (V)	200
Pulser2 Start Time (µs)	3.6450
Pulser2 Duration (µs)	0.075
C60 Pulser2 Voltage (V)	200

- For using C<sub>60</sub> as sputter gun, select required setting in the pull-down list then press *Load*. Confirm the aperture used after load the setting.
- Make sure the Intro pressure is below  $3.3 \times 10^{-3}$  Pa then put the gun to *Ready* mode and wait for stable *Current Monitor 1* (C60 tab  $\rightarrow$  Properties  $\rightarrow$  Column tab) at a few 10s nA for operation and *Heater* at ~30%. Verify the *Emission* (*Grid Current*) to be 10 mA and *Filament* <2.2 A (C60 tab  $\rightarrow$  Diagnostics  $\rightarrow$  Column tab).
  - If the temperature cannot be stable at ~410 °C, set *Idle* mode and allow the source to cool down to 350 °C then startup again.
- For sample exchange during operation, set *Standby* mode and wait Intro pressure to be below  $3.3 \times 10^{-3}$  Pa before put it back to *Ready* mode.
- For short idle, set *Standby* mode.
- When finished, set *Idle* mode. Do not select *off* directly.
- iii. GCIB (<u>not open for public use</u>)
  - Check and verify that pump station is running (on the panel of pump station or in **System** session, GCIB page: TMP1: 1000 Hz, TMP2 and TMP3: 1500 Hz, GC1-3 open) and Ar line is filled at 0.8-0.9 MPa. If Ar main gauge is 0, purge the line before open the tank.

- Select required setting in the pull-down list then press *Load* in Hardware session → GCIB page.
- Select *Ready* mode. Pressure Control would turn on and **Gas Leaking Valve (GC0)** would open automatically.
- Set *Target Pressure* to 650 kPa for optimized  $Ar_{2500}$  and the vacuum reading on GCIB controller should be ~2.2 Pa.
- Check and verify the manual isolation valve is open.
- When finished, close the manual isolation valve, close the Ar tank, set *Target Pressure* to 101.33 kPa then turn off *Pressure Control* and *Gas Leak (GC0)*, load *ZERO* setting then select *off* mode.
- iv. Gas Gun (open for public use)
  - Check and verify V4 is open.
  - Select required setting in the pull-down list then press *Load* in Hardware session → Gas Gun page.
  - Set *Standby* mode then switch to *Ready* mode. Verify V10 is open automatically.
  - Gun Pressure should be 10~15 mPa in *Ready* mode (~25 mPa in *Standby* mode). When setting for higher beam voltage, lower Gun Pressure is expected.
  - When finished, load ZERO setting and select off mode. Verify V10 is closed.
- v. E-Neut (open for public use)
  - Right-click on instrument status bar to load *E-NEUT* or *E+I-NEUT* or select *E-NEUT* setting in the pull-down list then press *Load* in Hardware session  $\rightarrow$  E-Neut page.
  - Select *Standby* mode then switch to *Ready* mode.
  - Open the **Diagnostics** page to read **Emission** by right-click on **Properties** button. Note the reading is not continuous by default.
  - Start current reading and tune the Filament current for ~100  $\mu$ A Emission shown in the Diagnostic window. The Filament current needs to be tuned regularly during operation.
  - When finished, load *ZERO* setting and select *off* mode. Verify **Filament** current is set to 0.
- 4. When required guns are stabilized, open V8 to allow ions to enter spectrometer.
- 5. Open the **Tools** menubar → **Joystick** window for moving sample position to be exactly 2 mm working distance from analyzer lens. The current stage position can be found at bottom-left of instrument control screen.
  - Always use the side-view camera on the touchpad (AMCap) during stage movement and make sure it does not touch the extractor.
  - During the movement, look at the shadow of analyzer in *Chamber* image and make sure the sample does not hit the extractor.
  - > THE BOTTOM-RIGHT SHADOW CAN NEVER TOUCH THE ANALYZER SHOWN AT TOP-LEFT OF THE *CHAMBER* IMAGE. THE SAMPLE CAN NEVER TOUCH THE EXTRACTOR IN THE SIDE-VIEW CAMERA.
  - For correctly prepared back-load sample platen without protrusion and is flat with respect to the cover piece, while the side-view camera indicate the sample is correctly at Z = 0 mm position after sample transfer, adjust sample Z height to 3 mm at Sample session  $\rightarrow$  Stage page.
  - For other platen (not open for public use), Z height needs to be carefully raised to make the *Chamber* image to coincide with a recognizable feature in camera *Photo* for rough height setting then carefully tune the 2 mm working distance using *Total Ion*, *SED* or *Detector* image.
- 6. Select desired *Positive* (+) or *Negative* (-) ion polarity by right-click on the status bar.

- 7. Open Tools menubar  $\rightarrow$  Image Tool window and check Sample Bias to be ~3000 V.
- 8. Verify the correct setting (C60, LMIG, or LMIG-EPon) that corresponds to the desired primary beam is loaded in **Hardware** session  $\rightarrow$  **Analyzer** page.
- 9. TOF session → choose Spectrum / Profile / Map page → select Gun Type (LMIG / C60) → Start to start collecting spectrum. Although there is no real function, select Primary Particle to make record.
  - i. Verify the **Raster Pattern** to be *Scatter* in all tabs in **TOF** session  $\rightarrow$  **Properties**. The **Raster Pattern** in the **Properties** tab of **Image Tool** should be *Flyback* and **Sputter Tool** should be *4-Fold*.
  - ii. Turn *off* **Detector Scanning Control** in the **Image Tool** window. Set **Raster Size** to 0 can provide more focused spot for easier tuning.
  - iii. Tune the **Sample Bias** to focus the spot (events of ions hitting detector) in *Detector* image and maximize the count rate.

If the image does not show correctly, enter 1.0 to the magnification factor.

iv. Use **Joystick** to fine-tune the stage Z height and make the spot inside the white box of *Detector* image.

\*For correct Z-height and sample bias, maximum **Raster Size** with **DEM Control** off will have centered circular FOV in *Total Ion Image* and FOV of *SED Image* will just touch the right edge.

- v. Turn "Detector Scanning Control" back on.
- 10. For imaging, set required magnification using **Raster Size**. Make sure the **DEM Raster Size** matches, **DEM Control** is on and **DEM Deflector Sensitivity** is ~0.0067 in **Hardware** session → **Analyzer** page → **Properties** → **Detector** tab.
- 11. For insulating sample, **Electron Neutralization** (right-click on the instrument status and load *E-Neut* then set *E-Neut* to *Ready* state) and **+ion Neutralization** (right-click on the instrument status and load *E+I-Neut* then set E-Neut and Gas Gun to *Ready* state) can be enabled with *Charge Comp* function.
  - It is better to enable Use Settings for force loading E-NEUT and I-NEUT setting for Charge Comp function. Otherwise, current parameters set in Hardware session will be used and may be incorrect.
  - > Always double check for actual parameter of guns during acquisition.
  - +ion Neutralization is not compatible with Extractor Pulser and –SIMS because the field will accelerate ion to sputter extractor or sample.
  - In –SIMS, it is possible to set Electron Neutralization to DC Beam for better neutralization. However, DC Beam cannot be used in +SIMS because electron will be accelerated by Sample Bias to bombard the sample.
  - > It may be necessary to increase **Sample Bias** on insulators.
- 12. For better mass resolution, **Contrast Diaphragm** (CD) can be enabling it using the *CD* button on *Detector* image or **Hardware** session  $\rightarrow$  **Analyzer** page  $\rightarrow$  **Analyzer** tab.
  - CD-in may lower the counts by 20-40%. For molecular ions or rough surface, the cut is expected to be more significant because of the larger angular distribution of secondary ions.
  - Note that the CD limits angular acceptance angle of secondary ions hence it does not work well with large raster size.
- 13. To filter out metastable molecular ions, Energy Slit can be set from the Hardware session  $\rightarrow$  Analyzer page  $\rightarrow$  Energy Slit tab.
  - ➤ 50 and 20 eV setting may lower the counts by ~10% and ~30% comparing to 240 eV setting, respectively. For atomic ions, the cut is expected to be more significant because of the larger energy distribution.

Note that the use of smaller Energy Slit limits the energy distribution of secondary ions hence it makes the intensity more sensitive to Sample Bias.

- 14. To fine adjust the signal count rate, it is possible to change acquisition duty cycle through LMIG Blanker Width (Hardware session → LMIG page → Properties → Column tab) or Pulser1 Duration (Hardware menubar → Sequencer → C60 Settings). Larger setting leads to longer pulse width (more ions in each pulse and higher counts) but decreases mass resolution.
- 15. Use **Calibrate** function and choose at least two known peaks to calibrate m/z scale then start a new acquisition for the calibration to be effective.
  - > The m/z calibration depends on the primary particle. Respective file in *Mass* Calibration folder is a good start when switching to a different particle.
- 16. If raw file is desired for further offline analysis, check **Save Raw File** in **TOF** session  $\rightarrow$  **Spectrum** page  $\rightarrow$  **Properties**  $\rightarrow$  **Spectrum** tab after the m/z scale is calibrated then reacquire the data.
- 17. Save data (spectrum, profile, image, etc.) in your own folder in Z drive.

#### C. Sample remove and system shut down

- 1. Drag icon of stage from *Main Chamber* to *Intro* in **System** session.
- 2. Right-click to *Backfill Intro* and take out the sample.
- 3. Put the sample platen back and right-click to *Pump Intro*. Move the empty sample platen into *Chamber* when vacuum is ready (V4 opened automatically).
- 4. Shutdown guns (LMIG *Standby* then *Off*; C<sub>60</sub> at <u>Idle</u>; GCIB, Gas Gun, and E-Gun Neutralizer *Off*).
- 5. **Precursor Selector (PS)** retracted, **Contrast Diaphragm (CD)** out, **Energy Slit** at 240 eV, close isolation valves of spectrometer (V8) and gun valves (V7, V10 and GC0). The vacuum page should only have V3, V4, V5, and two Turbo in green.
- 6. Right-click on the instrument status bar or use Automation menubar to *SHUTDOWN* the hardware. Verify the instrument is at *ZERO* state and guns at *Off* state (C60 may be at *Idle*).
- 7. Turn off the power extension socket to turn off monitors.

#### **D.** Sputter and depth profile

- 1. Verify the **Raster Pattern** to be *Scatter* in **Phased** tabs in **TOF** session  $\rightarrow$  **Properties**. The **Raster Pattern** in the **Properties** tab of **Sputter Tool** should be 4-Fold.
- 2. Align the beam position. For single beam (LMIG or  $C_{60}$ ) operation without the need of charge neutralization, **Sample Bias** can be set to *HV* as the beam position will not change under the same **Sample Bias** (HV).
  - i. Tools menubar  $\rightarrow$  Sputter Tool  $\rightarrow$  set up the sputter gun, sputter time and neutralizer. Normally, *Sample Ground* is set for sputtering.
  - ii. Setup the *Raster Size* of corresponding sputter gun in Image Tool.
  - iii. Click **Timed Sputter** to create a small sputter crater on a sacrificial area.
    - Set **Sample Bias** = 0 and use **Image Tool** can also prepare the crater.
    - Sample with defined structure (e.g. Faraday cup) can also be used to ensure the concentric overlap of ion beams with *SED* Image.
  - iv. With larger **Raster Size** of desired primary beam, collect a *Total Ion* or *SED* Image to image the crater. Steer the <u>sputter</u> beam to make the crater at the center of the image produced by primary beam to make the beam cocentric.
    - Steer the primary beam by right-click on the center of sputter crater observed in Total Ion Image and select Move Analytical Beam to Curser can also align

(concentric) the area. However, less spatial and mass resolution is expected due to the mismatch of **DEM Offset**.

- When Faraday cup is at the center of primary beam SED Image, maximized sputter beam current into Faraday cup also align (concentric) the beam.
- 3. Make sure that the analysis area is smaller (25-50%) and within the sputter area.
- 4. In **TOF** session → **Profile** page → select **Gun Type Gun** (*LMIG* / *C60*) and setup the parameter for *Sputter* and *Charge Comp* Phase. The **Sample Bias** should be selected according to how the sputter beam is aligned with respect to analysis beam and is usually *Ground* or *Ground+Charge Comp*.
  - ➢ In most case, Use Settings is not enabled in Sputter Phase and the guns will use the parameters currently set in Hardware session.
  - If Use Settings is enabled (e.g. switching Gas Gun between Sputter and I-NEUT), current parameters are automatically saved as DEFAULT setting and load in other phases. Unless set to load your own settings, reference parameters will be used without fine-tuning. Nevertheless, since E-NEUT and I-NEUT does not require fine-tuning, load them directly is fine.
  - Always double check the actual parameters of guns during different phases of sputter cycle to make sure desired parameters are used.
    - Right-click on TOF session → Spectrum / Profile / Map page → Application Log (Acquisition) can also reveal what system is doing in each step.
  - > If different current/aperture is required in single-beam operation, setup *Use Settings* and select personalized setting accordingly in the pull-down list.
  - If different aperture is used for higher current in sputter phase, set the Settle Time of Sputter Phase to 30 s in Properties to allow time for switching aperture.
- 5. If necessary, additional *Charge Comp (CC) Phase* can be used to remove residual charge during sputtering.

#### **E. Imaging**

- 1. For small-area and high-resolution image, small aperture and unbunched mode is desired.
- 2. Verify the **Raster Pattern** to be *Scatter* in all tabs in **TOF** session  $\rightarrow$  **Properties**.
- 3. Set desired **Raster Size** for required magnification in **Image Tool**.
  - ➤ Check if DEM Raster Size (Hardware session → Analyzer page → Properties → Detector tab) matches and make sure DEM State is on.
  - → Mind the minimum beam size of LMIG and C<sub>60</sub> is ~100 nm and ~2 µm, respectively. The Image Size (number of pixels, typically 256 or 512, set in TOF session → Spectrum / Profile / Map page → Properties), Raster Size, and beam size should match so that the analysis points are continuous and slightly overlapped.
- 4. For LMIG and C<sub>60</sub>, tune Lens 1 and Lens 2, respectively, to fine focus the *Total Ion Image*. If necessary, set Acquisition Mode to *Refresh*.
  - > If sample height is correct, images would be focused with reference lens setting.
  - For bunched LMIG, SED Image and Total Ion Image have different focus hence Lens 2 is ~200 V lower in Total Ion Image than that in SED Image.
  - If square (check board) artifact is observed in the image, increase DMCP Detector by 50 V or move the detector spot off-center using Detector Offset then turn off Detector Scan.
    - DO NOT TURN OFF DETECTOR SCAN FOR REAL ACQUISITION WHEN THE DETECTOR SPOT IS AT CENTER.
    - It is possible to move the alignment box in the **Properties** page of *Detector* image for the using the off-center spot for Z tuning. **REMEMBER TO MOVE THE ALIGNMENT BOX TO ORIGINAL POSITION WHEN FINISHED.**

- 5. To image large area (not compatible with depth profile), use **TOF** session → **Map** page to setup *Mosaic*. The mapping area can be defined with *Photo* image or numerically set **Number of Tiles** and **Mosaic Center** in **Mosaic Area** tab.
  - The Tile Size is different from the Raster Size defined in the Image Tool and it should be set before defining the mosaic area.
- 6. For small area (high magnification) imaging of insulating sample, pulsed *E+I-NEUT* may not be sufficient to completely compensate the surface charge due to the high density of primary beam current. As a result, count rate may decrease and image could become blur during long acquisition that improves the signal-to-noise ratio. It is possible to use the **Profile** page and enable *Charge Comp Phase* but disable the *Sputter Phase*. Effectively, additional *Charge Comp Phase* can be applied between every few *Acquisition Frames* to further neutralize the charge.

## C<sub>60</sub> TUNNING (<u>for advanced user only</u>)

• The whole timing sequence is source dependent so that correct analyzer setting is required.

### A. Source startup

- 1. Check and verify V4 and  $C_{60}$  manual isolation value is open.
- From *off*, set *Idle* mode in Hardware session → C60 page → C60 tab and wait for temperature to stabilize at set value (~200 °C). During this heat up process, V4 must remain open and Intro pressure at the level of 10<sup>-3</sup> Pa. Do not preform sample exchange during this process.
- 3. From *Idle*, select *Standby* mode and wait for temperature to stabilize at set value (~410 °C).

During this heat up process, V4 must remain open and Intro pressure at the level of  $10^{-3}$  Pa. Do not preform sample exchange during this process.

- 4. Press *Load* after select required setting in the pull-down list. Confirm the aperture used after load the setting.
- 5. Make sure the Intro pressure is below  $3.3 \times 10^{-3}$  Pa then put the gun to *Ready* mode and wait for stable *Current Monitor 1* (C60 tab  $\rightarrow$  Properties  $\rightarrow$  Column tab) at a few 10s nA for operation and *Heater* at ~30%. Verify the *Emission (Grid Current)* to be 10 mA and *Filament* <2.2 A (C60 tab  $\rightarrow$  Diagnostics  $\rightarrow$  Column tab)
  - ➢ If the temperature cannot be stable at ~410 °C, set *Idle* mode and allow the source to cool down to 350 °C then startup again.
  - ➤ Normally, <2.2 A *Filament* current could yield 10 mA *Emission* hence **Filament Current Limit** at 2.5 A should be enough for most operation. Over time,  $C_{60}$  could deposit on the filament during pervious cooling and require extra time to regain emission. Increasing **Grid** voltage to 80-85 V can help to bring the *Emission* higher while the yield of  $C_{60}^+$  (beam current) will be slightly lower. It is also possible to lower the **Repellor** (more positive value) to increase the *Emission* but the maximum beam current may decrease with lower **Repellor**.
- 6. For sample exchange during operation, set *Standby* mode and wait Intro pressure to be below  $3.3 \times 10^{-3}$  Pa before put it back to *Ready* mode.
- 7. For short idle, set *Standby* mode.
- 8. When finished, set *Idle* mode. Do not select *off* directly.
- Reference beam current. If current is lower than required, tune **Repellor** slightly could increase beam current but do not allow **Filament Current** to increase >0.1 A while tuning **Repellor**.

Aperture Number	1	2	3	4	5
Micrometer Position (mm)	1.900	5.900	9.900	13.900	17.900
Aperture Size (µm)	1000	300	100	30	10
Beam Current* at 410 °C (nA)	1.0	0.45	$0.08^{\dagger}$	0.01	0.001

\*current reads using **Current Monitor 3** or Keithley meter (note the manual switch box next to the Keithley meter) with **Pulser 1** = 0 V and is the difference between **Bend** on and off.

 $^{\dagger}0.16$  as of 2017/9/4 as the aperture became larger.

#### **B.** DC mode for imaging

- 1. Load the desired setting to set the default parameter. **Extractor** should be -1500~-500 V, **Repellor** should be -20~-30 and **Bend** should be -120~-150 V.
- 2. Adjust sample Z height to be 2 mm from extractor.
- 3. Use sample Faraday cup ( $Z \sim 2.96$  mm) or suspended grid for current measurement.

- ➤ When measuring Faraday Cup current, the stage always automatically moves to the position saved in Sample session → Stage page → Stage tab → FARADAY CUP setting. If necessary, update this setting file after current Faraday cup position is determined. It is also helpful to save stage position before measure the Faraday cup current for faster move back after measurement.
- Using Current Monitor 3 or Keithley meter (note the manual switch box next to the Keithley meter) with Pulser 1 = 0 V and the difference between Bend on and off could yield more stable reading. Due to a bug in the software, if Current Monitor 3 did not work correctly and stuck at a negative value, set the Anode Voltage to 1 kV and toggle the Current Monitor 3 a few times until it shows positive background value then set Anode Voltage back to 20 kV.
- 4. Select aperture in MVA tab (1 or 2 for sputtering, 3 or 4 for analysis).
  ➢ For standard operation, aperture 1 is used for sputtering and 3 is used to analysis.
- 5. Verify the Raster Pattern to be *Fly Back* in the **Properties** of **Image Tool**.
- 6. Maximize the Sample Current or SED Image intensity (Auto Contrast off by right-click on image and select Image Properties then Image tab) with Wien Filter (~-46 For C<sub>60</sub><sup>+</sup>, ~-64.5 For C<sub>60</sub><sup>++</sup>), Bend, Steering 1Y/2X/2Y, Extractor, Repellor, and MVA position.
  > The Repellor will affect Filament Current and the Filament Current should not
- increase by >0.15 A when setting **Repellor**.
- 7. Focus with Lens 2. Tune Stig Amplitude for best image resolution.
   ➢ If grid of known spacing is used, Tilt, Rotation, Trapezoidal Correction and Raster Calibration in the Raster tab of Properties.

### C. Pulse mode for acquisition

In general, bunched 20 kV  $C_{60}^+$  is used for acquisition.

- 1. Optimize the DC operation.
- 2. Set Pulser 1 and Pulser 2 to 200 V
- 3. In Hardware menubar  $\rightarrow$  Sequencer  $\rightarrow$  C60 tab, set Pulser 1/2 Start Time to 1.0 µs, Pulser 1 Duration between 0.10~0.15 µs.
  - Longer duration will give higher counts but less mass resolution. The duration set here is a DAC value. The actual **Pulse Width** in measured by the number of channels (default is 128 ps) of the FWHM of H peak in <u>unbunched</u> mode. The actual pulse width (ns) can also be estimated by 1000×DAC 70. For standard setting of 0.1, the actual time is about 30 ns.
- 4. Set **Pulser 2 Start Time** to 1.0 μs. Set **Pulser 2 Duration** to 5 μs, **Buncher Voltage** to 0 V to effectively disable the function of **Pulser 2** and **Buncher**.
- 5. If Bunch mode is desired (normal operation), set **Buncher Voltage** to 200-400 V to maximize the additional "peak" on the broad peak in the spectrum. Tune **Buncher Start Time** to move the additional "peak" to the left-edge (onset) of the broad peak.
- 6. Increase Pulser 2 Start Time while preserving the left-edge of peak.
- 7. Decrease **Pulser 2 Duration** while preserving the right-edge of peak, focused spot in *Detector* image, and peak shape.

#### \*Source (cartridge) exchange and initial startup (for service personnel only)

- 1. Setup for simulation mode and leave *SVC* enabled.
- 2. Close the  $C_{60}$  main isolation valve.
- 3. Close the manual valve on V4 line (under panel) and open the by-pass valve with intro line.
- 4. **Backfill Intro** to vent the gun head then take the cartridge out by loose 8 M8 screw with 5 mm Allen key. Do not loose other screws.

- 5. Remove the Cu extractor to expose and exchange the cartridge and gasket.
- 6. After reassemble, **Pump Intro** and wait for a good vacuum then open manual valve on V4 (under panel), close by-pass valve with intro, then open main isolation valve.
- 7. Perform local baking. Unplug baking zones 1, 3, and 4. Unplug LMIG baking power.
- 8. After baking, Using the Idle setting and make sure the Filament Current Limit is 0 A then condition the Anode Voltage to 20 kV, Grid Voltage to 150 V, Extractor Voltage to -2500 V, Repellor Voltage to -120 V and Wien Filter to -200 V. After conditioning, set the Anode Voltage to 1 kV, Grid Voltage to 70 V, Extractor Voltage to -600 V, Repellor Voltage to -34 V. Make sure the Filament Current Limit is still 0 A.
- 9. In following heater and filament initial startup, keep the Intro at lower  $10^{-3}$  Pa.
- 10. Slowly increase temperature to heat to 250 °C and leave it for at least 4 h.
- 11. After >4 h conditioning at 250 °C, slowly increase filament to 1.5 A and leave it for 1h.
- 12. Slowly increase to heat to 350 °C and leave it for 24-48 h. Perform heater cycle to make sure it does not outgas significantly at 99% output.
- 13. Slowly increase temperature to running temperature (~410 °C).
- 14. Set the **Emission Current** to 10 mA then slowly increase filament current to obtain 10 mA emission current. Do not excess 2.5 A filament current. If cannot obtain 10 mA emission at 2.5 A, leave it for some time for thorium to activate and <2.2 A filament should be able to provide 10 mA emission.
- 15. Set the Idle mode setting back to Source Temperature 200°C, Anode Voltage 1 kV, Grid Voltage 60 V, Filament Current Limit 0.5 A, Extractor Voltage -600 V, Repellor Voltage -34 V and Wien Filter -200 V.
- 16. Load reference and update the reference setting with new alignment.

### **GAS GUN OPERATION**

- 1. Adjust sample Z height to be 2 mm from analyzer lens.
- 2. Check the pressure reading of gas bottle. If the main gauge reads 0, purge the line then fill the gas line. Close the gas bottle when done as the pressurized gas in the line can allow normal operation for a long time.
- 3. If  $O_2^+$  is required (for advance user only), manually purge/fill the gas line by
  - i. Manually close V5 (need admin mode), V10, V7 and V8.
  - ii. Close the gray Ar valve.
  - iii. Open the green pump valve.
  - iv. After 5 min, close the green pump valve then open the pink O<sub>2</sub> valve to fill with O<sub>2</sub>.
  - v. Manually open V5 or preform *Pump Intro* again to resume normal vacuum operation.
  - > NEVER LEAVE V5 CLOSED OR VACUUM WILL CRASH.
  - > OPEN MORE THAN ONE VALVE AT A TIME AND DO NOT OVER-TIGHTEN THE VALVE.
  - **> DO NOT CHANGE REGULATOR SETTING.**
  - > Always refill the gas line with Ar when finished.
  - Check the main pressure gauge of both gas bottles. If it is close to 0, <u>open then close</u> the respect bottle to refill the pipeline.
- 4. Make sure the V4 is open.
- 5. Select required setting in the pull-down list then press *Load* in Hardware session  $\rightarrow$  Gas Gun page.
- 6. Set *Standby* mode then switch to *Ready* mode. Verify V10 is open automatically.
- 7. **Gun Pressure** should be 10~15 mPa in *Ready* mode (~25 mPa in *Standby* mode). When setting for higher beam voltage, lower **Gun Pressure** is expected. This is because *Ready* mode actually produced ion beam but the beam is blanked by **Bend**. Therefore, increasing beam voltage removed more ions from the source so that **Gun Pressure** is lower.

>  $O_2^+$  will **require** longer time to stabilize. Slow increase of **Gun Pressures** indicated the feed of  $O_2^+$ .

- 8. When finished, load ZERO setting and select off mode. Verify V10 is closed.
- 9. (for advanced user only) For parameter tuning, sample Faraday cup ( $Z \sim 2.96$  mm) or suspended grid is required for current measurement and beam centering.
  - ➤ When measuring *Faraday Cup* current, the stage always automatically moves to the position saved in **Sample** session → **Stage** page → **Stage** tab → *FARADAY CUP* setting. If necessary, update this setting file after current Faraday cup position is determined. It is also helpful to save stage position before measure the Faraday cup current for faster move back after measurement.
  - i. Based on existing alignment, different Beam Energy with or without Float can be set.
  - ii. Record the value then set **Sample Bias** to 0.
  - iii. Maximize the *Sample* or *Faraday Cup* current using **Condenser** and **Bend**.
  - iv. Verify the Raster Pattern to be *Fly Back* in the Properties of Image Tool.
  - v. Use SED Image to focus the ion beam with Objective.
  - If contrast is too weak, maximize *Faraday Cup* current with Objective and Raster Offset at Raster Size = 0.
  - vi. Tune Bend with Objective Lens wobbler.
  - vii. To set desire beam current, tune **Condenser** or lower **Emission**. Check the focus after current is set.
  - viii. Verify the Raster Calibration X and Y with structure of known size.
  - ix. Tune the **Raster Offset** to make sure the center of **Gas Gun** (grounded *SED Image* or Faraday cup position) is the same as **Primary Ion Gun** (~3000 V **Sample Bias**).

## **E-GUN NEUTRALIZER OPERATION**

- 1. Adjust sample Z height to be 2 mm from analyzer lens.
- 2. Select *E-NEUT* setting in the pull-down list then press *Load* in Hardware session  $\rightarrow$  E-Neut page.
- 3. Select *Standby* mode then switch to *Ready* mode.
- 4. Open the **Diagnostics** page to read **Emission** by right-click on **Properties** button. Note the reading is not continuous by default.
- 5. Start current reading and tune the Filament Current for ~100 μA Emission shown in the Diagnostic window. The Filament current needs to be tuned regularly during operation. Note that the gun needs to be in *Ammeter* or *Neut* mode to read Emission current. In *Neut* mode, it is pulsed so that the Emission current is about half than in *Ammeter* mode.
- 6. Usually, constant **Bias** at 10 V is used. In some case, higher **Bias** may be required to overcome the electrical field and maintain the count rate.
- When necessary, tune X and Y Offset to maximum Faraday Cup (Z ~ 2.96 mm) current. In Pulse mode, half of maximum Faraday Cup current is expected with fine-tuned X and Y Offset.
  - ➤ When measuring *Faraday Cup* current, the stage always automatically moves to the position saved in **Sample** session → **Stage** page → **Stage** tab → *FARADAY CUP* setting. If necessary, update this setting file after current Faraday cup position is determined. It is also helpful to save stage position before measure the Faraday cup current for faster move back after measurement.

### **ENERGY SLIT TUNING**

- 1. In Hardware session  $\rightarrow$  Analyzer page  $\rightarrow$  Energy Slit tab, select required Energy Slit (240 eV (out), 50 eV, 20 eV).
- 2. Use **Target Position** to obtain maximum intensity using the median of two values where signal disappears.
- 3. If necessary, different size of pass-band can be set by using off-centered slit position.

## LMIG TUNNING (not open for public use)

- DO NOT TURN OFF ELECTRONICS POWER WHEN EMITTER IS ON.
- The whole timing sequence is source dependent so that correct analyzer setting is required.

### A. Emitter startup

- 1. In Hardware session  $\rightarrow$ LMIG page  $\rightarrow$  LMIG tab, verify Beam Energy to be 30 kV, then select *Ready* mode to trigger the automatic Start Emitter function. The *Emission* should be stabilized at ~0.5 µA when finished.
  - > If the startup sequence got stock at lowering **Extractor** to set value while maintaining 0.5  $\mu$ A *Emission*, increase **Heater** by 0.05 A to help the progress. As the startup sequence will put the original **Heater** value once finished, it will be necessary to set the new **Heater** value immediately after the sequence is finished to maintain the *Emission*.
- 2. Select required setting in the pull-down list then press *Load*.
- 3. Open V7 for operation and monitor the **Suppressor** frequently. The ideal **Suppressor** is between -1500~-1000 V
- 4. For short idle, set **Emission** to lock at  $0.3 \mu A$ .
- 5. When finished, select Standby mode to trigger Shutdown Emitter then turn it Off.
- Do not start/shutdown the LMIG frequently. The firmware should allow LMIG to keep running during software restart.
- During operation, if the *Emission* is too high or **Suppressor** reaches 0 V, set the *Emission* to 1.5  $\mu$ A for a few minute then set back to 0.5  $\mu$ A may help to stabilize the **Suppressor**. If necessary, lower **Heater** by 0.02 A.
- If the *Emission* is too low or **Suppressor** reaches -2000 V, unlock *Emission* then set **Extractor** to a more negative value to get 1.5 µA *Emission* for a few minute than slowly set it back. If necessary, increase **Heater** by 0.02 A.
  - > It may be necessary to fine-tune the **Heater** during long acquisition to keep the **Suppressor** stable at around  $-1500 \sim -1000$  V.
  - > Note that change **Heater** may require slight tuning of **Beam Steering**.
- During short operation, if the *Emission* disappears, attempt following steps in order. Wait for a few minutes before attempt to use next step.
  - i. Unlock the Emission and set Suppressor to -2000 V.
  - ii. Slowly increase **Extractor**. If the current cannot be obtained when **Extractor** reaches –10000 V, set the **Extractor** to its original value.
  - iii. Increase Heater by 0.05 A and repeat step ii.This step can be attempted multiple times before the Heater reaches 3.6 A.
  - iv. When the current appears, decrease the **Extractor** and **Heater** at the same time while maintain  $<3 \mu A$  *Emission* until they are back to their original value. If necessary, **Heater** can be left at slightly higher (0.05-0.1 A) setting with slight tuning of **Beam Steering**. Do not leave the **Extractor** at a different value as the whole alignment will be different.
  - v. When the *Emission* stabilized, *lock* the **Emission** to desired current.
  - vi. If the *Emission* cannot be recovered, set to *Standby* mode to **Shutdown Emitter** then set to *Ready* mode to **Start Emitter** again if necessary.
- For manual shutdown, set **Heater** to 2.0 V then slowly ramp down **Suppressor**, **Extractor**, **Heater** in sequence.
- For manually hot restart, slowly ramp up **Heater**, **Extractor**, **Suppressor** in sequence to its original value.
  - > The **Heater** and **Extractor** should be controlled by the firmware and keep running if software crash/restart.

• Reference beam current for *deceleration* mode (~+25.7 kV Lens 1) and *acceleration* mode (+3~-3 kV Lens 1, very sensitive to Extractor in a linear and same polarity manner), measure with *Faraday Cup*, *Column* or Keithley meter (note the manual switch box next to the Keithley meter) with **Ouad Blanker Amplitude** = 0 (normally 30 V)

	(							
Aperture size (µm)	800	400	200	100	50	30		
Acceleration Mode 0.5 µA	12.6	9.3	2.56	0.64	0.16	0.05		
Beam Current (nA)								
Acceleration Mode 1.0 µA	14.8	10.1	2.95	0.72	0.19	0.06		
Beam Current (nA)								

<sup>➢</sup> With operation time, the aperture will become larger hence the beam current will increase. To compensate for similar current, *Emission* or **Beam Steering X** can be changed.

#### B. DC mode for column alignment (required when extractor voltage is changed)

- 1. Adjust sample Z height to be 2 mm from analyzer lens. Sacrificial area with suitable feature (*e.g.* 1000 mesh grid) should be used.
- 2. Load the desired instrument setting to set the reference parameter.
- 3. Select desired LMIG aperture (usually 50 or 200 µm).
- 4. Verify the Raster Pattern to be *Fly Back* in the Properties of Image Tool.
- 5. Adjust SED gain (~1500 V) and Raster Size (max: 640 µm) to get the SED Image.
- 6. Adjust Lens 1 (condenser) and Lens 2 (objective) to roughly focus image. Adjust Stigmator 1 and 2 if necessary.
- 7. Maximize the beam current or *SED Image* intensity (*Auto Contrast* off by right-click on image and select **Image Properties** then **Image** tab) with **Lens 1**, **Beam Steering X** and **Y** and refocus with **Lens 2**.
  - The Lens 1 voltage is very sensitivity to Extractor in acceleration mode (Lens 1 in ±3000 V). With higher Extractor (closer to 0), higher Lens 1 will be required. For deceleration mode, Lens 1 is always around +25700 V.
- 8. Enable Lens 1 wobbler (right-click) to center the aperture position with MVA.
  - With Lens 1 wobbler, the fluctuation in image brightness indicates that the beam is way off-axis. In this case, tune Beam Steering to the median of the numbers where SED images disappear then center the aperture. The window of Beam Steering is very small in acceleration mode but larger in deceleration mode.
- 9. Use Lens 2 wobbler to adjust the Lens 2 Steering.
- 10. Use **Blanker** wobbler to tune **Lens 2** so that the objective lens is focused at blanker for motionless blanking.
- 11. Tune Lens 1 to fine focus (iterate above steps for optimal alignment).
- 12. In Raster tab, tune Stigmator 1 and 2 Steering using respective Stigmator wobbler.
- 13. Fine tune **Stigmator 1** and **2** to get best image.
  - If grid of known spacing is used, Tilt, Rotation, Trapezoidal Correction and Raster Calibration in the Raster tab of Properties.

#### C. Pulse mode for spectrum and high-resolution imaging

Bunched ion beam and **Extractor Pulser** can improve mass resolution. If **Extractor Pulser** is required, different **LMIG** and **Analyzer** setting is required.

- 1. Adjust sample Z height using *pulse* mode and select LMIG aperture (usually 50 or 200  $\mu$ m).
- 2. In Hardware menubar  $\rightarrow$  Sequencer  $\rightarrow$  LMIG Settings tab, set Quad Blanker Amplitude = 0 and Buncher = 0.

- 3. Acquire spectrum and look at H region, multiple peaks would occur due to the time difference of Bi<sup>+</sup>, Bi<sub>3</sub><sup>++</sup>, Bi<sub>2</sub><sup>+</sup> and Bi<sub>3</sub><sup>+</sup>.
- 4. Set the reference timing.
- 5. Optimize the timing to obtain single H peak. By lowering the **Quad Blanker Start Time**, the window will move to the right of spectrum; by increasing the **Quad Blanker Duration**, the width of the window will extend to the left. Note that after the timing is tuned for different particle, the **Calibrate** needs to be used to get correct m/z scale.
- 6. Dose calculation (set in **Properties** in **TOF** session  $\rightarrow$  **Spectrum** page  $\rightarrow$  **Properties**)
  - i. The LMIG Blanker Width is a DAC value. The actual Pulse Width is measured by the number of channels (default is 128 ps/ch) of the FWHM of H peak in unbunched mode. The actual Pulse Width (ns) can also be *estimated* by 9.25×DAC-3.07 (subject to change with Quad timing). For standard setting of 1.5, the actual time is about 10.8 ns.
  - ii. The total **DC Current** can be measured in *Faraday Cup* or *Column*. The actual **DC Current** is only a portion (ratio of selected particle) of the total current.
    - The current can also be read from Keithley meter at *Ready* mode with Quad Blanker Amplitude = 0 (mind the manual switch between Keithley and computer, and set the Quad Blanker Amplitude back to 30 when finished).

#### D. Bunch mode for high resolution spectrum

- 1. Set the Buncher timing in Hardware menubar  $\rightarrow$  Sequencer  $\rightarrow$  LMIG Settings tab using reference value.
- 2. Optimize these values for best mass resolution.
- 3. If image is needed, Lens 2 needs to be lowered by 100~300 V to regain focused ion image.
- 4. The buncher would preserve all ions in the pulse hence the dose is the same as its unbunched counterpart.

#### E. Enable Extractor Pulser of Analyzer to further improve mass resolution

- 1. Based on Bunch mode, find a recognizable feature.
- 2. Load reference *LMIG-EPon* setting in **Hardware** session → **Analyzer** page or manually input **DEM Steering (Detector** tab), **Extractor Pulser** (±2000 V, **Spectrometer** tab) **Immersion Lens (Spectrometer** tab) in the **Properties**.
- 3. Use **Raster Offset** of in the **Properties** of **Hardware** session → **LMIG** page to find the feature back. Slightly tune **Lens 2** if necessary.

#### \*Tip exchange and initial startup (for service personnel only)

- 17. Setup for simulation mode and leave SVC enabled.
- 18. Confirm V7 is closed and perform hipot conditioning and plasma cleaning if leakage current is observed or vacuum spiked during hipot.
- 19. Backfill Intro to connect N<sub>2</sub> by-pass line then pump/purge the bypass line.
- 20. Open the manual pumping isolation valve on LMIG and Backfill Intro.
- 21. Preform plasma cleaning if required.
- 22. While by-pass line fully opened, perform tip exchange.
- 23. **Pump Intro** and wait for a good vacuum then start LMIG source IGP.
- 24. At vacuum  $<10^{-5}$  Pa, perform hipot and check for leakage current. If necessary, perform plasma cleaning.
- 25. Perform LMIG baking. Unplug baking zones 1, 3, and 4. Unplug  $C_{60}$  baking power.
- 26. Perform hipot conditioning of LMIG source IGP.

- 27. Ramp the **Beam Energy** to 30.000 V using **Hardware** menubar  $\rightarrow$  **Outgas Conditioning** and select LMIG Outgas. This is to condition the HV generator and ensure the stability of controller.
- 28. Condition Suppressor to -2000 V and Extractor to -12000 V.
- 29. Set Beam Energy to 15.000 kV for first ignition. Move MVA to old an aperture to preserve good aperture.
- 30. Set Extractor to -1000 V then initially heat the tip to 2.3 A with Hardware session  $\rightarrow$  LMIG page  $\rightarrow$  Properties  $\rightarrow$  Source tab  $\rightarrow$  *Bi* Source button  $\rightarrow$  Reheat tab.
- 31. Keep increasing the **Heater** (0.1 A per 2 min) and record heater resistance (**Diagnostics** page) to find the discontinue point. Keep outgas the heater to 0.4 A higher than the discontinue point (but do not excess 3.8 A).
- 32. Set initial Normal Heater Current in the Bi Startup tab according to 0.2 A higher than discontinue point of heater resistance, Normal Extractor to -4000, Step 1 Heater Current Extra to 0.2-0.3 A (so that actual Heater Current is within 3.8 A), Step 2 Suppressor to -1000 V and Step 3 Heater Current Extra to 0.2 A.
- 33. Use automatic Start Emitter function for initial startup.
- 34. Break the automatic sequence after initial burst. Set the **Heater** to 0.2 A higher than discontinue point then tune the **Extractor** for stable 3  $\mu$ A then 1.5  $\mu$ A **Emission**.
- 35. Tune the **Emission** to 1.0  $\mu$ A and start **Suppressor** feedback to verify its stability for 1 h. Tune **Heater** as required.
- 36. Set **Beam Energy** to 30000 V and leave it running overnight. **Emission** should dropout around 10  $\mu$ Ah but leave the condition unchanged until next day.
- 37. Put **Suppressor** to 0 V, **Extractor** to -1000 V then apply 0.2 A extra for **Heater**. Ramp **Extractor** slowly to regain emission.
- 38. Set Heater back to pervious value (from step 18), Suppressor to -1000 V and tune Extractor for 0.5  $\mu$ A Emission Current. Fine tune Heater as required.
- 39. Set Normal Heater Current and Normal Extractor in the Bi Startup tab.
- 40. Shutdown the emitter for at least 30 min and use automatic **Startup Emitter** to make sure a stable *Emission* can be obtained at 30 kV.
- 41. Tune the reference alignment.
- 42. After shutdown for at least 8 h, check for successful startup with **Step 2 Suppressor** to 1500 V in the **Bi Startup** tab. Confirm reference alignment is good.
- When the tip gets old, higher **Extractor** and **Heater** may be required. Changing **Extractor** affects the whole alignment so that try to keep **Extractor** constant.
- Note for acceleration mode: when **Extractor** is set to be more negative, the **Lens 1** voltage will also be more negative. **Lens 1** voltage can be estimated with 5.7965×**Extractor**+30162. If the Extractor is lower than -4600 V, it can be difficult to operate in acceleration mode. For new tip, check the tested Vext to be >4700 would be better.
- When off-axis occurred (FC dropped more than half when emission is set to half), it might be recover by drain the Bi on the tip through leave the emitter running until drop-out occurred.

### GCIB OPERATION (not open for public use)

- 1. Adjust sample Z height to be 2 mm from analyzer lens.
- 2. Check and verify that pump station is running (on the panel of pump station or in **System** session, GCIB page: TMP1: 1000 Hz, TMP2 and TMP3: 1500 Hz, GC1-3 open).
- 3. Check the Ar main pressure from the regulator on the large Ar tank. If it already dropped to 0, purge the Ar line by opening the manual green by-pass valve and **Gas Leak Valve (GC0)** until the vacuum reading on GCIB control is below E–2.
- 4. Turn on the main value on the large Ar tank and confirm the line pressure is at  $8 \sim 9 \text{ kg/cm}^2$  (0.8~0.9 MPa).
- 5. Select required setting in the pull-down list then press *Load* in Hardware session  $\rightarrow$  GCIB.
- 6. Select *Ready* mode. Pressure Control would turn on and **Gas Leaking Valve (GC0)** would open automatically.
- 7. Put the gun to *Ready* mode. Gas Leak Valve (GC0) should open automatically.
- 8. Set *Target Pressure* to 650 kPa for optimized  $Ar_{2500}$  and the vacuum reading on GCIB controller should be ~2.2 Pa.
- 9. Check and verify the manual isolation valve is open.
- 10. When finished, close the manual isolation valve, close the Ar tank, set *Target Pressure* to 101.33 kPa then turn off *Pressure Control* and *Gas Leak (GC0)*, load *ZERO* setting then select *off* mode.
- 11. For parameter tuning, sample Faraday cup ( $Z \sim 2.96$  mm) or suspended grid is required for current measurement and beam centering.
  - ➤ When measuring Faraday Cup current, the stage always automatically moves to the position saved in Sample session → Stage page → Stage tab → FARADAY CUP setting. If necessary, update this setting file after current Faraday cup position is determined. It is also helpful to save stage position before measure the Faraday cup current for faster move back after measurement.

(for advanced user only)

- i. Based on existing alignment, different **Beam Energy** can be set.
- ii. Record the value then set **Sample Bias** to 0.
- iii. The cluster size is controlled by the pressure and 650 kPa yields optimized Ar<sub>2500</sub>. The cluster size can be estimated by  $4.254E-6\times P^4 0.00936\times P^3 + 7.64\times P^2 2728\times P + 358840$  where P is between 500–700 kPa.

Cluster size	1000	1500	2000	2500	3000	3500	4000
Pressure set (kPa)	515	545	600	650	680	695	705
Gauge reading (Pa)	1.41	1.54	1.79	2.00	2.15	2.20	2.25

- iv. If cluster size is changed, tune **Bend** and **Wien Deflection** for maximum *Sample* or *Faraday Cup* current first. Unless **Wien Deflection** is approaching the limit value, keep **Magnet** unchanged.
  - ➤ For smaller cluster, lower Magnet may be used, and *vice versa*.
- v. Maximize the *Sample* or *Faraday Cup* current using **Extrator**, **Focus**, **Bend**, and **Wien Deflection**.
- vi. To set desire beam current, tune Focus (condenser lens) or Emission.
- vii. Use *SED Image* to focus the ion beam with **Objective** and **Stigmation**. Verify the **Raster Pattern** to be *Fly Back* in the **Properties** of **Image Tool**.
  - If grid of known spacing is used, Tilt, Rotation, Trapezoidal Correction and Raster Calibration in the Raster tab of Properties.

Tune the **Raster Offset** to make sure the center of **GCIB** (grounded *SED Image* or Faraday cup position) is the same as **Primary Ion Gun** (~3000 V **Sample Bias**).

### POST ACCELERATION (not open for public use)

- 1. For trace analysis, lower (200 V) post acceleration may be used to suppress background.
- 2. To enhance count rate, especially for low intensity molecular ions at high m/z, post acceleration can be increased. As it enhances the intensity of all ions, low m/z peaks need to be blanked to avoid saturation/damage to the detector.
- 3. Acquire spectrum at normal condition (5 kV post acceleration)
- 4. Blank high-intensity peaks at low m/z region Correct timing sequence and blanking region of 1 amu may be required for the blanker to function properly.
- 5. In Hardware session  $\rightarrow$  Analyzer page  $\rightarrow$  Analyzer tab  $\rightarrow$  Properties  $\rightarrow$  Detector tab, increase Post Accel Energy.
- 6. If the count rate is too high (>5E4), lower the post acceleration and blank more unused m/z.

### ADVANCED INSTRUMENT SETTING (not open for public use)

- Setting files (.phi text files) are located in C:\SmartSoft-TOF\Setting\[PHI LMIG, C60-20kV, GCIB, Gas Gun, Analyzer]\Settings. Backup your own setting files as they can be removed to keep the list clean. The File... button allows loading setting files saved in any directory but does not appear in the list. To add the setting back to the list, put it back to respective directory and press Load to refresh the list.
- For repeating work, it is possible to setup personalized instrument settings that start/switch *all* required functions with desired parameters. Right-click on the status bar of instrument state and select *Instrument Load* will bring on a window that specify the pre-loading of settings of each component. These instrument files are saved in "C:\SmartSoft-TOF\Setting\Instrument\Settings" and should be backup by yourself.
- For repeating work, it is possible to save analysis parameters set in the **TOF** session. These files are saved in "C:\SmartSoft-TOF\Setting\Acquisition\Settings" and should be backup by yourself.

## SETTINGS FOR BLANKER TIMING

### (not open for public use)

#### Do not change other timing settings as they can mess-up the whole timing.

The reference starting point of pulse sequence is defined with the buncher time of the primary ion guns. When different primary particle is used, the actual time it hits the sample differs. As a result, different timing is required.

If significant portion of spectrum is missing, different timing is required.

- ➤ The effect is more apparent when switching between C<sub>60</sub> and LMIG as the flight time is very different.
- > Wrong setting in **High Mass** Blanker can distort the spectrum and prevent correct m/z calibration.
- > Wrong timing will prevent **Post ESA** blanker from blanking desired m/z range.
- It is helpful to visualize by setting larger windows (e.g. 2 amu) and plot the spectrum in log scale.
- > In TOF session  $\rightarrow$  Analyzer page  $\rightarrow$  Analyzer tab  $\rightarrow$  Properties  $\rightarrow$  Spectrometer tab

	LMIG	C60
High Mass Start Slope	0.287	0.273
High Mass Stop Slope	0.320	0.320
PostESA Slope	2.040	2.040

It may be possible to use C60 blanker setting for LMIG work, but peaks lower than set **Start Mass** may appear. On the other hand, if mistakenly use LMIG setting for C60, low mass region will be cut.

- 1. High Mass blanker
  - i. Set **Start Mass** to 1.5 amu for acquisition. Tune **High Mass Start Slope** in 0.001 step to remove H. Set **Start Mass** to 0.5 amu and make sure H is present.
  - ii. Set End Mass to desired amu for acquisition. Tune High Mass Stop Slope in 0.001 step.
    - Do not set the m/z range close to strong peaks. It is not as effective to blank strong peaks close-by as Post ESA blanker.
    - Increase or decrease the setting if the actual cut-off is before or after the desired value, respectively.
    - Because of the software also "cut" m/z outside the set range, tune the blanker carefully to the edge of the m/z to ensure correct hardware blank.
- 2. Post ESA blanker
  - i. Start with reference setting. The reference values can be different due to the change in **Extractor Pulser**, Mass Calibration and Z-height
  - ii. Blank  $\pm 0.5$  amu of desired peak and tune **Post-ESA Blanker** in 0.001 step to blank selected peak.

### **DEM TUNING**

#### (service personnel only, should never change)

- 1. Use the standard Al/Cu grid at 3 mm height or sample Faraday cup at 2.96 mm.
- 2. Turn off Detector Scanning and DEM in TOF session  $\rightarrow$  Analyzer page  $\rightarrow$  Analyzer tab  $\rightarrow$  Properties  $\rightarrow$  Detector tab.

The **Detector Steering** and **DEM Steering** are normally active.

- 3. Set Analyzer setting to LMIG or LMIG-EPon for reference values without and with the use of Extractor Pulser.
  - Extractor Pulser can help m/z resolution. Fixed -/+ 2000 V is used for positive/negative ions.
  - > The use of Extractor Pulser affects Immersion Lens, Beam Position and DEM Steering.
- 4. Turn off High Mass Blanker, MS2 Blanker and Post ESA Blanker (TOF session → Analyzer page → Analyzer (MS2) tab → Properties → Spectrometer tab) to allow *Direct Ion Image*.
- 5. In *SED Image* mode with maximum **Raster Size**, slowly increase **DMCP Voltage** (**TOF** session → **Analyzer** page → **Analyzer** tab → **Properties** → **Detector** tab) to obtain the direct ion image. The **DMCP Voltage** should never go above 1200 V.
- 6. Tune Immersion Lens (Spectrometer tab, ~6300 and ~7500 V with and without Extractor Pulser) to focus the *Direct Ion Image*.
- 7. Tune the **DEM Steering** to obtain homogeneous diamond-like image and center the circular FOV (or brightest region) in total ion image (**CD**-in can help the contrast). Iterate 6 and 7 to optimize.
- 8. Set High Mass Blanker = 160 V, Post ESA Blanker = 200 V and MS2 Blanker = 1500 V.

### **REFERENCE SPOT POSITION FOR Z HEIGHT** (service personnel only, should never change)

- 1. Use the standard Al/Cu grid at 3 mm height or sample Faraday cup at 2.96 mm.
- 2. Turn off Detector Scanning and DEM in TOF session  $\rightarrow$  Analyzer page  $\rightarrow$  Analyzer tab  $\rightarrow$  Properties  $\rightarrow$  Detector tab.
  - > The **Detector Steering** and **DEM Steering** are always active.

- 3. Set the Raster Size to maximum 640 µm, Sample Bias to 3000 V.
- 4. Check default/reference **DEM Steering** from the parameter table.
- 5. Check default/reference LMIG Beam Position from the parameter table
- 6. The LMIG *SED Image* and *Total Ion Image* will show the FOV of immersion lens. The FOV of *SED Image* should just touch the right edge of the image and the *Total Ion Image* should have circular FOV in the center.
- 7. Turn on **DEM** and tune **DEM** Sensitivity for uniform luminance in *SED Image* and *Total Ion Image*. In *Direct Ion Image*, smallest spot should be obtained.
- 8. Tune the **Sample Bias** for highest count rate.
- 9. Turn on **Detector Scanning Control**.
  - i. Set **Detector Scan Size** to 0.
  - ii. Move the spot in *Direct Ion Image* to the box region with **Detector Steering**.
  - iii. Set Detector Scan Size back to 4.
  - The Detector Scan Factor (Detector Calibration File) is tight to Detector Scan Size so that always set it to 4. If Detector Steering is significantly changed, new Detector Calibration from Hardware menubar is required.

### **DETECTOR CALIBRATION**

#### (service personnel only, should never change)

- 1. Use the <u>unsputtered</u> Si and well calibrated/tuned LMIG Bunched mode.
- 2. Set  ${}^{28}\text{Si}^+$  or  ${}^{16}\text{O}^-$  in *Peak* window for respective mode.
- 3. Set 0 Raster Size, DEM off, Detector Scan off, CD-out, PS-out, 0-1000 amu acquisition.
- In Hardware menubar → Detector Calibration, set desired filename and start <u>50</u> frame (~<u>5</u> min) acquisition without charge compensation. Detector Scan will start automatically according to set Detector Scan Size (normally 4).

The set filename has a tendency to revert to pervious setting and overwrite pervious setting. Make backup before new acquisition so that in case overwritten, can copy back.

- 5. Once finished, set to newly created calibration file in **Hardware** session  $\rightarrow$  **Analyzer** page  $\rightarrow$  **Properties** tab and save then load the setting for new file to be active.
- 6. Use 600 μm **Raster Size**, **DEM** on, **Detector Scan** on to verify the result. Uniform *Total Ion Image* should be observed and the peak intensity should increase uniformly around the center and its position do not move during **Detector Scan**.

With bad **Detector Calibration**, peak intensity of one side of the peak will increase first then jump to increase at another side during **Detector Scan**.

### TANDEM MS/MS OPERATION (not open for public use)

- VERIFY THE ANALYZER TMP IS AT FULL SPEED (1000 HZ) AND MANUAL VALVE IS OPENED BEFORE OPEN V15 TO INTRODUCE GAS INTO CID.
- The **Precursor Selector** (**PS**, plate that diverts desired secondary ion to MS2) will lower the angular acceptance angle to ~40° so that it should be retracted when not in use.
- Currently, peak blanking (function of Post-ESA blanker) is not functioning.
- DO NOT TRY TO LOAD SETTING DURING MS2 ACQUISITION OR TUNE SETTINGS FAR (>0.2) FROM REFERENCE VALUE.
- Retract **Precursor Selector** when finish.
- 1. Align and obtain standard SIMS and calibrate the mass correctly. Because the timing for **Precursor Selector** to pick correct ion depends on the calibration of standard SIMS, it has to be aligned and calibrated correctly.
- 2. Set an unlimited acquisition so the spots on *Detector* image is always visible during the insertion of **Precursor Selector**.
- 3. Slowly insert the **Precursor Selector** while observing the spots on *Detector* image. The spot will disappear first then come back and reach maximum. From fully retracted, it takes 10 turns and should align to the marker for the **Precursor Selector** to reach correct position. Do not over-insert the **Precursor Selector** so the spot disappear again as the assembly can be damaged.
- 4. In **Hardware** session  $\rightarrow$  **Analyzer** page  $\rightarrow$  **MS2** tab, load *MS2\_Analyzer* setting.
- 5. In TOF session → Spectrum/Profile/Image page, Active MS/MS function and set desired Precursor Mass (with a mass precision of 0.01 to 0.0001 amu as determined in correctly calibrated standard SIMS spectrum). If necessary, set the Duty Cycle (ratio of frames using MS2) to preserve 1-50% intensity in standard SIMS spectrum.
- 6. Switch to *MSMS Detector* image and start acquisition to observe the spot in the *MSMS Detector* image.
- To maximize the counts, slightly (use up/down arrow key for course then control-up/down for fine step, DO NOT use shift-up/down or key-in number directly) adjust *Precursor Selector Slope* (around <u>2.06</u>, with course/fine step of 0.001/0.0001) *Width Factor* (around 0.7, with course/fine step of 0.01/0.001)

in Hardware session  $\rightarrow$  Analyzer page  $\rightarrow$  MS2 tab.

- Enlarge the region near *Precursor Mass* and set to *Log* scale in standard SIMS, a window of ~6-7 m/z should have no counts as they are deflected toward MS2. Lowering the *Precursor Selector Slope* moves the deflected m/z window toward lower m/z and counts at higher m/z will start to appear. For maximum counts, the *Precursor Mass* should be at slightly lower m/z side of the deflected window.
- Decreasing the Width Factor will move the spot in MSMS Detector image to the right. For maximum counts, the spot should be close to the center of the MSMS Detector image.
- Owing to the low intensity of MS2, it is possible to tune *Precursor Selector Slope* and *Width Factor* using peaks of higher intensities then set to desired peak for acquisition using otherwise the same analysis parameters and sample position. However, if any beam parameter is changed (*e.g.* raster size, bunch/unbunch, timing, pulse width, *etc.*), need to calibrate standard SIMS again and then tune *Precursor Selector Slope* and *Width Factor* again.
- It is possible to enhance the signal intensity (dose of primary beam) by setting larger aperture (sacrifice spatial resolution) and/or longer pulse (sacrifice mass resolution).

- LMIG: Hardware session → LMIG page → Properties → Column tab → LMIG Blanker Width. Operate in *Bunched* mode (can be further improved with Extractor Pulser) could preserve some mass resolution but sacrifice spatial resolution.
- C60: Hardware menubar  $\rightarrow$  Sequencer  $\rightarrow$  C60 tab  $\rightarrow$  Pulser 1 Duration. When set Pulser 1 Duration to be >0.2, should also increase Pulser 2 Duration so the overlapped time window can really increase to produce longer pulse. Simply set Pulser 1 Duration to 0.5 has no real benefit from set to 0.2.
- When count rate is too high in standard SIMS, set Start Mass to 50 amu or higher to blank intense peaks at low mass or lower DMCP Detector voltage (may cause nonuniform intensity in standard SIMS image when Detector Scanning is used) to avoid saturation of TDC and preserve DMCP.
- There is no real tuning for the spot position on the MSMS Detector image but the size of DMCP is much larger (40 mm cf. 27 mm for standard SIMS) so that it can still acquire signal even the spot is out-of-center. Since the spot is rather close to the edge of the assembly of ~26 mm, reflection is observed and two spots with mirror symmetry are normally observed.
- V15 (Ar gas in collision cell) can be either opened or closed. With Ar feed, collision with gas can fragment ions more effectively and produce more useful pattern. Without Ar feed, only surface-induced fragmentation and signal is very limited.
- With the fixed entrance aperture of collision cell and only ~mm separation of secondary ions at **Precursor Selector**, the ion directed to into MS2 has a fixed range of ~0.5, 1 and 2 amu for 250, 500 and 1000 m/z, respectively while ~6-7 amu range was lost from standard SIMS around the selected mass.
- 8. Mass calibration of MS2 requires at least 4 known peaks to fit for actual *Precursor KE*, *Buncher* voltage and time, and *Acceleration* voltage for a system of a known distance. However, only the parent peak is generally known.
  - i. With correctly calibrated standard SIMS spectrum, the peak position of the parent peak in MS2 served as *1-Peak* calibration with respect to the value set in *Precursor Mass*. With this simple calibration, the mass precision should be better than unit mass.
  - ii. Set at least 3 more known peaks to *Optimize* the calibration for best mass precision (<20 ppm).
  - iii. If there is not enough peaks with known chemical formula or molecular weight, it is possible to save the spectrum as *MSMS unit-mass spectrum (\*.msp)* and use **NIST Mass Search** software to identify possible structure and molecular weights (formula) of fragments.
- Due to a software issue, MS2-related function of (3D-)Imaging and Profiling may not be available in the Peaks window of ToF-DR especially after software restart. In this case, select a MS2 spectrum and use "Add a Profile/Image from cursor range" button in Spectra window or "Add new peak" in Peaks window to enable MS2 settings.
- Due to a software issue, live *Chamber* (sample) image at upper-right panel has a lag when showing *MSMS Detector* image at lower-right panel. Therefore, when moving stage, always switch back to standard SIMS *Detector* image for lower-right panel.

### **CRYOGENIC OPERATION** (not open for general use)

- The temperature range is ±150 °C. Heater *Power Limit* should be <60%.
- The maximum sample size is **15 mm in diameter**, **2 mm in thickness**.
- Do not use adhesive to secure the specimen.
- Refer to "sample platen" file for how to use the cold stage correctly.
- DO NOT TIGHTEN SCREWS, JUST SNUG THEM.
- BEFORE BACKFILL INTRO AND WHEN INTRO IS EXPOSING TO AIR, SET POWER LIMIT TO 0% in Sample session → Stage page → *Hot Cold* tab.
- MAKE SURE THE *TEMPERATURE CONTROL* IS INACTIVE AND *HEATER POWER LIMIT* 0% WHEN PLACING/REMOVING HEATER.
- ▶ Right-click *Temp Control* button can show the temperature log.
- WHEN SAMPLE IS AT INTRO, V4 NEEDS TO BE CLOSED.
- Frequently verify that LN<sub>2</sub>/N<sub>2</sub> flow is not blocked and is removed by the plastic pipe on the wall. The elastic tube should not be directly plugged into the plastic pipe in order to avoid vibration they should be separated by around 1 cm and close to each other. If there is no suction from the plastic pipe, check for the green light on the breaker box (and switch below it) between N<sub>2</sub> generator and air compressor.
- Check the  $O_2$  monitor near the ceiling to be ~21% at all times. If it drops, leave the door open and use fan to draw outside air into the lab.
- The green pressurization loop on the  $LN_2$  tank should be kept closed.
- The default temperature reading time is every 60 s and acquisition/sputter will be paused during temperature reading. If necessary, change the setting in **Sample** session → **Stage** page → **Hot Cold** tab → Properties→ *Polling Interval*.
  - The PID value should be set/tuned to ensure correct temperature control especially if precise temperature is desired (the default P=1.50, I=0.15, D=3.00, PID Interval=60 is only optimized with 10-60 s reading interval).
  - > Confirm the **Sensor Type** is set to *RTD* and **Relay Enabled** is *True*.
- During the initial pre-cooling of receiver and rapid sample cool-down, the heater *Power Limit* can be set to 0% in **Sample** session → **Stage** page → **Hot Cold** tab to prevent unexpected heating. Before **Backfill Intro** and when the intro is up to air, heater *Power Limit* should be set to 0%.
- Mount the glove box and hook the N<sub>2</sub> line. Purge the box with continuous flow of N<sub>2</sub> by open the green valve on the isolation platform. The humanity should drop to <5% and the dew point (t<sub>d</sub>, switchable by pressing the ▲ of the sensor) <-20 °C. Press-and-hold the power bottom for 2 sec from off state will make the sensor to enter the setting mode for turning ON/OFF of auto-off function.</li>
  - Close the by-pass valve for the intro volume of glove box and lock the isolation door with the two clamps can achieve low humanity in the main volume faster.
  - For introducing anything into the main volume of glove box, make sure the isolation door is locked with the two clamps. Open the by-pass valve then open the front door of the intro volume then put the required parts into the intro volume. After the humanity dropped to <10%, open the isolation door and transfer parts into the main volume.</p>
- 2. Move the stage to the *Intro* position.
- 3. Make sure the Type in TOF session  $\rightarrow$  Data Manager page  $\rightarrow$  Sample tab is set to 100 *mm* before Backfill Intro and removing the platen.
- 4. Loose the 4 screws on the standard sample platen with 2.5 mm hex driver then remove its receiver (6 screws) with 3 mm hex driver.

- 5. If plastic heater is not installed or was removed due to the use of the Zalar rotation or high-temperature platen,
  - i. Remove the protective supporting plate at right-hand side with by loose (not to be removed from the plate) 4 screws (with washers) using 1.5 mm hex driver.
  - ii. Place the plastic heater on the stage. The connector is at right-hand side. Snug (not tighten) the 2 screws with 1.5 mm hex driver to secure the plastic heater. Be careful of washer on the bottom side.
  - iii. Put the protective plate back. Snug (not tighten) the 4 screws with 1.5 mm hex driver. Be careful about the 4 ceramic spacers and washers.
- 6. Check the resistance of heater at right-hand side to be ~100  $\Omega$  between the connector pole. The bottom pole is ~1  $\Omega$  to ground.
- 7. Place the cryo receiver. The connector for thermo-couple is at left-hand side. At room temperature, the resistance should be  $\sim 100 \Omega$  between the connector pin and open to everything else.
  - Make sure the CuBe contact plate is firmly secure on the receiver. When install on the stage, make sure it does not get too close to the supporting plate around the connector pole. Loose connection or shorts due to thermal expansion will cause the temperature reading unstable.
- 8. Secure the 6 screws with 3 mm hex driver. Make sure the screws go through the respective hole of the plastic heater.
- 9. Pump Intro and wait for a good vacuum and allow V4 to open automatically (<5E–3 Pa).
- 10. Connect the  $LN_2$  tank (green outlet) and allow the  $LN_2$  to feed into the system. Make sure the plastic venting line on the wall works correctly.
  - > 0.05 MPa (7.25 psi) of LN<sub>2</sub> and open the liquid valve (green) by  $\frac{1}{4}-\frac{1}{2}$  turns would be more than enough.
  - Ice formation on more than half of the LN<sub>2</sub> evaporator indicates too much LN<sub>2</sub> is fed to the system.
- 11. In TOF session → Data Manager page → Sample tab, select Hot and Cold in Type or use Create Sample in System session → Sample Transfer page or right click on Acquisition Start button.
- 12. Pre-cool the receiver to -50~-80 °C by active Temp Control.
- 13. Prepare and mount sample on the holder and secure the 4 screws with 1.5 mm hex driver. The holder can be pre-cooled and keep in  $LN_2$ . The cap/sample/holder should have good thermal contact for consistent temperature.
- 14. When ready, **set Power Limit to 0% then Backfill Intro** to secure the sample holder with *4* screws with 2.5 mm hex driver on the receiver. **Pump Intro** as soon as possible. Minimize the exposure to atmosphere to minimize icing because the sample temperature is lower than dew point.
  - > Do not allow continuous exposure to air when the stage is cold.
  - ➤ To prevent ice-burn, the software does not permit to Backfill Intro if the temperature is outside of -10 to 50 °C. For pre-cooled stage that prevents water sublimation during pump down, temporarily set the Cold Backfill Intro Limit to be lower than the required temperature in Properties page. Set the limit back to -10 °C as soon as the Pump Intro started.
- 15. Set desired temperature for required operation. To prevent sublimation of water, <-100 °C maybe required. Regulate the LN<sub>2</sub> flow from the tank to achieve required temperature with minimal LN<sub>2</sub> consumption and vibration. Heater *Power Limit* can be set to 7~10% to avoid abrupt over-heating.
- 16. When the temperature and vacuum is stabilized, transfer the sample to analysis position.

➤ The sample cover is about 0.6 mm higher than sample. Therefore, avoid working near this region and move Z down (-0.6 mm) when moving to cover.

- 17. During the operation, check the  $LN_2$  level and  $N_2$  venting line frequently. Do not allow the flow of  $LN_2$  to stop.
- **18.** Check the O<sub>2</sub> monitor near the ceiling to be above 20% at all times. If it drops, leave the door open and use fan to draw outside air into the lab.
- 19. When finished, make sure the humanity is <5% then move the sample back to *Intro*.
- 20. Set **Temperature** to 25 °C and **Power Limit** to 60% and active *Temp Control*. Connect the  $LN_2$  line to the gas outlet (black) on the  $LN_2$  tank. Use the  $N_2$  gas to purge the  $LN_2$  line (open the valve by  $\frac{1}{4}-\frac{1}{2}$  turns would be enough) and wait for the stage to return to a safe temperature.
- 21. After the main chamber vacuum is recovered and stabilized at E-5 Pa, set Power Limit to 0% then set type in TOF session → Data Manager page → Sample tab is set back to 100 mm.
- 22. **Backfill Intro** to recover the sample (4 screws with 2.5 mm hex driver) cyro receiver (6 screws with 3 mm hex driver) and heating tape if non-standard platen will be used. Inspect the stage to be condensation free.
- 23. Put the standard sample receiver (6 screws with 3 mm hex driver) and its holder (4 screws with 2.5 mm hex driver) back on the system then **Pump Intro**.
  - Mind the orientation of the receiver the straight edge to the right needs to be parallel to the support and Faraday cup of standard platens should be at bottom.
  - When stage is cold and the intro was exposed to air for too long, the system might not pump down successfully (if it cannot reach 7E-3 Pa in 60 min, the process will give up and V3 will be closed automatically). In this case, **Pump Intro** again. After V3 is opened automatically, abort the vacuum task and open V3 manually with admin or service mode to keep it pumping.
- 24. Put the standard back-load platen back under vacuum and close the  $N_2$  gas purging valves when finished.
- If the vacuum spiked during the sample extraction and caused DSI shutdown, it is possible to use the **Diagnostic** page (**General** tab) of **Analyzer** to reset the shutdown state. If necessary, re-establish communication by *Reinitilize* **E-Neut** and **Gas Gun** in their **Diagnostic** page and switch status of other guns. For **LMIG**, **C60** and **GCIB**, reload setting could help re-establish communication.
- If necessary, close the software, power cycle electronics, then start the software again. Note that LMIG will crash when power cycle electronics so that **turn off LMIG emitter before power cycle**.

### HIGH TEMPERATURE OPERATION (not open for general use)

- The holder is meant for 150-600 °C. Heater *Power Limit* should be <40%.
- The maximum sample size is 10 mm × 10 mm, 2 mm in thickness.
- Do not use adhesive to secure the specimen.
- Refer to "sample platen" file for how to use the high temperature stage correctly.
- DO NOT TIGHTEN SCREWS, JUST SNUG THEM.
- BEFORE BACKFILL INTRO AND WHEN INTRO IS EXPOSING TO AIR, SET POWER LIMIT TO 0% in Sample session → Stage page → *Hot Cold* tab.
- MAKE SURE THE TEMPERATURE CONTROL IS INACTIVE AND HEATER POWER LIMIT 0% WHEN PLACING/REMOVING THE PLATEN.
   > Right-click Temp Control button can show the temperature log.
- WHEN SAMPLE IS AT INTRO, V4 NEEDS TO BE CLOSED.
- The default temperature reading time is every 60 s and acquisition/sputter will be paused during temperature reading. If necessary, change the setting in **Sample** session → **Stage** page → **Hot Cold** tab → Properties → *Polling Interval*.
  - The PID value should be set/tuned to ensure correct temperature control especially if precise temperature is desired (the default P=0.05, I=0.20, D=1.00, PID Interval=60 is only optimized with 10 s reading interval).
  - > Confirm the **Sensor Type** is set to *RTD* and **Relay Enabled** is *True*.
- 1. Make sure the Type in TOF session  $\rightarrow$  Data Manager page  $\rightarrow$  Sample tab is set to 100 *mm* before Backfill Intro and removing the platen.
- 2. Loose the 4 screws on the standard sample platen with 2.5 mm hex driver then remove its receiver (6 screws) with 3 mm hex driver.
- 3. If plastic heater is installed,
  - i. Remove the protective supporting plate at right-hand side with by loose (not to be removed from the plate) 4 screws (with washer) using 1.5 mm hex driver.
  - ii. Undo the 2 screws that secure the plastic heater using 1.5 mm hex driver and snug (not tighten) the connector with 1.5 mm hex driver. Be careful of washer.
  - iii. Put the protective plate back. Snug (not tighten) the 4 screws with 1.5 mm hex driver. Be careful about the 4 ceramic spacers and washers.
- 4. From the bottom of the *Hot Platen*, check the resistance of heater to be  $\sim 20 \Omega$  and thermocouple to be  $\sim 100 \Omega$  (at room temperature).
- 5. Mount the *Hot Platen* in the same manner of Standard 100 mm Platen.
- 6. **Pump Intro** and wait for a good vacuum and allow V4 to open automatically (<5E–3 Pa).
- 7. In **TOF** session → **Data Manager** page → **Sample** tab, select *High Temp (Hot)* in **Type** or use *Create Sample* in **System** session → **Sample Transfer** page or right click on Acquisition Start button.
- 8. Close V4 then active *Temp Control* and slowly increase the **Temperature** to desire value. Watch the change in vacuum (<5E–3 Pa) during the initial heating.
- 9. When the temperature and vacuum is stabilized, transfer the sample to analysis position.
- 10. When finished, move the stage to Intro position and set Temperature to 25 °C.
- 11. Wait for the stage to return to room temperature.
- 12. Make sure the Type in TOF session  $\rightarrow$  Data Manager page  $\rightarrow$  Sample tab is set to 100 *mm* before Backfill Intro and removing the platen
- 13. Put the standard back-load platen back under vacuum.

### FAST ZALAR ROTATION OPERATION (not open for general use)

- The size of specimen is limited to be smaller than 2" in diameter and 10 mm in thickness.
- Only the rotation center of the motor does not have XY offset during rotation. Therefore, only the 1 position is useable in each sample load.
- This holder is higher than that of back-load platen so that the Z is ~2.5 mm.
- Refer to "sample platen" file for how to use the high temperature stage correctly.
- DO NOT TIGHTEN SCREWS, JUST SNUG THEM.
- 1. Loose the 4 screws on the standard sample platen with 2.5 mm hex driver then remove its receiver (6 screws) with 3 mm hex driver.
- 2. If plastic heater is installed verify the resistance of heater feed poles at right hand side.
  - i. The resistance should be  $\sim 4 \text{ k}\Omega$  between the poles (resistance is from electronics).
  - ii. If the resistance is  $\sim 100 \Omega$ , remove the upper screw using 1.5 mm hex driver through the hole of supporting plate to disconnect heater. Because the motor is driven through the same poles of heater, heater must be disconnected.
- 3. Install the receiver plate for rotation platen (6 screws) with 3 mm hex driver. Be careful not to damage the heater tape if it is present.
- 4. Mount the Fast Zalar Rotation Platen in the same manner of Standard 100 mm Platen.
- 5. Mount the sample to the holder in the same manner of back-loading holder.
- 6. Insert the sample holder to the platen. Mind the alignment pin so the holder does not stick out.
- In TOF session → Data Manager page → Sample tab, select Fast Zalar in Type or use Create Sample in System session → Sample Transfer page or right click on Acquisition Start button.
- 8. In **Sample** session  $\rightarrow$  **Stage** page  $\rightarrow$  **Fast Zalar** tab, click *Fast Zalar* to start rotation briefly and make sure is secure. Do not allow it to rotate for more than 1 min under air.
- 9. Transfer sample to the analysis position as normally do.
- 10. Since only the rotation center of the motor does not have lateral movement, acquisition should be done at this position. The reference position *Zalar Center Position* can be obtained in **Sample** session  $\rightarrow$  **Stage** page  $\rightarrow$  **Stage** tab.

### LMIG Timing

### 2017/10/28

	$Bi^+$	$Bi_{3}^{++}$	$Bi_2^+$	${\rm Bi_5}^{++}$	$Bi_3^+$
	(24%)	(12%)	(18%)	(7%)	(27%)
Buncher Start Time (µs)	4.8100	4.8080	4.7350	4.7130	4.6930
Buncher (V)	800	725	690	570	565
Quad Blanker Start Time (µs)	3.3250	2.9400	2.6350	2.3780	2.1350
Quad Blanker Duration ( $\mu s$ )	0.1500	0.1500	0.1500	0.1500	0.1500
Quad Blanker Amplitude (V)	30	30	30	30	30
LMIG Blanker Start Time (µs)	5.000	5.000	5.000	5.000	5.000
LMIG Blanker Duration ( $\mu$ s)	1.000	1.000	1.000	1.000	1.000

# <u>*C*<sub>60</sub><sup>+</sup> Timing</u> (410 °C)

Buncher Start Time (µs)	2.3100
Buncher (V)	300
Pulser1 Start Time (µs)	1.0000
Pulser1 Duration (µs)	0.1000
Pulser1 Voltage (V)	200
Pulser2 Start Time (µs)	3.6450
Pulser2 Duration (µs)	0.075
C60 Pulser2 Voltage (V)	200

#### Raster Offsets

	Extractor	Immersion Lens	DEM Steering			
	Pulser		Х	Y	R	
+ion	0	6360	6.4	-4.8	2.0	
-ion	0	6450	-4.8	0.0	180.6	
+ion	-2000	7550	13.6	-4.8	2.0	
-ion	+2000	/550	-2.0	-9.0	180.6	

	Extractor	<b>Detector Offset</b>		LMIG	Offset	C60 Offset	
	Pulser	Х	Y	Х	Y	Х	Y
+ion	0	2.8	8.4	122.0	-34.0	-721.0	224.0
-ion	0	-4.0	-1.6	-100.0	-22.0	-144.0	177.0
+ion	-2000	4.8	6.8	182.0	-30.0		
-ion	+2000	-4.8	-1.6	-208.0	-23.0		