

How to Operate the Nu Plasma MC-ICP-MS

A quick guide

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Note: This is a complement reading of the *Nu Plasma* manual

How to Start

- 1- Switch on the **Compact computer** and the **monitor** (**Login Name = Nu-Plasma; Password = plasma\$16a**). NOTE: The computer might already be “ON”.
- 2- Single click with the left mouse button on the “**Plasma.exe**” program located in the toolbar at the bottom screen. Go to “**Control**” menu and select “**Preferred**”.
- 3- Check the **torch, gas connections** (*Cool, Aux, Neb*) and **cones** for any unusual arrangements or conditions. (You might want to clean the cones before beginning your session. Check that the safety control switch for the hood is in the position released (unlocked). Move the torch “back” to the rear closure, under Plasma Control window, and inspect visually the sample cone. In this case you must, however, received a cleaning demonstration before hand from Claude or Bill. *** *Please* *** *wear gloves when working underneath the Plasma Hood. This will avoid having grease from hands on the glassware and cones.*

Figure 1 (not available yet)

- 4- Set up your sample introduction configuration to the torch (i.e., *Scott double pass, Cyclonic Spray chamber, Aridus, LASER*, etc.). Connect the **red wire** from the electronic relay located above the torchbox and the stainless steel Plasma Shield. The **Plasma Shield** should be located *symmetrically to the RF Coil* (~1 mm apart) and **inserted** between the **Si-glass torch** and the **Si-bonnet**.

Figure 2 (not available yet)

- 5- Close the hood and make sure it’s well locked with the pneumatic piston using the “**HOOD LOCK**” chrome toggle switch on the instrument bench (the small **PVC grey box**).

Figure 3 (not available yet)

- 6- Check **room temperature** (*should be below 20-22 °C – 67-71 °F*). With the plasma and extraction rotary pump off. If not call **CCMS HVAC Control at x52222** and specify a temperature problem for CHEM-0225 room.

- 7- Check **compressed air pressure** (should read 80 psi from the gauge located inside the lab along the corridor wall behind the *ELEMENT2*). The **yellow handle should be parallel with the copper pipe**.

Trip 000008 means that the compressed air is off or too low

Figure 4

- 8- Switch on the **RF generator** located behind the instrument bench (the white switch in front of the **RFPP unit**).

Figure 5 (not available yet)

- 9- Select the fan extraction knob to maximum speed.

Figure x (not available yet)

- 10- Check the **Ar-dewar (tank)** main gas valve located inside the gas cabinet (the one labelled **GAS USE**). Pressure should be maintained between 110-120 psi using the “**stainless steel regulator**” (SS regulator) and the “**Pressure Builder valve**”. The last one is located behind the **GAS USE** exit port.

What to do if the pressure on the SS regulator is less than 80-85 psi:

Set the “**Pressure Builder**” knob to 120-140 psi (chrome knob that can be turned clockwise or counterclockwise). Then open the valve located below written “**Pressure Builder**”. **Stay there** and watch the pressure increase with time on the SS regulator. Control the amount of building pressure with the “**Pressure Builder**” valve and the position of the chrome knob to ≈ 95 -115 psi.

Figure 6

Need another Ar-dewar:

You will have to call **Roberts Oxygen Co. at: 301-927-5600** and ask for a new 200 liter Ar-dewar delivery (part# RLN178). They provide next day Ar-dewar delivery. The price for a 200 liter of liquid Ar from Roberts Ox. is approx. \$140.65 (**can be paid by VISA on Roberta’s credit card using the PlasmaLab account**). For the high-pressure backup cylinders, the part number is R104D3 at a cost of \sim \$80 (300 ft³ of Zero Grade Argon). Their price includes the Ar gas delivery at the gas cabinet (dewar or cylinders).

- 11- Go to next room 0227 (i.e., ICP Preparation Lab). Check the distilled **water level reservoir** from the front of the big **JULABO chiller (level should be between min and max)**. Switch on the chiller using the green knob located at the rear top on your left and then go back to the instrument room.

Figures 7a, -b

- 12- Check the vacuum conditions on **Pirani-1** ($\approx 1.5E-3$ mBar), **Pirani-3** gauge ($\approx 2.5E-4$ mBar), **Penning** gauge ($\approx 3.6E-8$ mBar) and the **ionic pump** ($\approx 1.9E-9$ mBar) (refer to the “**NU Plasma Vacuum Schematic**” plastic-coated colour picture for further information). If vacuum state is satisfied then proceed ahead. If not, then **check the turbo pump speed and ion pump condition** (not in “**standby**” condition / please refer to the manual for more details or contact me at home: **CDalpe @ 9-301-441-1392** or call the factory @ **011-44-1978-661-304**). *There is also a section below concerning the different steps to follow when the instrument has experienced a power shutdown.*
- 13- Double-click with the left mouse button the “**Cool Gas**” valve gray square at the bottom right of torch schematic (inside the **Plasma Control** window) in order to flush trapped “**air**” inside the torch. This will help plasma ignition.

For the Aridus user:

Turn “**on**” the **Aridus** from behind the beige box and install the spray chamber + the neb (**T1H** from **CETAC** or **PFA** from **Elemental Scientific Inc.; referred as ES**) on the **Aridus**. Click on the “**Open Valve 1**” inside the “**Switches**” menu from the Nu Plasma software and then regulate the pressure to “**neb**” via the **black regulator** on the Aridus table to 0 psi (i.e., no pressure). Preset the “**Sweep gas**” flow rate between 0.8-1.0 L/min. If you use the **CETAC spray chamber you must select** the set heater temperature to **70°C max (VERY IMPORTANT)**. If you use the **ES** spray chamber (PFA) you can select the heater temperature up to a maximum of 100-105°C (normally we run at 100°C). To change the temperature heater for the **Spray Chamber** press the blue set button and then simultaneously press the “**up**” or “**down**” grey arrow button to reset to the new temperature. Wait until both heaters reach their setting point temperature (i.e., 70° or 100°C for the spray chamber and 160°C for the “**desolvator oven**”). During this stage set the “**neb**” gas flow rate from the Plasma software to 0 L/min. No need to set the mass flow controller when you connect the Aridus. If you are not ready to ignite the plasma close the “**neb**” **solenoid valve** by removing the tick located beside “**Neb Gas**” under “**Plasma**” inside the “**Digital Control**” window. By doing this you will avoid wasting Ar gas.

Figures 8a, -b, -c, -d

For the Cyclonic Spray Chamber:

Make sure that you connect correctly the cooling system of the **NESLAB chiller** (Tygon tubing from the RTE-101 unit), the drain to the **Peripump**

and the micro-concentric nebulizer to the spray chamber. Installed correctly the Black/White Tygon tubing around the **perilstatic pump** (pump turn clockwise) and the end part inside the “**Waste**” bottle (the one that has an hole in its lid).

For the Scott-Double pass + Peltier Cooling system:

Connect properly the neb and drain to the spray chamber. Then make sure the “**Peltier**” option in the “**Plasma Control**” panel selected. You can modify the cooling temperature by pressing the up or down arrows (normal setting point around 4 °C). The 4 mm blue polyurethane tubing must be connected to the base of the “Peltier” heat sink in order to remove the extra heat during the run “before switching ON the large Julabo chiller”. The large Julabo chiller is responsible to cool down the heat sink.

- 14- Insert the Polymer, PTFE or PFA sampler tubing into MilliQ water or 2% HNO₃.
- 15- The instrument is now ready for ignition.
- 16- Press the green “**START**” button inside the “**Plasma Control**” window from the Nu Plasma software and watch the process. Should take approx. 90-120 sec.
- 17- It might be possible that you will need to adjust the **Air Capacitor** position (up or down arrows) during the ignition. However, I strongly recommend that you allow the software the first three trials. A good guess for the **Air Capacitor** and the **Vacuum Capacitor** should be around **95-100** and **6500-6600**, respectively, when a new plasma shield is used.
- 18- After ignition:
For Aridus users:
 - Increase the Ar-sweep gas with the CETAC mass flow controller to 2.5 L/min and the “**neb**” pressure, with the black regulator, to **≈40 psi**. for the **PFA-ES** nebulizer or **≈60-80 psi** for the **T1H-CETAC** nebulizer. No need at this point to increase the nitrogen (or helium) additional gas.

See Figure (not available yet)

- the software will go into a start up routine where the backing valve will close, the first LOS will open, the backing valve will reopen, and then the High Tension (HT) will switch on.

- 19- Wait 10-20 sec. before switching “**on**” the “**Plasma Shield ON**” (under the **Switches** window).

- 20- You must **open manually the second LOS** from the “**Valve Control**” window if you want to check at this point the instrument signal intensity. To do this **double-click** on the **dark grey symbol** representing the second LOS (if you don’t have any response at this stage please check if you still have compressed air again; see Pt. 7 above).

Quad 1 = set value for your isotopic system (e.g., Quad 1 for Li = -0.3).

Quad 2 = set value for your isotopic system (e.g., Quad 2 for Li = 46).

Those Quad values can be modified for other isotopic systems under **Setup menu in Quad Lens Setup**.

Also please **make sure** that those lenses are set to **0 volt**:

LIN 1 = 0.00

LIN 2 = 0.00

LIN Corr = 0.00

Cubic = 0.00

Q15 = 0.00

Q16 = 0.00

under “**Deflector and HT Supplies Control – QUADS**).

- 21- A trip message such as:

0000001

or

0000101

or

0000100

or an ERROR message such as:

00200000

means the neb flow rate generated from the Aridus” is too low. Most of the time this is created by a discharge between HV1 and HV2. Adjust the neb pressure (black regulator) and sweep gas according to the step #17 above and then reset the trip by clicking under “**Protection**” menu the “**Reset Trips**” bar. If you still have the appearance of the trip, switch off the “**PlasmaShield**” (under Switches), switch off the “**HT**” (under **Digital Control – Power**), reset the trip again (should disappear at this point if not please call CDalpe). Then switch “**on**” the “**HT**”... wait 10-15 sec...switch “**on**” the “**PlasmaShield**”.

The instrument should be fine from then. You should never get involve with this hassle if you use a regular spray chamber such as the “Scott double” pass or the “Cyclonic” models.

- 22- *Let the instrument sit in this configuration for the next 20-30 minutes* in order for the instrument to achieve its new vacuum conditions and plasma stability.

- 23- *Typical Signal Intensity:* Signal intensity can vary from day to day basis. However, it is strongly dependent regarding the sample system introduction

you use, the neb aspiration rate (Micromist, PFA-Elemental Scientific, T1H-Cetac, etc.), tubing I.D., gas flow, quality of cones, tuning configurations, etc.

- 24- *Tuning steps:* I strongly recommend to start with:
- **Sweep Gas flow rate** (between 2.00-5.00 L/min for Ar and 8-25 ml/min for N, no miracle values). Make sure if you use nitrogen that the Nitrogen cylinder valve is open and the regulator is set to 50 psi. (cylinder located at the left of the big chiller in 0227).
 - the **source** (torch position controlled via the Up/Down – In/Out – Backward/Forward arrows under “**Plasma Control**” window, neb pressure/flow rate (neb regulator or neb mass flow controller), *then*
 - **HV1** (the plateau intensity should be achieved between 4002-4008 volts)
 - **HV2**
 - Deflectors “**Source H1**” and “**Source V1**”
 - **HV3**
 - Deflector “**Source H2**”
- Then into the transfer chamber:*
- **HV6**
 - Deflectors “**Transfer V2**”
 - Deflectror “**Transfer H1**”
 - **HV5**
 - Deflector “**Tranfer V1**”
- (Please refer to the plastic-coated “**Location of Nu Plasma Ion Beam Steering and Focusing Potentials**” color diagram).
- 25- For further instruction please refer to the white binder entitled “**Nu Plasma Operating Manual**” located in my office.

How to Shut-off the Plasma

- 1- Clean the **Polymer/PTFE** tubing of the neb with **2 or 5% nitric acid** for 90 sec and then with **MilliQ water** for another 90 sec. CETAC recommends to aspirate air for their Aridus for another 90 sec in order to dry the “**desolvator cell**” before shutting down the Aridus.
- 2- **Switch OFF** the “**Plasma Shield**” from the “**Switches**” window.
- 3- Press the red “**STOP**” button from the “**Plasma Control**” window.
Aridus Users:
 - Press the “**Close Valve 1**” from the “**Switches**” window under Plasma software (this will close the argon line supplying the desolvator cell, i.e. sweep gas, and the nebulizer gas, i.e. the black regulator). Turn counterclockwise the black knob on top of the small regulator until you feel no friction; i.e. minimum pressure.
 - Switch off the power supply of the “**ASX-100**” (the red knob located behind the autosampler).
 - Switch off the “**Aridus**” using the black switch located behind the unit (located below the “Ar In” gas tubing).
 - Close all main gas valves of each high pressure cylinders located in 0227 (Nitrogen and/or Helium; clockwise).
- 4- Switch off the big **JULABO chiller** in room 0227 (the green knob located near the “top left”, on front of the chiller).
- 5- Switch off the **RF generator** located behind the instrument bench (the white switch in front of the RFPP unit).

Figure x (not available yet)

- 6- Select the fan extraction knob to minimum speed.

Figure x (not available yet)

- 7- Close the green “**GAS USE**” valve from the **Ar-dewar** (turn clockwise). Close the “**Pressure Builder**” valve (if it was open).

Figure 6

- 8- Export your data to the computer via the network.
- 9- Shutdown the **Window NT** on the “**Gateway**” computer. The monitor will turn to sleep mode 20 sec. after you shut down the computer.
- 10- **Remove Polymer or PTFE tubings** from any solution to avoid spillage during night.
- 11- ***Release the tension*** on each tubing attached to the peristaltic pump (Gilson Peripump; Aridus Peripump located behind the unit).

Figure 10 (*not available yet*)

- 12- *I recommend to remove the spray chamber + neb from the Aridus heater and then to store both parts under the fume hood in 0227. Be careful the spray chamber might be very **HOT**. Please cover the hole of the spray chamber oven with “**Parafilm**” + masking tape to avoid falling debris from the HVAC system.*

How to Reset the Instrument Configurations After a Short Power Outage

- 1- From the Nu Plasma software go to the “**SETUP**”, than under preferences unclick “**Run OFF Line**”.
- 2- Again from the “**SETUP**” menu click on “**Mass Spec**” and than answer “**YES**” (it will take a few seconds).
- 3- Again from the “**SETUP**” menu go to “**Constants**”, “**Instrument**”, and then “**Initialise**”. Answer “**YES**”.
- 4- Under the “**Protection**” menu bar clear the “**tick**” beside the “**Instrument Settings**” and the “**Vacuum**”.
- 5- Go to “**Setup**”, “**Contants**”, “**Tuning**”, “**Initialise**” (e.g., Lithium-2 (1mm spacer.tun; for lithium setting conditions).
- 6- The #\$\$@ **Trip** is still there...don’t panic it can be worse... The problem can be resolved if you reset manually the “**Discriminator Voltage**” on each “**Ion Counter**” (**IC1**, **IC2**, **IC3**). To do this you must go under the “**IC Supplies**” window and then under “**Disc**” you insert manually the new values of -0.110 (minus), -0.190 (minus), -0.160 (minus) Volts for **IC0**, **IC1** and **IC2**, respectively (instead of 0.000 volt).
- 7- Go to “**Protection**” and click again on “**Reset Trip**”. The “**trip**” should be erased from then.
- 8- “**Ion Pump**”. You must restart the “**Ion Pump Power Supply**” manually following those steps:
 - Walk behind the instrument near the “**RF PP Generator**”. The “**Ion Pump Power Supply**” is located just above the “**RF PP**” written “**Digital MPC**” for “**Multiple Pump Control**”. From the “**Supply 1**” (top green “**Digital Display**”) it should be written at this stage “**Standby**”
*** (if not then the Ion Pumps should work properly) ***

Figure 10 (*not available yet*)

This means that the “**Ion Power Supply**” do not provide any voltage to the three “**Ion Pumps**”. From the white keyboard press:

- (1) High Voltage Operate (dial #9)
- (2) Select “**on**” with the “**Display Select**” button.
- (3) Press then the “**Enter**” white button. The voltage should increase gradually up to **6-7 kV (approx. 6800 Volts)**.

9- You should be ready to operate the instrument in normal condition from then. Go to Point # 10 (under “**How to Start the Nu Plasma MC-ICP-MS**”) to verify the vacuum conditions (mostly the “**Analyser**”). You might have to restart the “**Penning**” gauge manually under “**Digital Control**” and then tick the Penning gauge box. It will switch to “**OFF**” state if the vacuum condition inside the “**Transfer**” chamber is above **5E-6 mbar**.

10- Good luck!

***** END *****

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