

Operations and Installation Manual

Pulsed Dynamic Focusing (PDF) Technology for AP/MALDI

October, 2003

This product is to be used only in laboratory conditions by trained and skilled technicians and in accordance with these operating and maintenance instructions. Primary safety is ensured by correct assembly of the instrument and by following the instructions and warnings in this manual. To ensure continued safety, please ensure that any voltage source connected to this product has a suitable safety ground and complies with local and national electrical regulations.



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PREFACE



UNDER NO CIRCUMSTANCES should you open the PDF unit as this would expose you to possible electrical shock.

During operation, the PDF module is completely safe to use as long as the precautions for AP/MALDI * are followed.

DO NOT ATTEMPT services or repairs that are not covered in the Troubleshooting section, section 5 of this manual. For services and repairs beyond those specifically provided in the Troubleshooting section, contact the manufacturer:

MassTech, Inc. 6992 Columbia Gateway Dr; Columbia, MD 21046
(443) 539-1758.

*** See the AP/MALDI Installation, Operation, and Maintenance Manual – you may request this manual from MassTech, whose address is on the front of this PDF manual.**

1. INTRODUCTION: PDF TECHNOLOGY FOR IMPROVED SENSITIVITY

AP/MALDI has conventionally used continuous electric fields to extract ions into a mass spectrometer. Pulsed Dynamic Focusing (PDF) Technology applies a new electric field scheme whereby the extraction field is applied for only a brief timed interval after the laser pulse. By removing the electric field while ions are in transit from the target plate surface to the entrance of the mass spectrometer, ions avoid being lost to the entrance tip and walls and are instead entrained by the gas flow into the MS. The technique is termed Pulsed Dynamic Focusing because the high voltage (on the target plate) is *pulsed* to zero potential at the optimal time, i.e., *dynamically*, to *focus* the ions into the MS. PDF Technology significantly improves the signal level and reliability of AP/MALDI.

2. PDF PRINCIPLE OF OPERATION

The voltage scheme for PDF is described in Figure 1. Each laser pulse is used to trigger a high voltage switch, after a user-defined timed interval (pulse delay). The switch immediately grounds the HV for a hold time of >1ms, and then afterwards allows the HV to return back to its original level.

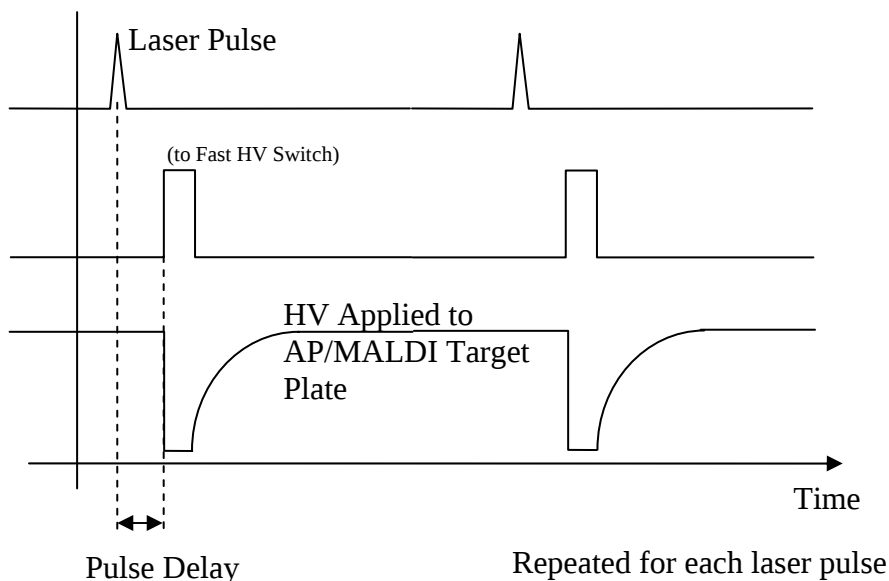


Figure 1. Voltage scheme for AP/MALDI PDF

PDF improves S/N ratio at higher voltages. PDF also allows larger laser spot sizes to be effectively utilized. The combination of working at higher voltages and utilizing larger laser spot areas result in a sensitivity improvement of over 10X in comparison to classical AP/MALDI without PDF.

3. PDF INSTALLATION

3.1 *Checking that all components have been received.*

Before you start installing your PDF module, ensure that all necessary Parts and Accessories have been delivered. Figures 2a and 2b below show these components and introduces some definitions and part names used in the installation explanations. Figures 3a and 3b show the PDF Module's front and rear views.

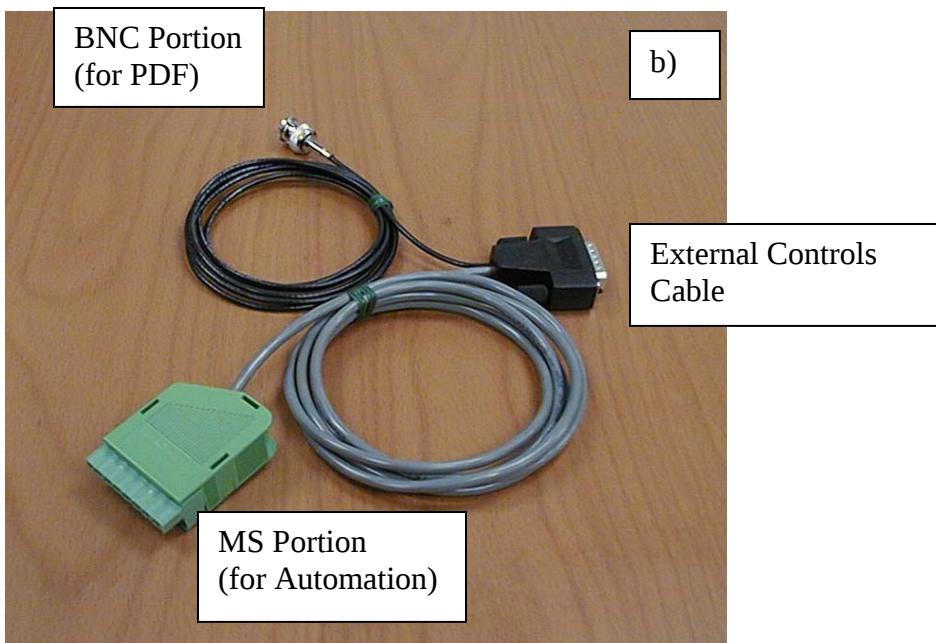
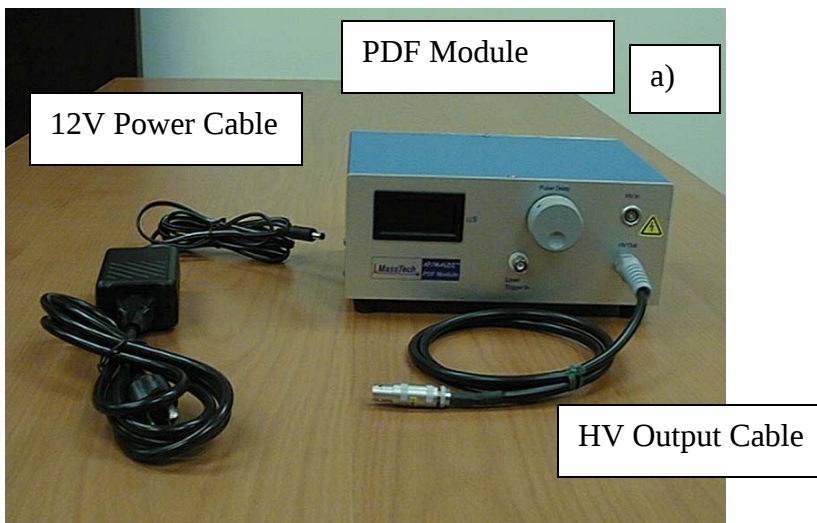


Figure 2a, 2b. Out of the box, these components should be present

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**Figure 3a, 3b. a) PDF Module Front View;
b) PDF Module Rear View**

3.2 Installation of PDF onto an AP/MALDI ion source.

Installing/Uninstalling: Before the PDF Module is installed onto the AP/MALDI ion source, or uninstalled, the LCQ instrument must be in either “Standby” or “Off” mode (the “Scan” light on the LCQ front panel must be turned OFF), and the AP/MALDI Control Unit should be turned OFF.

When uninstalling, again: make sure that the LCQ is in Standby or Off mode (“Scan” is not blinking); switch OFF the power at the rear panel of the AP/MALDI Control Unit; then start any disassembling operations or source detachments.

To begin installation of the PDF Module, first ensure that the AP/MALDI ion source is set up and tested. Figure 4 shows an AP/MALDI ion source attached to an LCQ instrument.

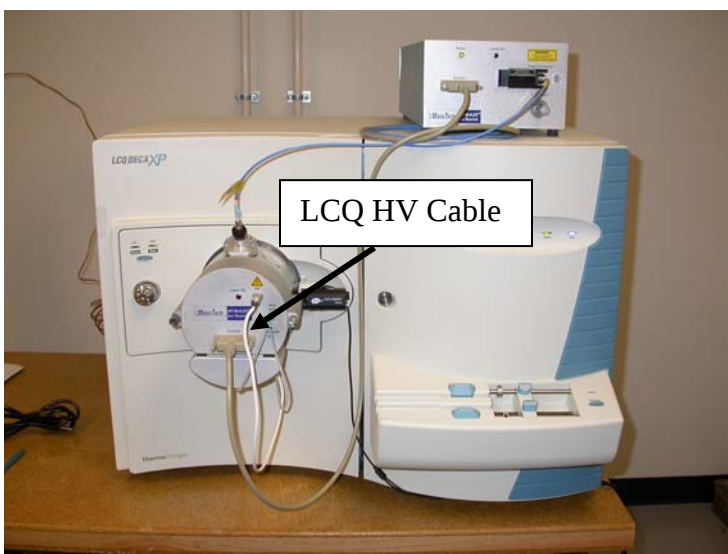


Figure 4. Classical setup of an AP/MALDI ion source with an LCQ instrument.

After the AP/MALDI ion source has proven to be operational, place the PDF Module within reach of the LCQ HV Cable. A good place to locate the PDF Module is on top of the LCQ instrument (Figure 5).

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Figure 5. PDF Module located in close proximity to the AP/MALDI ion source and LCQ HV Cable.

Plug in the 12V power supply to the PDF Module (Figure 6a), and then to an electrical outlet. Tighten the one end of GND to ion trap (Figure 6b). It is important to plug in this power cable prior to connecting any other cables, as it provides electrical protection to the user. After switching the PDF Module on, the light on switching block should come on (Figure 6c).

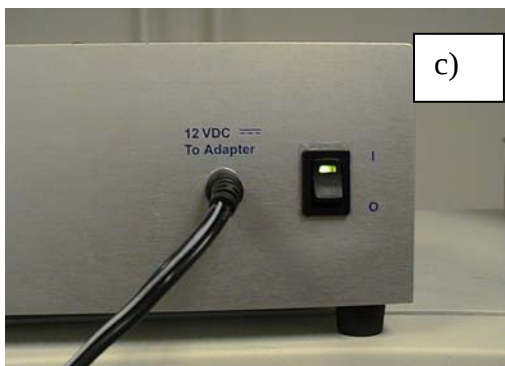


Figure 6a, 6b, 6c. a) 12V power supply receptacle on the PDF Module; b) Ground to Ion Trap; c) Light after switching on PDF Module



Figure 7a, 7b. Disconnect the LCQ HV Cable from the AP/MALDI ion source by **a)** loosening the rear nut on the LEMO connector and **b)** pulling the connector off.

Insert the LCQ HV Cable into the HV In connector on the PDF Module as shown in Figure 8. There should be a snap or click when the LEMO HV connector is properly engaged into its receptacle.

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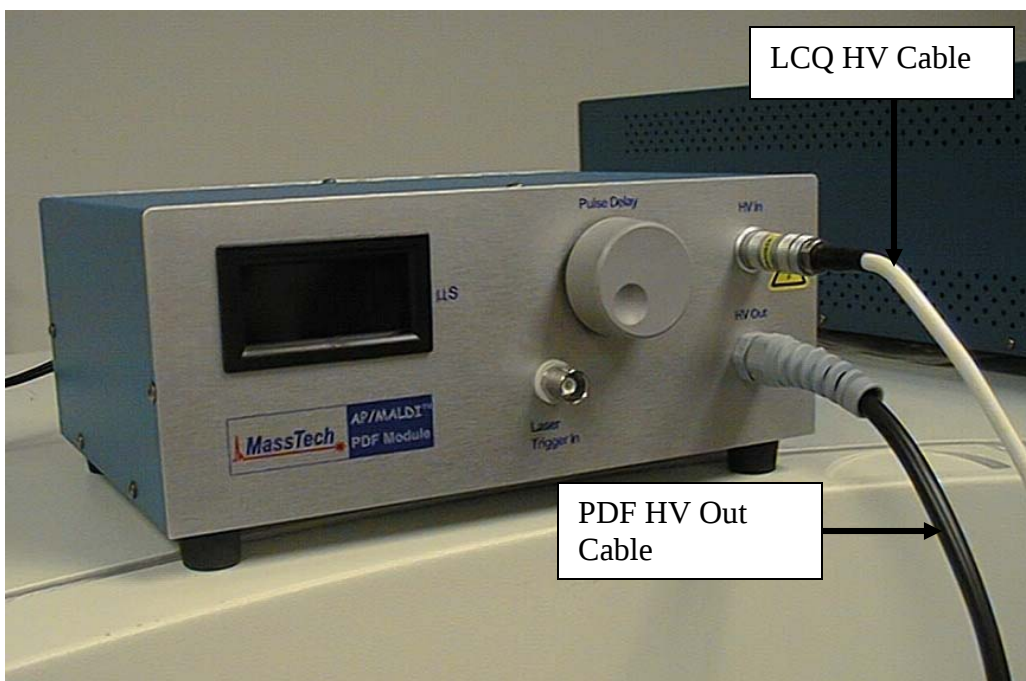


Figure 8. Connect the LCQ HV Cable into the PDF Module HV In receptacle



Figure 9. Connect the HV Out cable from the PDF Module into the AP/MALDI ion source.



Figure 10. Connect the External Controls Cable to the External Controls output on the rear panel of the AP/MALDI Control Unit.

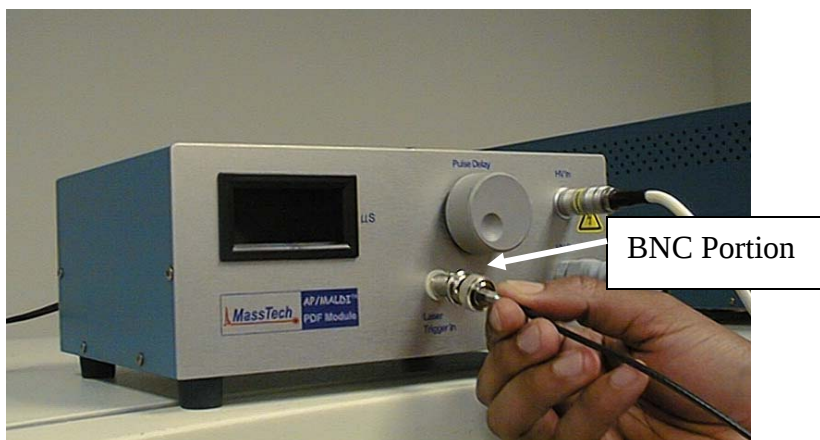


Figure 11. Connect the BNC portion of the External Controls Cable into the “Laser Trigger In” on the PDF Module.

The External Controls Cable can also be connected to the LCQ Peripheral Controls port should automated sample analysis be desired. Please consult the AP/MALDI manual for automated sample analysis.

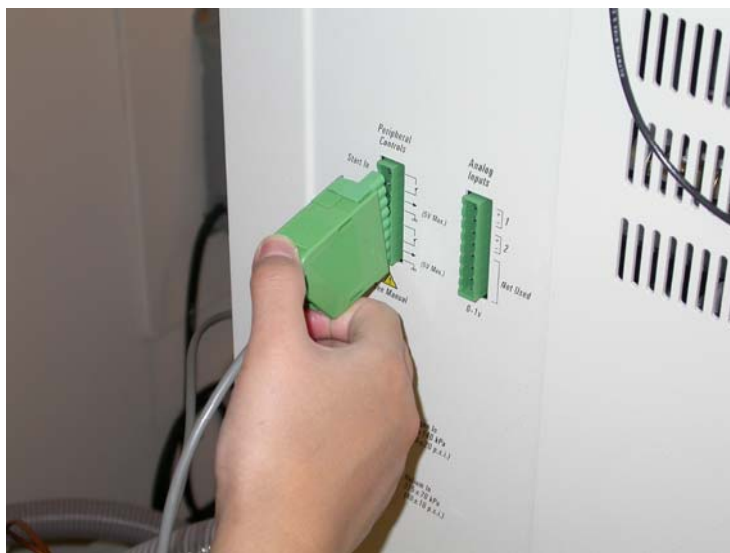


Figure 12. Connection of the External Controls Cable to “Peripheral Controls” on the LCQ Mass Spectrometer.

After all the cables are connected, the PDF Module should look as follows (Figure 13).



Figure 13. PDF Module fully connected to an AP/MALDI ion source

4. PDF MODULE OPERATION

The PDF Module allows the user to adjust the time interval that the HV is applied to the Target Plate on the AP/MALDI ion source. The time interval is controlled by rotating the knob, referred to as the “Pulse Delay” (because it is the *delay* time before the HV on the Target Plate is rapidly *pulsed* to zero voltage). This timed interval is displayed in microseconds on the front panel of the PDF Module (Figure 14).



Figure 14. Pulse Delay adjustment knob

To achieve the best performance from applying the PDF Module, the user should first operate the PDF Module in the “OFF” mode (rotate the Pulse Delay knob clockwise until “OFF” appears on the display ($>200\mu\text{s}$) – see Figure 6b). A standard peptide at 100 fmol level can be used to tune the AP/MALDI PDF setup (see AP/MALDI manual for sample preparation procedures).

Record the spectrum from classical AP/MALDI operation, making note of the signal level. Change the HV on the LCQ instrument Tune Plus page, so that the “Spray Voltage” is increased from classically 2.0kV to 4.0kV (Figure 15). This is the HV on the Target Plate.

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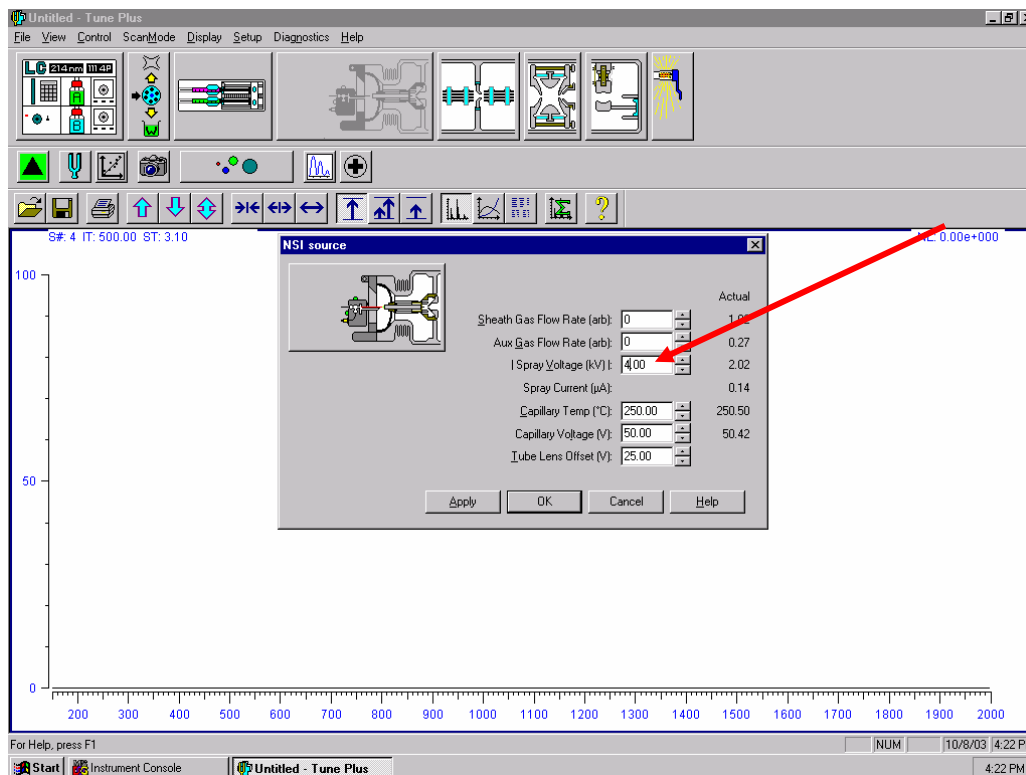


Figure 15. Changing Target Plate high voltage to 4.0kV

Adjust the Pulse Delay to around 20 microseconds, and adjust it +/-15 microseconds to determine the time for the highest signal level. When the AP/MALDI laser is firing, a small decimal point after the pulse delay time is shown that blinks at the laser repetition rate (Figure 16). This indicator is also useful to confirm communication between the AP/MALDI Control Unit and the PDF Module.

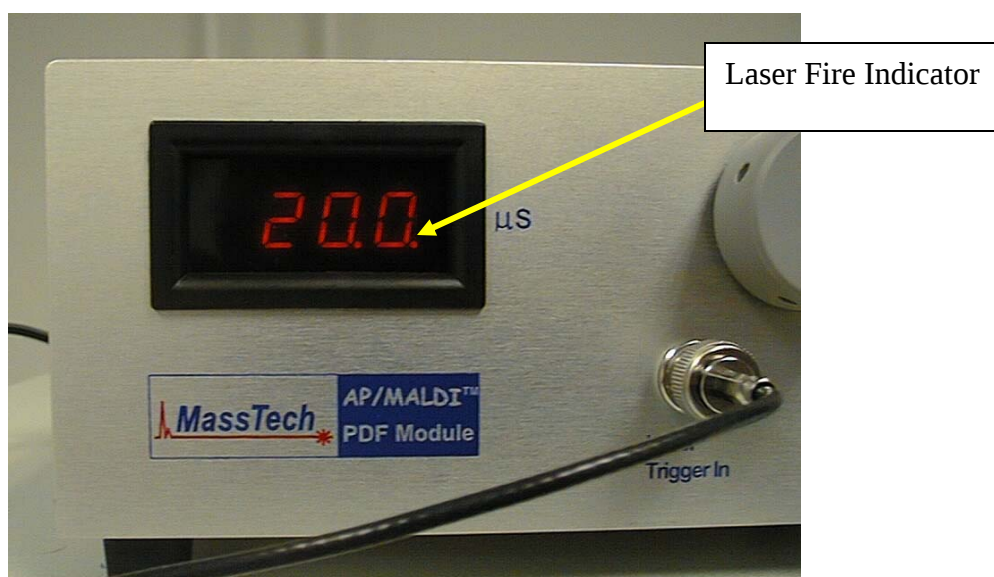


Figure 16. Indicator for Laser Firing on the PDF Module

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When the HV and Pulse delay are optimized, the improvement level should be between 2 to 4 times. A higher voltage can be used on the Target Plate, but above 4.5kV, electrical discharge and arcing may occur. It is not recommended that you operate the AP/MALDI PDF system above 4.5kV.

To further enhance the throughput of the AP/MALDI PDF setup, de-focus the UV laser by loosening the set screw and pushing in the focusing tube on the ion source about 1-2mm (Figure 17). There is a scale on the side of the focusing tube to facilitate this adjustment.

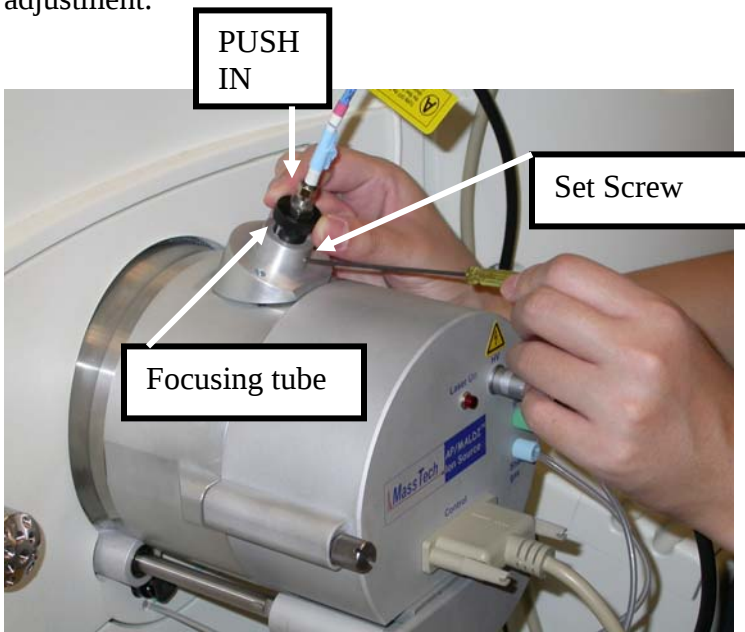


Figure 17. Adjustment of the focusing tube on the AP/MALDI ion source.

Then increase the laser energy on the AP/MALDI Control Unit (Figure 18) so that roughly the same laser fluence (energy/area) is maintained. The purpose behind this exercise is to generate more analyte ions per laser shot. With the PDF Technology, ions generated far away from the MS entrance can even be entrained into the MS.



Figure 18. Increase laser energy on the AP/MALDI Control Unit.

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Similar to the operation for optimizing the Pulse Delay, tune the focusing tube position and the laser energy, while optimizing the signal intensity for a standard chemical. AP/MALDI is sensitive to the laser fluence. If the fluence is too high, there can be increased chemical noise, and poor analyte S/N. If the fluence is too low, analyte peaks may not be present due to insufficient ionization energy. Carefully adjust the laser energy and laser focusing until the signal is optimal. An improvement in signal intensity by a factor of an additional 2 to 3 is reasonable in these laser-related adjustments.

5. TROUBLESHOOTING

DO NOT ATTEMPT services or repairs that are not covered in this Troubleshooting section. For services and repairs beyond those specifically provided in the Troubleshooting section, contact the manufacturer: MassTech, Inc. 6992 Columbia Gateway Dr; Columbia, MD 21046 (443) 539-1758.

5.1 *PROBLEM: Arcing & Electrical Breakdown*

Just above 4.5kV, arcing may begin to occur sporadically when the HV is near the threshold of breakdown. In some instances, arcing may not occur initially when a HV setting is applied, but may occur after the laser is fired due to additional electrons and ions generated from the laser, facilitating breakdown. At much higher voltages, continuous arcing can occur, where it is visible on the AP/MALDI imaging screen (Figure 19).

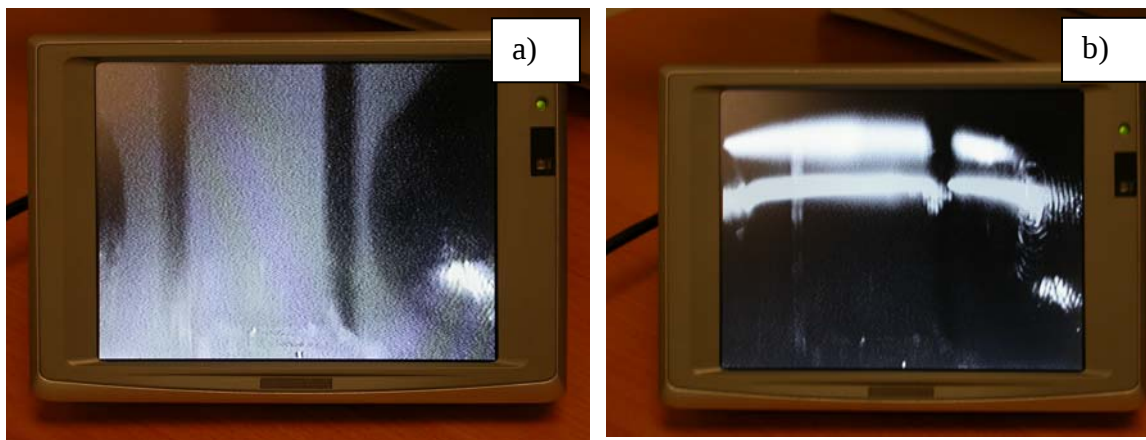


Figure 19. AP/MALDI imaging system showing **a)** normal Target Plate and MS inlet image, **b)** Arcing between the Target Plate and the MS inlet.

If an electrical discharge occurs, the HV should immediately be turned back down to an acceptable level. A check should be made to see whether the capillary Temperature read back on the Tune Plus page is off-scale. If the capillary Temperature read-back is not off-scale, you can continue operation of the MS system. However, in the event that the capillary Temperature readback is $>500^{\circ}\text{C}$, close Tune Plus, leave the computer on, and restart the hardware on the LCQ.

5.2 PROBLEM: Unexpected Spectral Response

Because AP/MALDI PDF allows many more ions into the ion trap than does classical AP/MALDI, it is important to be aware of space-charge effects that can occur in the trap. It may be necessary to reduce the injection time of the ion trap to 100ms so as to not saturate the trap (Figure 20). An alternative way to solve this problem would be to adjust the Laser Energy attenuator.

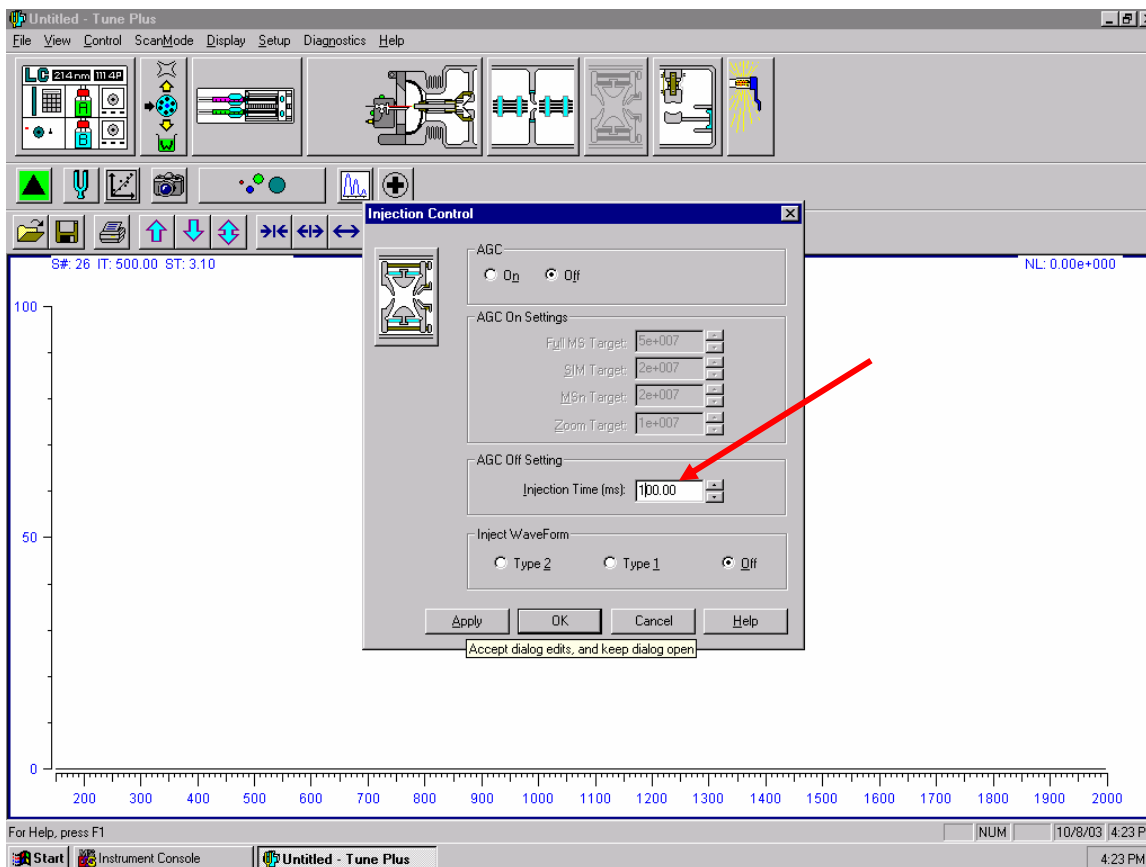


Figure 20. Lower the injection time for the ion trap to 100ms if space-charge effects are noticed.

We are ready to provide you any technical assistance! Call us at (443) 539-1758 or e-mail the problem to: msms@apmaldi.com

WARRANTY INFORMATION – SIX MONTH LIMITED WARRANTY

MassTech, Inc. provides to the original purchaser the following limited warranty from date of invoice.

MassTech, Inc. warrants each PDF module to be free from defects in material and workmanship. Liability under this warranty covers servicing of the module when returned from the customer's facility within the United States pre-paid to our factory. MassTech, Inc. will repair any component(s) or part(s), that it finds to be defective during the period of this limited warranty, which is six months from the date of invoice. Should a defect become apparent, the original purchaser must first notify MassTech, Inc. at (443) 539-1758 of the suspected defect and request a Return Merchandise Authorization number (RMA#). The PDF module (or suspect components) should be carefully packaged in the original container (if the original shipping container has been lost, trashed, or damaged, another one must be purchased from MassTech, Inc. prior to shipping). Then, mark the original container with the RMA#, and ship prepaid to:

MassTech, Inc.
6992 Columbia Gateway Dr;
Columbia, MD 21046
Attn: Service Dept.

The instrument will be repaired in the shortest possible time and returned prepaid by the same shipping method as received by the factory. During the warranty period, no charge will be made to you for parts, service, or labor.

This limited warranty is void if the module has been damaged by accident, misuse, negligence, act of God, or serviced (including opening of the PDF module) by any other person not authorized by MassTech, Inc. The warranty also does not apply to modules that have had the serial lot number altered, defaced or removed.

This limited warranty contains the entire obligation of MassTech, Inc. and no other warranties expressed, implied, or statutory are given. No representative or employee of MassTech, Inc. is authorized to assume any further liability or grant any further warranties except as set herein.

MassTech, Inc. disclaims liability for indirect, incidental or consequential damages. Exclusion or limitation of incidental or consequential damages are not permitted by some states and this limitation or exclusion may not apply to you. Warranty rights vary from state to state; and, therefore, you may have other rights in addition to those provided by this warranty.