ARI Aerosol Mass Spectrometer

Operation Manual

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Instrument Description

The ARI Aerosol Mass Spectrometer provides real-time size resolved composition analysis of volatile and semi-volatile particulate matter. The combination of size and chemical analysis of sub-micron aerosol mass loading with fast time resolution makes the ARI AMS unique.

Aerosol particles in the size range ~ 0.04 to ~1.0 micrometers are sampled into a high-vacuum system where they are aerodynamically focused to a narrow beam (~ 1mm diameter). The particle beam is directed onto a resistively heated surface where volatile and semi-volatile chemical components are thermally vaporized and detected by electron impact ionization quadrupole mass spectrometry.

Particle aerodynamic diameter is determined from particle time-of-flight (TOF) velocity measurements using a beam-chopping technique.

An optional optical module makes it possible to correlate light scattering, aerodynamic diameter and chemical composition analysis on a particle-by-particle basis for particles larger than 200 nm diameter.

Specifications

Performance

- Size-resolved mass analysis of non-refractory aerosol components.
- Chemical analysis by thermal vaporization and electron impact ionization mass spectrometry (unit mass resolution to 340 AMU, scan rate 1 msec/amu). Single ion detection ability.
- Real-time mass spectral analysis of inorganic (nitrate, sulfate, ammonium) and organic mass (OM).

- Particle vaporization temperature adjustable from 200 ^{o}C to 900 ^{o}C.
- Single-particle detection diameter approximately 100 nm.
- Sensitivity of ~ 0.01 mg/m^3 in several minutes.
- Sampling flow rate: 100 cc min^{-1}.
- Aerodynamic particle size measurement in the range of 40 nm to ~1 µm. Size resolution of 5 to 10 ($D_{aero}/\Delta D_{aero}$, FWHM) over that size range.
- Quantitative particle collection efficiency (~ 100%) in the range of 50 to 500 nanometers in diameter. Collection extends to particles about a factor of 2-3 smaller and larger with reduced efficiency.
- Fast time resolution of seconds. Maximum data rate ~100 Hz.
- Data output format: ASCII, HDF. Wavemetrics© Igor program license supplied.

Physical

- Size: Approximately 41" wide x 24" deep x 53" high.
- Weight: Approximately 170 kg.
- Power: Approximately 600 W. Universal power 110VAC/60Hz or 220VAC/50Hz. Vacuum system fully operational on 24 VDC.
- Packaging: Shipped in one reusable container. Total shipping weight ~280 kg. Approx. outside dimensions 30" wide x 51" long x 63" high on forklift skids.

Theory of Operation

Particle Beam Formation

The ARI Aerosol Mass Spectrometer measures the size and chemical composition of volatile/semi-volatile submicron aerosols. It provides composition information on ensembles of particles, with limited single-particle information. The instrument combines standard vacuum and mass spectrometric techniques with aerosol sampling techniques.

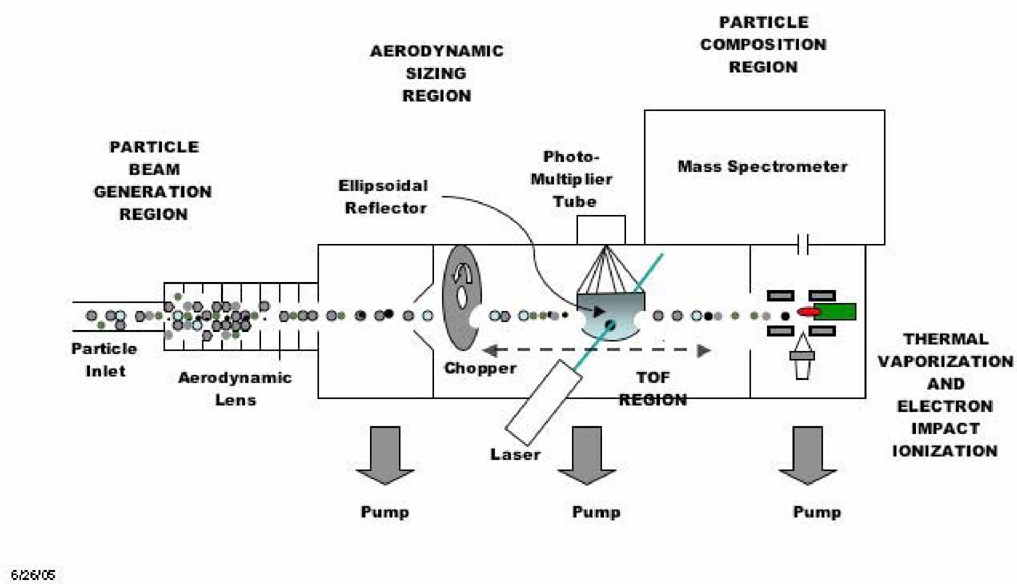

Figure 1: Aerosol Mass Spectrometer

Aerosols enter the AMS through a particle-sampling inlet that restricts the flow with a 100 mm (or similar diameter) critical orifice. They proceed through a lens[1] that focuses the aerosols into a tight beam of approximately one millimeter in diameter, using 6 apertures. Gas is removed later by differential pumping. As the aerosols exit the lens, they are accelerated in a supersonic expansion caused by the difference in pressure between the aerosol-sampling chamber and the aerodynamic particle-sizing chambers. This expansion gives different velocities to aerosols of different sizes.

After passing through the lens, the aerosols enter the particle-sizing chamber. A rotating chopper wheel, with two radial slits located 180^o apart, intercepts the focused particle beam. The chopper can be placed in any of three positions: completely blocking the beam so that no particles pass through (beam closed); not blocking the beam so that all particles pass through (beam open), and a chopping position that allows particles to pass through the radial slits only (beam chopped). The time of flight (TOF) between the chopper and the detector is the measurement of a particle's velocity; from this measurement the particle's aerodynamic diameter (D_{aero}) can be determined.

The particles passing through the flight chamber are directed onto a resistively heated surface. Upon collision with this heated surface, non-refractory particles flash vaporize under high-vacuum conditions. The vaporization process occurs directly inside an electron impact ionizer where the vaporized constituents are converted to positive ions, which can then be detected by the mass spectrometer.[2] The electron impact ionization process is a universal process; therefore, any species in the gas phase will be detectable. The AMS does not efficiently detect low-volatility materials such as black carbon, NaCl, crustal oxides and certain metals. However, lower volatility species adsorbed on such material can be detected.

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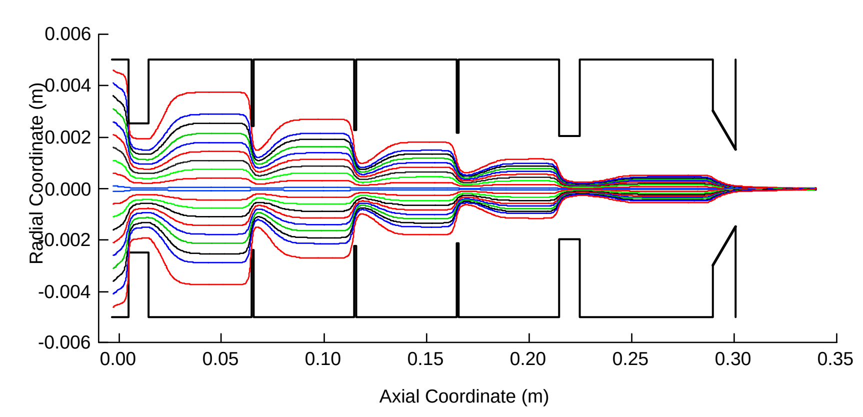
[1] Following the design in Liu *et al* [1995].
[2] "Non-refractory" refers to any material that rapidly vaporizes (on the time scale of <100 μs) at 600 ^oC temperature.



Calculated particle trajectories for 100 nm diameter unit density spheres through the aerodynamic lens.

Figure 2: Calculated Particle Trajectories

Particle Detection

Aerosol Mass Spectrometer signals originate within the electron impact ionizer. Typically, a few ions are produced from each million neutral molecules or atoms present in this ionization volume. This relatively low ionization efficiency requires high efficiency amplification to be detected by conventional means. The ions produced in the ionizer are focused into the quadrupole, which acts as a mass/charge filter. Ions of a given mass/charge (m/z) ratio emerge at the exit of the quadrupole filter and are directed to an electron multiplier for fast and high gain multiplication, of order 10^6.

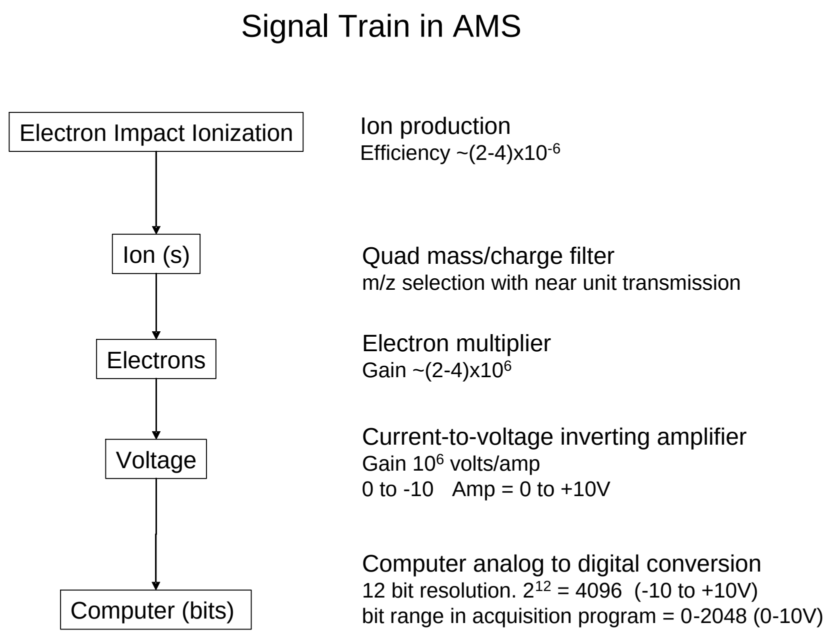


Figure 3: Signal Train in AMS

The electron flow emerging from the multiplier is directed into a current-to-voltage amplifier set to a gain of 10^{-6} amps per volt gain. Figure 3 shows the flow of signal from the ionizer to the data acquisition computer.

The signals within the data acquisition program are usually displayed as "bits," which relate directly to the analog-to-digital conversion process. The relationship between the bit-level signals and the actual ion signal is also explained in Figure 3.

Electron Multiplier

The electron multiplier is a fast-response (10 ns) high gain ($\leq 1 \times 10^7$) very low noise amplifier that can only operate under vacuum condition. A schematic representation of the type of multiplier used in the AMS is shown below. This is a discrete dynode style multiplier with 20 stages (or 18, depending on which model is used). Each stage is an active surface that ejects secondary electrons when an energetic particle strikes the dynode surface.

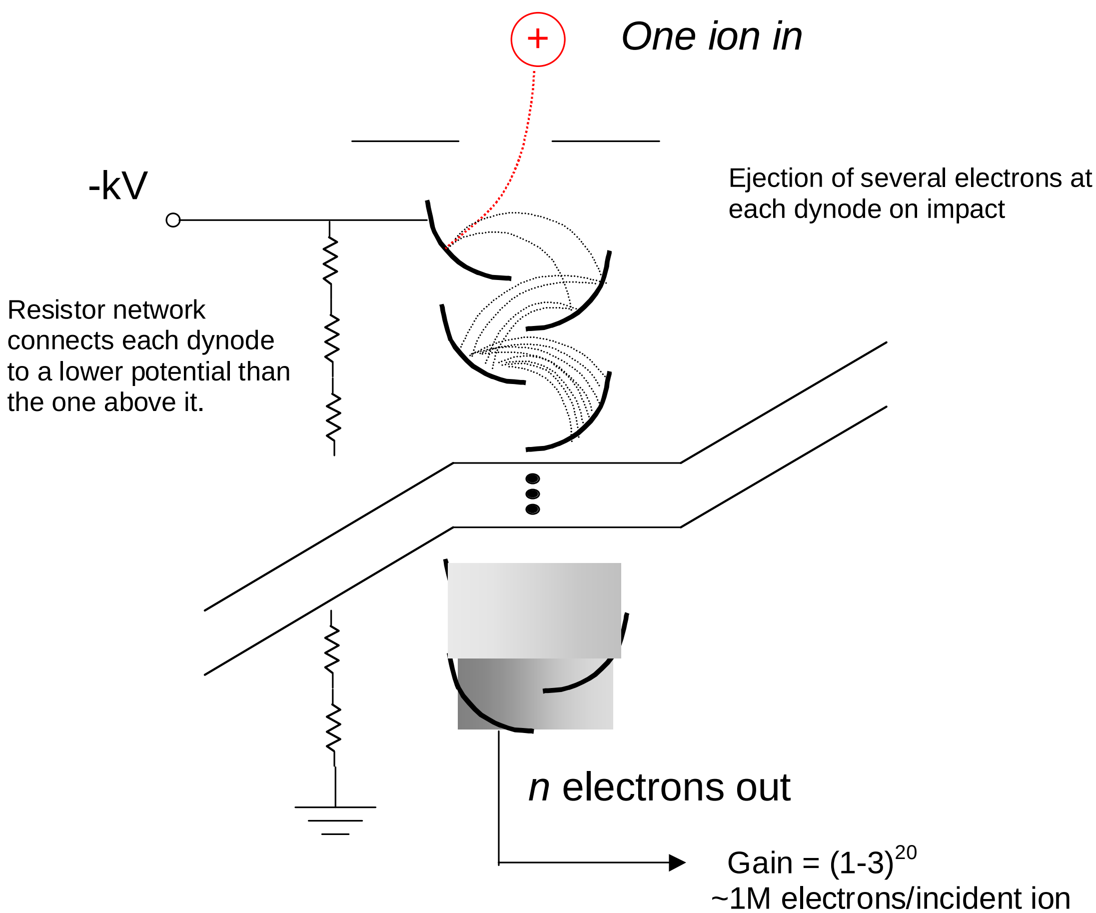


Figure 4: Multi-stage Discrete Dynode Multiplier

An electron multiplier can amplify positive or negative ions, photons, electrons and metastable atoms/molecules – any energetic particle. In the AMS, the multiplier amplifies positive ions that are filtered through the quadrupole and outputs many (of order 10^6) electrons. To detect positive ions, the device requires an applied negative voltage of -2 to -4 kV. A resistor network built into the multiplier provides the appropriate voltage drop at each dynode for successive amplification of electrons.

At the last dynode, a large number of electrons can be collected in a ~10 ns pulse. However, the time constant of the AMS electronics has been set to ~10 µs, so the fast electron pulse is broadened.

The useful life of the electron multiplier depends on the how many electrons are "pumped" out of it and the quality of the vacuum. For the AMS operating 24 hours a day during a field study, the multiplier life could be as short as several months. If used less it can last more than a year. The multiplier has only a finite amount of electrons that can be emitted. The faster they are removed, the shorter its lifetime.

Calibration of the electron multiplier is performed by integrating the average pulse area resulting from the detection of individual ions. To perform this calibration and avoid coincidence effects, the ion rate at the multiplier should be below ~2000 ions per second.

The acquisition program can be configured to look for single ion pulses that exceed the electronic noise level, using a threshold approach. This involves applying a threshold that is just above the electronic noise level by shutting off the ion production (turning off the filaments) and manually adjusting the threshold level. When the filaments are turned back on, any signal exceeding this threshold level can be counted and processed. Figure 5 illustrates this concept.

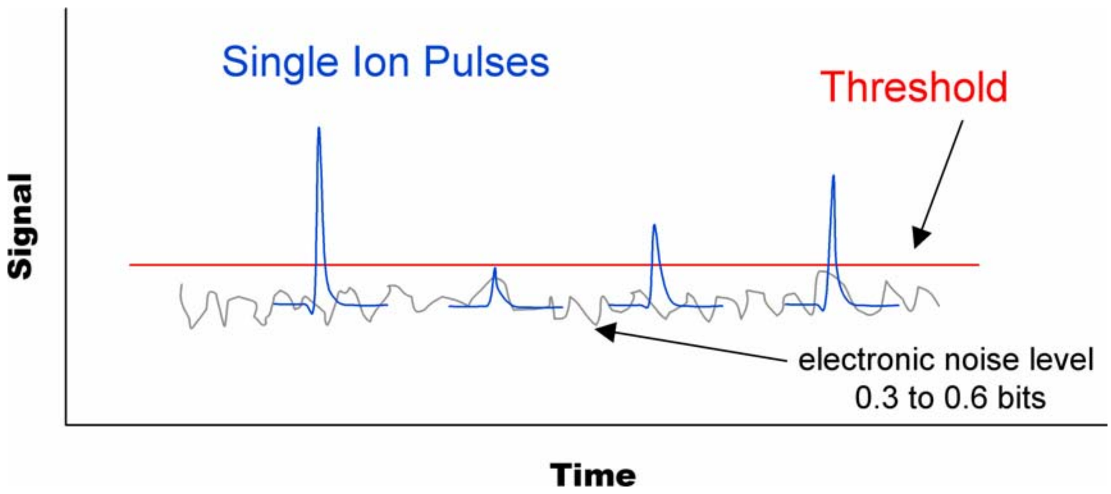


Figure 5: Setting Electron Multiplier Threshold to Detect Individual Ions

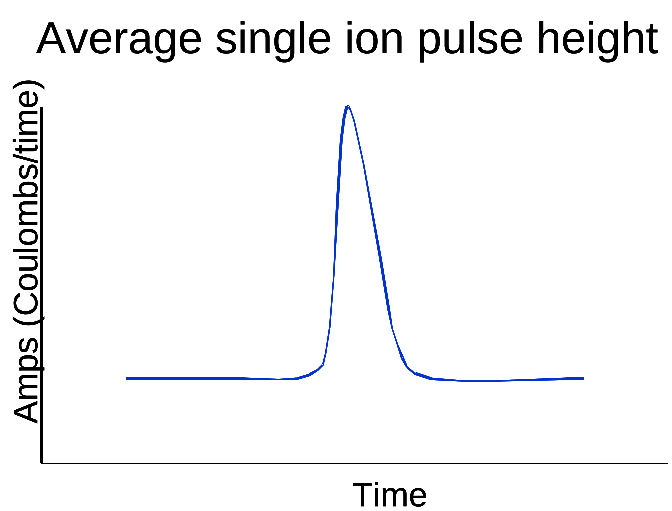
Some of the smaller ion pulses can be buried within the electronic noise. If there are a large number of pulses that do not exceed the electronic noise level, the resulting gain value will be overestimated, since the threshold approach will bias the measurement toward the larger pulses in the distribution. In practice, if the actual gain is $<1 \times 10^6$, this direct-pulse counting method will not provide an accurate gain value and will also be sensitive to the chosen threshold level value.

In some cases, depending on the quality of the multiplier, pulses can emerge even in the absence of an incident particle. These pulses, referred to as dark counts, will be observed when the ion production (filament) is off. Even if dark-count pulses are observed, the threshold should be set to just above the electronic noise level.[3]

[3] Make note of the count rate, since this may indicate when the multiplier is beginning to fail. The dark count rate will usually increase rapidly over several days and lead to a premature failure mode.

The data acquisition program can measure the area of single ion pulses, and from this measurement it can compute the gain of the electron multiplier. There is a large distribution of pulse heights that can be observed, due primarily to the fact that the ejection of secondary electrons at any given dynode probably ranges from 0 to 3. This variation, which scales to the 20^{th} power, results in a Poisson probability distribution of pulse heights. For this reason we determine the gain from the "average" pulse height.

The integrated area of the average pulse height has units of charge (amps x time) and, when divided by the Faraday Constant (1.6×10^{-19} Coulombs per charge), gives the number of electrons per incident ion, or the multiplier gain.



Area = Coulombs (charge)
Gain = Area/Faraday constant

Figure 6: Pulse Height vs. Time

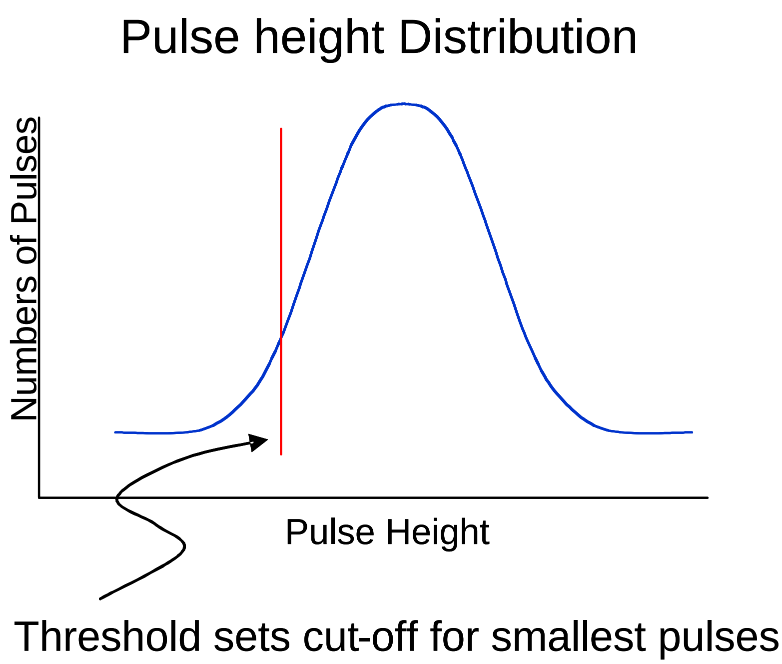


Figure 7: Pulse Height DistributionIonization Efficiency

Effective ionization efficiency (IE) of the AMS is determined by sampling particles of known size and composition. Typically, pure 300 nm NH_4NO_3 particles, pre-sized by a Differential Mobility Analyzer (DMA) are used. Individual particles' ion pulses are measured, using the same threshold procedure that was used to determine the multiplier gain. However, in this case the threshold level is set above the single ion level rather than just above the electronic noise level as was done in determining multiplier gain. Again, the data acquisition program determines the average area of the single particle pulses. This area is then divided by the average single-ion pulse area. The resulting ratio yields the number of ions per particle (IPP).

$$\frac{\text{Average single particle pulse}}{\text{Average single ion pulse}} = \text{Ions per particle (IPP)}$$

Ionization Efficiency = IPP/Molecules per Particle

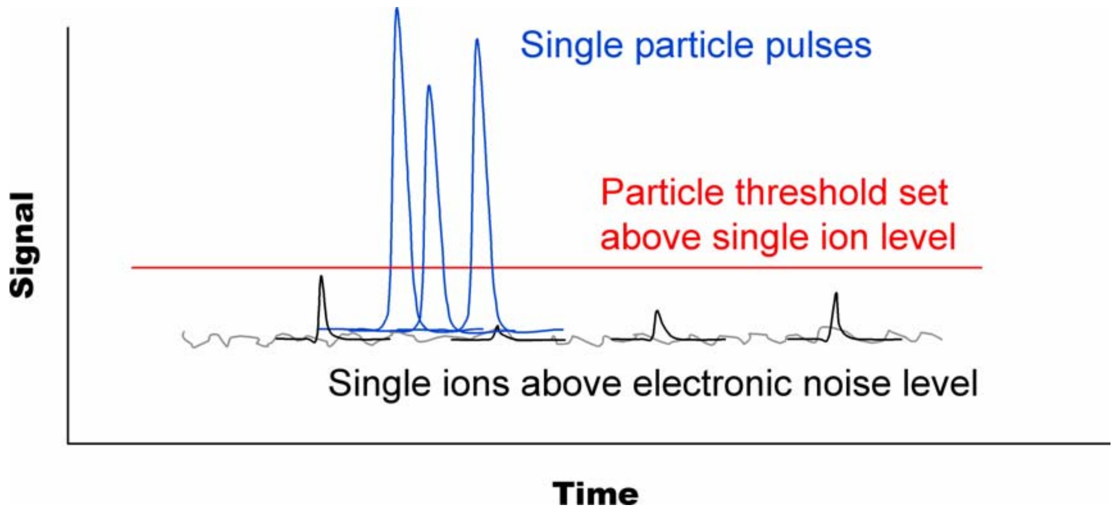


Figure 8: Setting Electron Multiplier Threshold to Detect Individual Particles

From this point, assuming spherical particles and an effective particle density, ionization efficiency can be calculated with knowledge of the number of molecules that were in the size-selected DMA particles. The mass per particle is given by

$$\rho_{eff}\left(\frac{4}{3}\pi r^3\right)$$

where ρ_{eff} is the particle effective density.

This discussion is focused on signals that cross a threshold and therefore give rise to a "counting" mode. Threshold-crossing signals can be either single ions or single particles depending on the set threshold level. The EM gain is based on the average area of the threshold-crossing single ion signal. The IE calculation is based on the average threshold-crossing signal area of individual particles of known size and composition (*i.e.*, known number of molecules). The level of the threshold is important: while calibration is based on single ion and particle events, the AMS data includes the total integrated signal from multiple particle events.

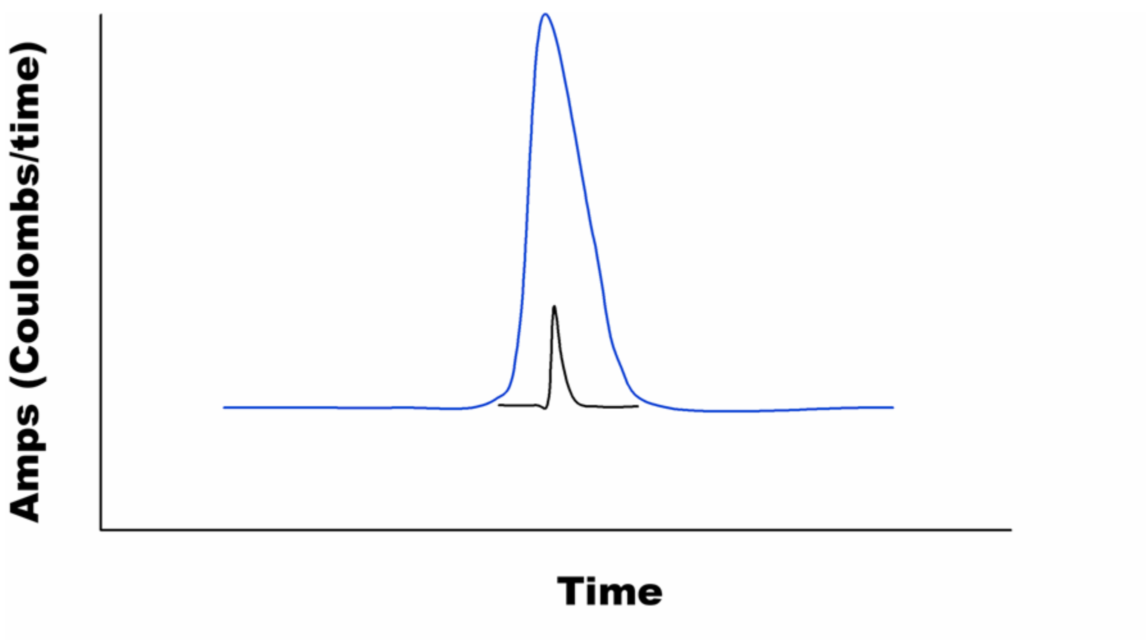


Figure 9: Relationship Between Single Ion and Single Particle Pulses

Volumetric Sampling (Flow) Rate

Knowledge of the precise rate of flow of gas into the AMS is required to normalize the measured particle mass to the volume of sample taken. This makes it possible to report particle mass loadings in conventional units of µg m^{-3} (s^{-1}), where µg comes from the mass spectrometer measurement and the m^{-3} (s) comes from the volume of air sampled.

The flow of sample into the AMS is fixed by a critical aperture (~100 µm diameter pinhole) mounted upstream of the aerodynamic lens. This critical aperture plays a fundamental role in fixing the lens inlet pressure that defines the focusing and transmission properties of a particular lens system. The pressure drop across this aperture is large enough that a choked-flow condition exists (ambient pressure to ~1 torr). Under these choked-flow conditions, the volumetric flow through this orifice is constant. A 0-10 torr pressure gauge is used to measure the pressure on the low-pressure side of the pinhole. In the range of operation, the pressure is proportioned to the mass flow rate. This flow rate is continually monitored by the data acquisition program.

There is a unique relationship between lens pressure and flow. In this system an absolute pressure gauge is used to monitor the lens inlet pressure, and volumetric flow is extracted from calibration. This relationship between pressure and flow is based on the Poiseuille Equation, relating the volume flow rate of a viscous gas through a laminar flow element. In this case, the laminar flow element is the aerodynamic lens tube, which has an entrance pressure of ~ 1 torr and an exit pressure of ~ 0.005 torr. The calibration is performed by replacing the critical aperture with a variable leak (a needle valve) and recording the lens inlet pressure for a series of different flow rates. For the calibration, the volumetric flow is measured using a soap-film flow meter or equivalent.

This approach uses a mass measurement device (an absolute pressure gauge) to report a volumetric flow. For this reason, it is important to record the ambient pressure and temperature at which the calibration was performed so that subsequent measurements at different ambient pressures and/or temperatures can be converted back to a volumetric flow.

Particle Size/Velocity

The AMS reports particle size based on a measurement of particle velocity. As a consequence of the weak supersonic expansion of gas into the vacuum at the lens exit, particles seeded in the gas are accelerated to different terminal velocities. Smaller/lighter particles are accelerated more efficiently than larger/heavier particles. Thus, there is a distribution of particle velocities that scale with the size/shape and mass. The AMS reports a vacuum aerodynamic diameter, since the particles expand into a free molecular flow regime. Particle velocities are calculated from a measurement of particle time-of-flight (TOF). Particle velocities range from ~ 50 - 200 m/s for particles in the size range of ~1000 to 40 nm.

A particle size calibration is performed by sampling particles of known size and measuring particle flight times. Polystyrene latex spheres (PSLs) and particles delivered from a differential mobility analyzer (DMA) are used as particle-size standards. A mechanical beam-chopping system is used to modulate the flight of particles to the detector. The detection is synchronized to the phase of the beam chopper. A key requirement for precise sizing by the AMS is to have rapid particle vaporization and detection relative to the particle flight time. In other words, the resolution of the size measurement can be limited by the rate of particle vaporization. For typical ambient particles, the particle vaporization time scale is 50-100 µs (FWHM), which is fast compared to the 2-4 ms particle flight time.

System Set-Up

AMS Hardware

Vacuum System

The AMS consists of five (5) internal chambers separated by apertures. There are five (5) turbo-molecular pumps and one optional hybrid turbo-drag pump in the vacuum system.

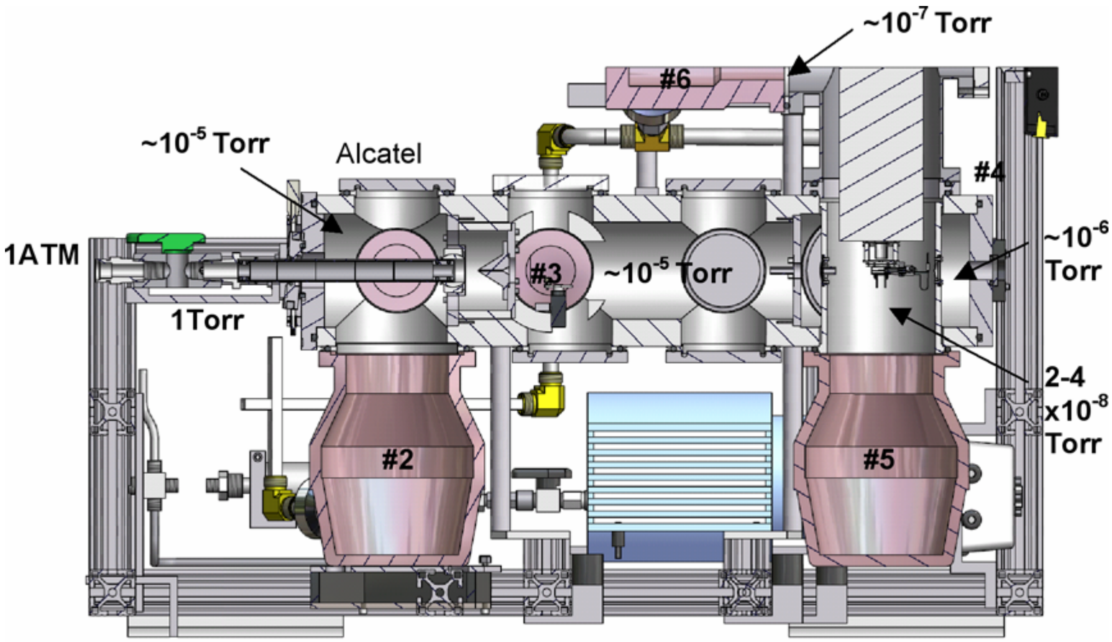


Figure 10: Cross-Section of Vacuum Chamber

Each of the pumps performs a specific pumping task, such as pumping a specific chamber. The vacuum system is completely oil-free and operates on 24 VDC. The combination of pumps has been designed to provide efficient differential pumping to separate atmospheric gases from aerosol particles. A cross-section of the vacuum chamber is shown in Figure 10.

The pumps on the inlet remove most of the gas but need reach only a modest vacuum. The pumps on the detector end have a very light gas load but must achieve the high vacuum needed for operating the mass spectrometer. The pressure in the various parts of the instrument ranges from tens of millitorr at the exit of the aerodynamic lens (the skimmer region) to ~10^{-5} torr in the TOF region to 10^{-8} torr in the ionizer chamber.

All turbo pumps are backed by a ~ 20 L/min adjustable pumping speed diaphragm pump/MDI. On this pump, high motor speeds provide a large throughput of gas but the ultimate vacuum is reduced. Operating at lower speeds provides a better ultimate vacuum but at reduced pumping speed. The optimum speed is set by monitoring the backing pressure (using the 10 torr Baratron gauge) as a function of the power consumed by the MD1 (this is done while the AMS is under load, sample valve open). The MD1 speed is adjusted to reach the minimum in the MD1 current and pressure relation.

The dimensions of the internal components and apertures in 255-xxx series vacuum chambers equipped with the 1/2" OD aerodynamic lens are listed in Figure 11. For the 215-xxx series chambers, subtract 102 mm from dimensions listed beyond the chopper wheel.

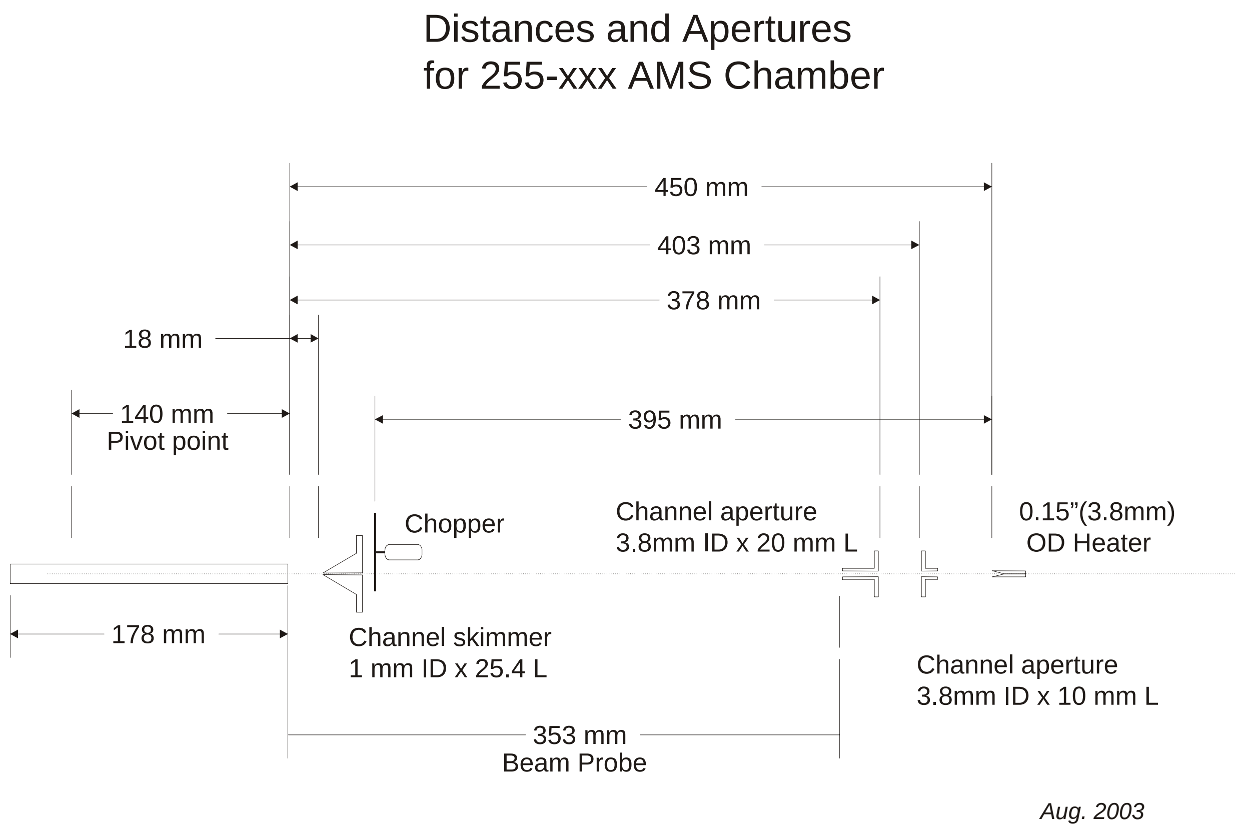


Figure 11: Chamber Distances and Apertures

Electronics

The AMS is controlled and operated by five (5) separate 19" rack-mounted components:

- Custom PC Computer/Data System (4U height) (described, page 23).
- Balzers QMG 422 quadrupole controller (4U height) (described page 24).
- AMS Power Supply - PS (3U height) (described, page 24).
- Turbo Pump Control Box - TPC (2U height) (described, page 27).
- Electronics Box - EB (2U height) (described, page 31).

A description of each of these components follows.

Computer/Data System

Power Requirement: Universal 100-240 VAC, 50/60 Hz, 400 W max (all post AMS-008 serial numbers). Dimensions: 19" wide x 22" deep x 7" high (4U).[4] The PC-based data acquisition system is built with standard currently available technology/parts. The computer contains two National Instruments (NI) data boards, a PCI 6110E 5 MHz board and a PCI 6024E general-purpose board. The data acquisition system software is written in Visual Basic and interfaces with the NI boards. The 5 MHz board performs the core acquisition, measuring the mass spectrometer ion signal and chopper signal, and the general-purpose board is used to monitor parameters such as flow and temperature.

The computer system has two RS 232 serial ports, one of which is dedicated to interfacing with the Balzer QMG422 quadrupole controller. The second serial port can be used to interface with any one of the three TSI CPCs (3010, 3022A or 3025A). The AMS acquisition software is set up to read, display and log the CPC data from these TSI instruments as well as data from the AMS.

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[4] Note that computer systems vary and the particular power requirements may be different from that listed above. To be sure, check power specification on the power supply in your specific computer.

Balzers QMG 422 Quadrupole Controller

Power Requirement: Universal 90-265 VAC, 47-63 Hz, 300 W max. Dimensions: 19" wide x 18" deep x 7" high (4U).

The QMG422 is the quadrupole controller. It is supplied with the following optional components 1: RS232 interface control, 2: IS420 ion source power supply and 3: front panel user interface. For a description of the components refer to the Balzers manual.

AMS Power Supply

The AMS Power Supply Box (PSB) (See Figure 12, page 26) contains two switching AC/DC supplies; both are universal input, ~ 85% efficiency. Both units deliver 24 VDC: one powers the Electronics Box (EB) (fused at 2A) and the other powers the diaphragm pump (fused at 5A) and the Turbo Pump Control (TPC) (fused at 15A). The specifications of each switching supply are the following:

Electronics Box supply

60-watt single output 24VDC **2.5 amp** rating.

Input voltage: 85-264 VAC

Frequency: 47-440 Hz

Diaphragm Pump and Turbo Pump Control supply

500-watt single out put 24VDC **20-amp** rating

Input voltage: 85-264 VAC

Frequency: 47-440 Hz

The dimensions of the Power Supply Box are 19" wide x 18" deep x 5.25" high (3U).

Power Supply Box Front Panel Controls

(See Figure 12)

SW1 - Toggle switch in "up" position turns on AC power to 500W AC/DC supply that outputs 24 VDC to start the diaphragm pump. The **lower** red LED should turn on indicating that 24V is present at the rear connector labeled Diaphragm Pump. If the LED fails to light, check Fuse F3 in rear (5A slow-blow fuse).

SW2 - Toggle switch in "up" position turns on 24 VDC to the Turbo Pump Control connected to the 500W AC/DC supply. The **middle** red LED should turn on indicating that 24V is present at the rear connector labeled Turbo Pump Box. If the LED fails to light, check Fuse F2 in rear (15A slow-blow fuse).[5]

SW3 - Toggle switch in "up" position turns on AC power to the 60W AC/DC power supply that outputs 24 VDC to the ECB. The **top** red LED should turn on indicating that 24V is present at the rear connector labeled Electronics. If the LED fails to light, check Fuse F1 in rear (2A fast-blow fuse).

DP Speed – Diaphragm pump speed control.

The analog meters next to each switch measure DC current at 24V to each of the three connectors. The Power Supply Box uses a standard IEC-320 connector for AC power input. This connector contains a 5 x 20 mm 2.5A fast-blow fuse to protect both AC/DC supplies. If no LEDs turn on, check this fuse.

[5] Note that this switch is inactive if SW1 is in the "off" down position. The turbo pumps cannot be turned on unless the diaphragm pump is on.

Figure 12: AMS Power Supply, front and rear views

Turbo Pump Control (TPC) Box

(See Figure 13)

The Turbo Pump Control controls the turbo pumps. It is powered by 24 VDC from the 500 W supply in the AMS Power Supply Box. This Turbo Pump Control rack box houses up to six separate pump controllers. The turbo pump rotational speed and current consumption can be viewed on the LCD, and pump error status information is displayed via red LED lamps.

For the Varian V70 LP pumps, the LED lamp will flash at different rates to indicate specific errors (*i.e.* repeated double flash indicates "pump not connected" – see Varian manual 969-9518 for a complete listing of error codes). For systems with the Varian TV 301 Navigator pump on the inlet end, the error status LED simply turns on and stays illuminated (no flashing) when there is an error, providing only general error information. Similarly, for systems that include the Alcatel ATH31+, the red (error) LED will stay lighted when there is an error, but provides no information on the nature of the error. The Alcatel pump also has a yellow LED that indicates the pump is not running up to speed and a green LED that indicates pump is at normal operating speed.

More detailed error and status information can be obtained for the front end TV301 pump by running the Varian Navigator Software (pre-installed on the computer). This is done using a straight through serial cable (DB9 M-F) from one of the computer's COM ports to the 9-pin DB connector on the rear of the Turbo Pump Control box.[6]

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[6] Note that the LCD for pump speed and current is very informative for determining vacuum status. For systems that are equipped with the Varian VT301 and Alcatel ATH31+, all pumps should operate at 100% full speed with nominal gas load of 1.5 cc/s (100 µm diameter pinhole). The user should note and monitor "typical" operating currents with and without gas load. Systematic deviation (*i.e.* lower rotational speed and higher current draw) is an indication of possible vacuum leaks and/or aging (increased friction) of the pump bearings.

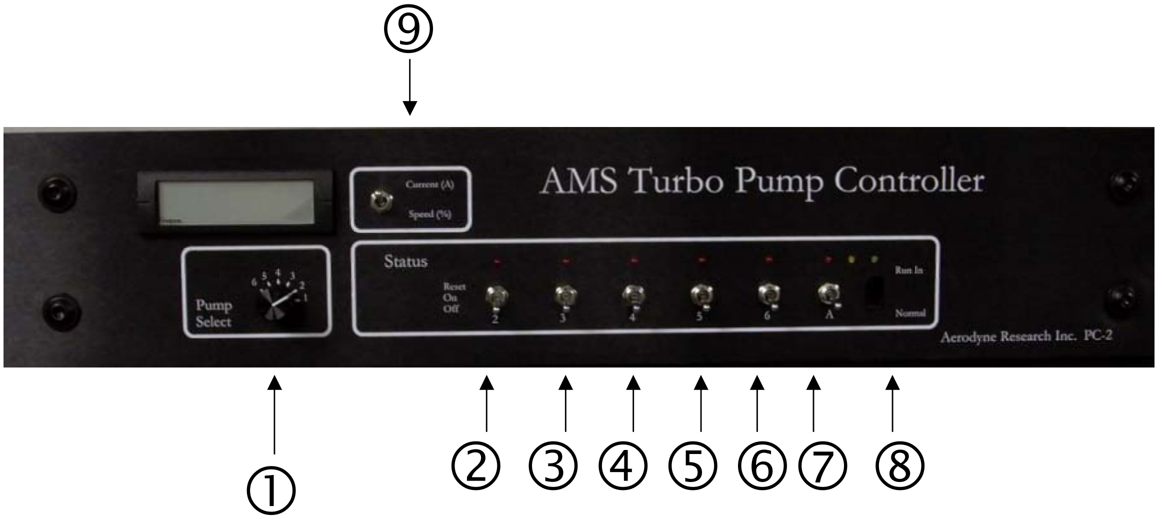

Figure 13: Turbo Pump Control, front panel controls

Turbo Pump Control Box Front Panel Controls

1. Six-position rotary selector switch displays speed and current for any of the six turbo pumps.
2. Toggle switch activates V301 pump on front end of AMS.
3. (3 - 6) Toggle switches 3 through 6 activate all V70 turbo pumps and the V301 pump on the detector end of the AMS. For these switches off = down; on = middle; reset = up/momentary hold. Pushing the switch up and holding it for several seconds resets the microprocessor in the controller following a fault condition (i.e. flashing red LED).

7 Toggle switch "A" turns on optional Alcatel ATH31+ pump.

8. Slide switch activates the "Run-In" procedure for the Alcatel pump. Down is off, up is on.

The run-in procedure is designed to redistribute grease in the pump bearings. This procedure is recommended if the pump is idle for more than 3 months. The procedure is initiated from a fully powered-down state (all turbo pumps off) with both toggle switch **7 (A)** and slide switch **8** in the off (down) position. Turn on power to the Turbo Pump Control box. Move slide switch **8** to the on (up) position. Start the pump by putting toggle switch **7 (A)** in the up position. At this point, the pump will begin to spin (yellow LED on to indicate pump starting). The pump will reach a reduced speed (~ 40%) and "hold" there for ~ 2 minutes (green LED should be on). After ~ 2 more minutes, the pump will turn off automatically. When the pump has stopped spinning, it will repeat this cycle two more times, reaching progressively faster "hold" speeds (~ 60%, then ~ 85%). After progressing through these three speed levels, the controller will repeat the process in an endless loop until the controller is powered down.

If the pump runs smoothly after one run-in cycle (no squealing from bearings) the controller should be powered down and both switches **7(A)** and **8** should be returned to their off (down) positions. At this point the pump has been "run-in."

9. Toggle switch selects display output: either **pump current in amperes** or **pump speed as percent of full speed**.

IMPORTANT: The TV301 pump (or V250 on pre 255-008 SNs) on the detector end is driven by a V70 controller that has been REPROGRAMMED to drive this larger pump. In the event that controller #5 fails or the user is exchanging V70 controllers DO NOT exchange controller #5 for the detector TV301 (pump 5). Contact ARI for assistance.

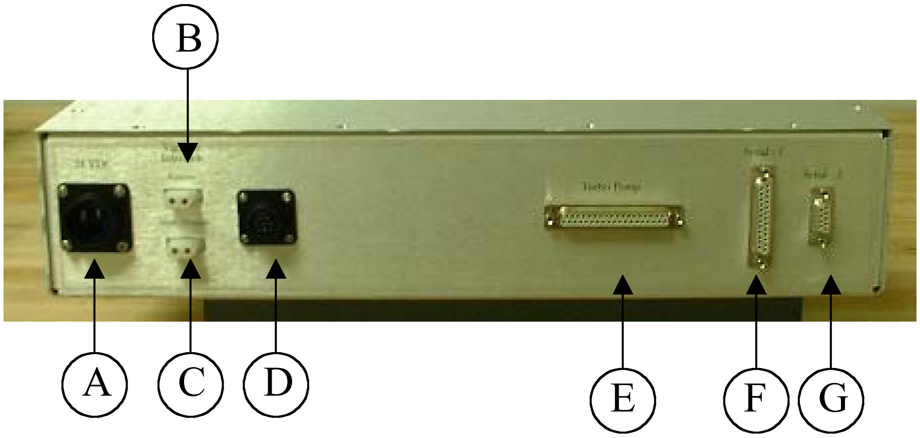

Figure 14: Turbo Pump Control, rear panel connectors

Turbo Pump Control Box Rear Panel Connectors

A. Power input for Turbo Pump Control box (24V). Pin 1 is $^{+}$24V. Pin 2 is 0V.
B. Vacuum interlock for filaments in ionizer, connects to Balzers QMG422 DB9 "ctrl" connector (relay closes when pump #5 reaches full speed).
C. Vacuum interlock for multiplier/vaporizer/vaporizer bias, connects to Electronics Box (EB) (relay closes when pump #6 reaches full speed).
D. Connector for optional Alcatel pump.
E. DB37 (F) connector to mating connector on turbo cable junction box on AMS frame.
F. DB25 (F) - future use, for serial control of all pumps.
G. DB9 (F) - dedicated serial port for remote control of V301 on inlet end of AMS. (Varian Navigator software).

Electronics Box (EB)

The Electronics Box is powered by 24 VDC and controls the beam chopper, multiplier high voltage (-4 KV), an optional -6 KV voltage (future conversion dynode), vaporizer (heater) bias and vaporizer power. Each of these features/voltages can be computer-controlled via an externally supplied 0-10 VDC input, <1 mA or manually controlled by recessed trim pots located on the front panel. The typical operating mode is to have only the vaporizer bias and multiplier voltage computer-controlled and all other features manually controlled.

The multiplier voltage (and the optional high voltage supply), vaporizer bias voltage and vaporizer power are interlocked to the status of turbo pump #6 (the V70 mounted on the quadrupole chamber). If this pump falls below ~ 95% full speed (as displayed on the Turbo Pump Control box LCD) these voltages will be disabled. This will be evident if the vaporizer V/A display stops working. The interlock system is designed to protect the multiplier and vaporizer in the event of a vacuum (pump) failure. The interlock operates by closure of a relay on the turbo pump control board (when the pump reaches full speed), which completes the circuit in the Electronics Box.

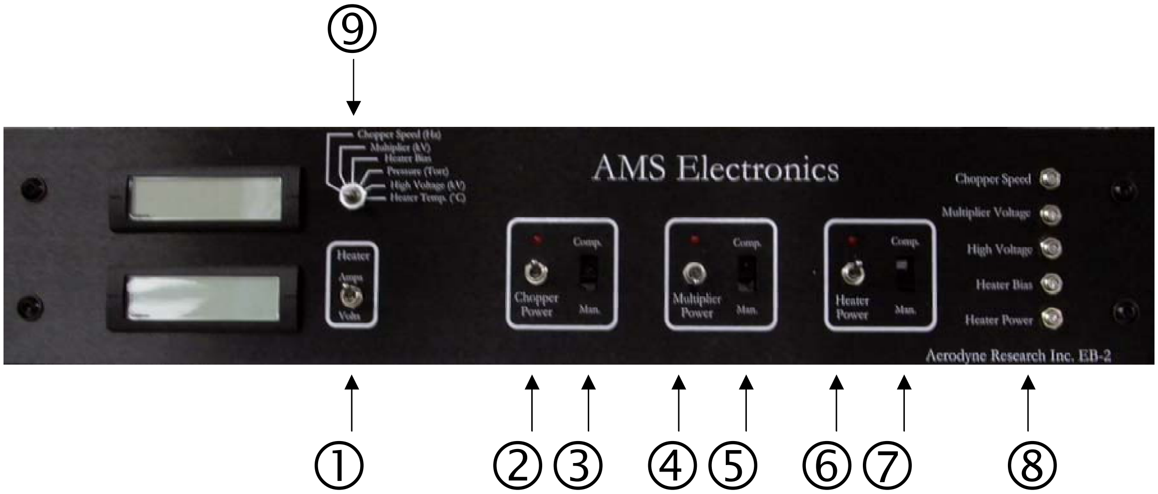

Figure 15: Electronics Box, front panel controls

Electronics Box Front Panel Controls

1. Toggle switch selects display of current or voltage delivered to the vaporizer (heater).
2. Toggle switch turns on chopper motor, on = up; red LED on. The chopper servo is still active if this switch is off.
3. Slide switch selects computer or manual control of chopper speed. Normally manual control is used.
4. Toggle switch turns on multiplier (and optional -6 kV high voltage) supply. Status LED lights when on. Vacuum interlocked to speed of pump #6.
5. Slide switch selects computer or manual control of multiplier voltage only. Normally computer control mode is used. Optional high voltage supply is only controlled manually and is independent of computer/manual switch position.
6. Toggle switch turns on vaporizer (heater) power, status LED lights when on.

7. Slide switch selects computer or manual control of vaporizer bias. This is normally in computer control mode. Vaporizer power is manually controlled via trim pot even if this switch is in the "computer" position. A jumper connection inside the box may be removed to allow for future computer control of vaporizer power.
8. Trim pots for manual control of labeled voltages.
9. Six-position rotary selector switch for display of various parameters.

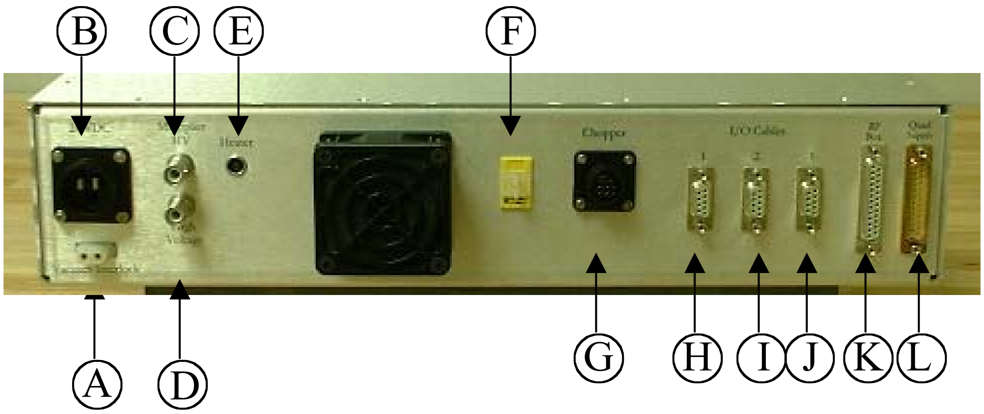

Figure 16: Electronics Box, rear panel controls

Electronics Box Rear Panel Connectors

A. Vacuum interlock receptacle to mating connector on Turbo Pump Control box.

B. 24VDC input power from Power Supply Box. Pin 1 is $^{+}$24 VDC, Pin 2 is 0V

C. Multiplier voltage out (0 to -4KV) to SHV connector on Balzers Quad flange labeled HV-.

D. Optional high voltage output.

E. Vaporizer power/vaporizer bias connector to mating connector on quadrupole. (CAUTION HIGH VOLTAGE: these leads are floating at the vaporizer bias potential).

F. Thermocouple input for vaporizer temperature monitor (CAUTION HIGH VOLTAGE: these leads are floating at the vaporizer bias potential).

G. Chopper cable, connection to chopper flange.

H. DB9 (F) I/O-1 connector to turbo junction box on AMS frame (See Figure 17).

Pin description for Input/Output (I/O) connectors on rear of AMS Electronics Box

Nov. 18, 2004

I/O 1, 9-pin D-sub to turbo pump junction box on AMS frame

Pin	Direction	Function		Description	
1	out	GND		---	
2	in	Signal return		10 Torr Baratron (p1 on DB9 of gauge)	
3	in	Signal return		1000 Baratron (or Aschroft dP gauge)	
4	in	Signal return		ion gauge/not used	
5	in	GND		Baratron gauge power ground (p9 on DB9 of gauge)	
6	out	+24VDC		Baratron gauge power (p4 on DB9 of gauge)	
7	out	+15V		preamp power (p3 on DB9 of PMT-5)	
8	out	-15V		preamp power (p8 on DB9 of PMT-5)	
9	out	GND		preamp power ground (p7 on DB9 of PMT-5)	

I/O 2, 9 pin D-sub to data system

Pin	Direction	Function	DB9-5BNC Cable Color	Description	
1	out		Red, Ch3, SB	pin 3 of I/O 1, 1000 torr Baratron sig(or dP gauge)	
2	out	vaporizer temp	Green, Ch1, SB	heater temperature, 0-10V = 0-1300C	
3	In	chopper Servo drive	Blue, CTR0, FB	50Hz 5V square wave, variable pulse width 1-2ms	
4	out	chooper output	Gray, Ch1, FB	~5V pulse at chopper frequency	
5	out	10 torr Baratron	Black, Ch3, SB	pin 4 of I/O 1, 0-10V	
6	GND				
7	GND				
8	GND				
9	GND				

I/O 3, 9 pin D-sub to data system

Pin	Direction	Function	DB9-5BNC Cable Color	Description	
1	in	heater power control	Red, not connected	0-10V input ~ 0-2 amps to heater	
2	in	heater bias	Green, DAC0, SB	0-10V input 0-200V bias to heater	
3	in	multiplier control	Blue, DAC1, SB	0-10V input, 0 - 4kV (neg) to SHV connector	
4	in	Quad resolution control	Black/gray, DAC0, FB	0-10V to pin 5 of 25 pin D-sub (F) connector	
5	in	Quad mass command	Gray/black, DAC1, FB	0-10V to pin 8 of 25 pin D-sub (F) connector	
6	GND				
7	GND				
8	GND				
9	GND				

Figure 17: Pin Description for I/O Connectors

SB= Slow board (NI 6024E)
FB= Fast board (NI 6110E)

I. DB9 (F) I/O-2 connector to National Instruments Board (See Figure 17).

J. DB9 (F) I/O-3 connector to National Instruments Board (See Figure 17).

K. DB25 (F) connector to Balzers QMH 410-5 RF supply box.

L. DB25 (M) to QMH 422 Balzers quadrupole controller supply.

Operational Procedures

Start-Up

This procedure assumes a starting condition where the AMS vacuum system is fully powered down, *i.e.* no turbo pumps are running, the MD1 diaphragm pump is off and both the inlet valve and the MD1 valve are closed. All power is off, but all units are plugged into AC power.

1. Turn on the switch for the AMS Electronics on the AMS Power Supply Box (SW 3 in Figure 12, page 7). Set the 6-position selector switch on the Electronics Box (EB) to "Pressure" (torr) position (⑨ in Figure 15, page 32). Verify that the 3-way Whitey valve connecting the 10 torr Baratron gauge is in the "inlet" position. Obtain a reading of the vacuum chamber pressure on the liquid crystal display on the Electronics Box. For the 10 torr range gauge, a display of ~12 indicates that the gauge is over range.
2. Turn on the diaphragm pump at the AMS Power Supply Box (SW 1 in Figure 12, page 26) Verify that the pump is spinning. Open the MD1 valve to start rough pumping out the chamber. The DC current to the MD1 pump should decrease as the pressure decreases. Typical MD1 operating current is 1-1.5 amps. Monitor the chamber pressure until it is <10 torr. If the base pressure is not below 10 torr, it is likely that there is a small leak, which may or may not be significant.
3. After the pressure drops below 10 torr, turn on the turbo pump power at the AMS Power Supply Box (SW 2 in Figure 12). Turn on the Alcatel pump (Turbo Pump Control Panel TCP, Figure 13) and let it reach 100% speed. Next, turn on pump #2 (V301) and let it reach full speed. Note that this pump will reach >7 amps at full power load. Next, turn on all other pumps and verify that they reach full speed. Normally, Pump #5 (V301) will be the last to reach 100% speed.

4. After all pumps are at full speed, turn on the Balzer Quadrupole power supply and the vaporizer power (Electronics Box ⑥, Figure 15).
5. Let the system pump for at least 1 hour. Record and compare pump speeds and currents.
6. After pumping for ~1 hour, start the AMS program and enter the MS mode of operation. Press "Shift B" and turn on the ionizer filament at the lowest setting (0.01 mA). Verify that the filament turns on by looking through the window in back. At this point – to make certain all systems are working – it is useful to turn on the multiplier switch briefly and verify that a mass spectrum is observed.
7. Over the course of ~ 2 hours, step the filament current up from 0.01, 0.02, 0.05, 0.1, 0.25 mA in 20-30 minute intervals. You may choose another, similar sequence.
8. Once an emission current of 0.25 mA is reached, the system should, ideally, pump overnight before use. A calibration done before ~ 24 hours of pumping will likely be different than one performed after several days. A gradual increase in performance, an increase in Ionization Efficiency (IE) and Air Beam (AB) values, is usually observed over a week-long period.

Shut-Down

Controlled Shut-Down Procedure

(Assumes instrument is fully operational)

- Use software to turn off electron-emitting filaments and exit AMS acquisition program.
- Close inlet valve.
- Turn off power to multiplier, chopper and vaporizer. Turn off main DC power to Electronics Box.
- Turn off all turbo pumps; keep MD1 backing pump on. Main DC power to

Turbo Pump Box can be shut down. Ideally, the turbo pumps should be allowed to spin down until they stop (~30 min for a leak-free system). This avoids putting additional stress on the pump bearings.

- If the system is being prepared for shipping, the system should be shipped under vacuum. After the turbo pumps have fully stopped, close the isolation valve at the MD1 diaphragm pump. Shut down computer and any other AC power component.
- If the system is being shut down for venting and must be vented before the turbo pumps have stopped spinning, venting should only be done via the vent port on pump #5 (V301). Open this vent port for just a fraction of a second and let the pumps reach a reduced speed (~ 1-2 minutes) before fully venting to atmosphere.

Emergency Shut-Down Procedure

(Assumes instrument is fully operational)

- Turn off AC power to QMG422 quadrupole power supply. This will shut off filaments and RF voltage.
- Close inlet valve.
- Close the isolation valve at the MD1 diaphragm pump.
- Turn off main DC power to Electronics Box (Electronics Box SW3 on AMS Power Supply).
- Turn off main DC power to Turbo Pump Box (Turbo Pump Control SW2 on AMS Power Supply).

Calibrations

Checks and calibrations must be performed to keep the AMS operating in a known and optimal condition. The tables on pages 40 and 41 list the checks and calibrations that must be performed during instrument set-up. The instructions for performing these operations are included in this manual on the pages listed in the table. The table also indicates how often these calibrations and checks must be done before and during each new experiment and how often these operations should be performed. Many of these calibrations/checks produce a screen display. The calibrations and checks are often used as diagnostic aids and provide a history of instrument performance. The screen displays should be copied on a regular basis and pasted into an electronic file or kept in a paper file (a logbook, PowerPoint file or spreadsheet).

There are two categories of system calibrations:

1. Those that impact ion signal intensities. These include: Quadrupole Mass and Resolution Calibration, Electron Multiplier Calibration, and Ionization Efficiency Calibration.
2. Calibrations that do not have a direct impact on sensitivity but which are required for accurate reporting of aerosol mass values. These include: Flow Rate Calibration, Aerodynamic Lens Alignment, Particle Sizing Calibration and Servo Motion/Position Calibration.

The order or sequence of calibrations will depend on the state of the system. For a system that appears to be operating properly, the typical sequence would be as follows: Quadrupole Mass and Resolution Calibration, Electron Multiplier Calibration and Ionization Efficiency Calibration. On completion of these checks/calibrations, the system is at the reference calibration state.

SYSTEM CALIBRATIONS THAT AFFECT SIGNAL INTENSITIES			
Checks and Calibrations	Output/Use	After initial set-up, this procedure should be performed…	Comment/Cautions
ELECTRON MULTIPLIER CALIBRATION See page 42	Determines EM gain	Every 3-4 days when AB decreases to 70%; or when beginning new experiment	May be done more often, but may cause wear on multiplier
TUNE MASS SPECTROMETER	Maximizes MS signal	After moving or major change in instrument	**Perform only when necessary**; can change ionization efficiency; do NOT do when an experiment is in progress
QUADRUPOLE MASS CALIBRATION See page 51	Verifies m/zs	Automatically done during AutoSave mode; otherwise daily, more frequently if temperatures vary widely; temperature dependent	Calibrate for low MS and/or TOF AB; very important for TOF measurements
QUADRUPOLE RESOLUTION CALIBRATION See page 50	Determines optimum peak shape	After moving, or every 6 months	Repeat MS tuning if resolution changed
IONIZATION EFFICIENCY CALIBRATION See page 54	Determines ionization efficiency	Every 3-4 days, unless the instrument is moved	

SYSTEM CALIBRATIONS THAT AFFECT ACCURATE REPORTING OF AEROSOL MASS VALUES			
Checks and Calibrations, continued	Output/Use	After initial set-up, this procedure should be performed…	Comment/Cautions
FLOW RATE CALIBRATION See page 58	Determines the volume of air sampled per time and sets the T and P conditions for all subsequent measurements	Prior to new experiments, when T and P conditions change dramatically (*e.g.* sea-level vs. mountain site)	Should be ~1.44 cm^3/s with 100 µm pinhole
AERODYNAMIC LENS ALIGNMENT See page 61	Aligns particle beam with vaporizer	After moving or inlet change	Check for unexpectedly low IPP values; frequent checks are important for mobile AMS application
PARTICLE SIZING CALIBRATION See page 63	Determines TOF/ Aerodynamic diameter relationship	After moving, or every 6 months	Especially important when operating the instrument at a different ambient pressure (*e.g.* at high altitude)
CHECK SERVO MOTION/POSITION See page 67	Sets the open/closed/ chop positions of the chopper	After physically moving the instrument; after chopper/servo replacement or maintenance	If ratio of TOF/MS AB deviates from unity

Electron Multiplier Calibration

Electron multiplier calibration determines the gain of the multiplier and tracks and compensates for its decrease over time. The gain is calculated by comparing the observed area of a single ion signal (in contrast to the burst of ions produced by a particle) with the expected area of the single ion signal. The area of the single ion signal is measured in bitsteps (voltage multiplied by time, where time is recorded in 10 μs time steps).

See Figure 8 and

Figure 9, pages 16 and 17.

Inside the multiplier, every ion that impacts the first stage produces 1-3 or more electrons; this current is increased by the value of the gain. This produces the current (or charge) measured at the output of the multiplier.

$$V = IR = \frac{qR}{t}$$

$$Vt(bitsteps) = qR = G\left(q/e^{-}\right)R$$

where R is the preamplifier input resistance ($1x10^{-6}$ Ω), I is the current, q is the charge, q/e^{-} is the charge per electron ($1.6x10^{-19}$ Coul), t is the time, and G is the gain of the multiplier.

Since q/e^{-} and R are not changing over time, the area of the single ion signal (V• t) depends only on the gain of the multiplier.

Except for the first calibration of a new multiplier, the automated calibration procedure described on page 43 can be used to calculate the gain. As the multiplier ages, the decay in gain can be corrected by changing the value of the scaling factor in the gain equation.

The multiplier calibration process determines the scaling factor necessary to represent the actual gain of the multiplier as it decays.[7]

AUTOMATED ELECTRON MULTIPLIER CALIBRATION PROCEDURE:

- In Mass Spectrum mode, toggle the chopper (shift T) and record the air beam signal (usually N_2, m/z 28) for reference.
- Choose "Calibrate Electron Multiplier" from the main menu and wait while the single particle thresholds are set automatically (screen message: Setting SP Thresholds). The software automatically uses m/z 46 for this procedure.
- After the voltage is finished scanning (the multiplier voltage is scanned +/- 175 V around the initial setting), note values:
    - **kV chosen** (kV) – new multiplier voltage value.
    - **kV change** (kV) – difference between initial and new voltage setting.
    - **Gain chosen** *(*usually around 3 x 10^6*)* – new gain, determined by the multiplier voltage and the scaling factor.
    - **G used change** – fractional gain change.
    - **Calibration Change** (%) – represents the degree to which the multiplier gain has decreased or increased since the last calibration (ratio of the ion pulse area to the green line in the lower panel).
    - **Final Scaling Gain** – gain scaling factor; decreases over the lifetime of the multiplier (*i.e.*, measures "age" of multiplier).
    - Display curves

---

[7] The automated multiplier gain calibration procedure in the AMS software (version 3.4.3 and higher) determines the correct multiplier voltage and gain by measuring single ion pulses and plotting multiplier gain vs. voltage.

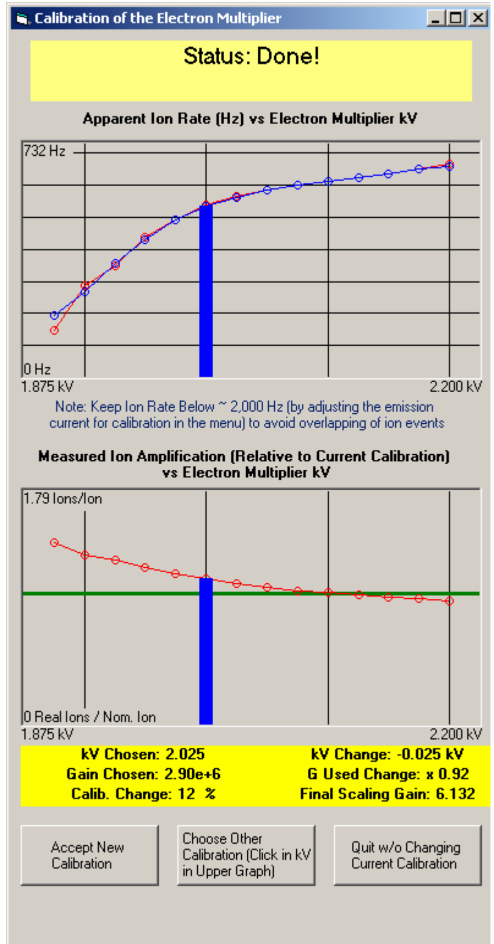

Figure 18: Screens: Electron Multiplier Calibration

- The upper panel shows the background count rate for the mass chosen for calibration (the count rate should be ~ 0.2-1 kHz).
- If the rate is high or low, adjust the emission current down or up: Choose "Emission Current for Calibration" in the Mass Calibration tab in the parameter menu.

- For proper multiplier gain calibration, the filament is set to the low emission current value that is set in the multiplier tab of the menu, and the multiplier voltage at which the calibration is to be performed is selected.[8]
- Upper curve: the ion count rate should not increase with multiplier voltage after the gain becomes large enough that all ions give pulse heights above the electronic threshold.
    - Once the curve plateaus, the increase in signal with gain is maximized, and a further increase in voltage will not increase the signal but will shorten the lifetime of the multiplier.
    - The blue bar attempts to choose the optimal multiplier voltage at which to measure the gain based on the plateau: this can be changed by selecting "Choose Other Calibration" at the bottom of the screen and then clicking at the optimal multiplier voltage/gain point on the upper curve. The results of the gain calibration at that voltage are displayed in the yellow section of the window.[9]
- Lower curve: shows the single pulse area normalized to the multiplier gain curve. The gain curve is set by the gain parameters (coefficients) in the Multiplier & Chopper tab of the parameter menu. After the gain is properly calibrated at a given voltage, then the value of the lower curve at that voltage should be equal to 1 measured ion/1 calculated ion.
- Select "Accept New Calibration"

---

8 This selection is made so that calibrations are not performed in regions of the gain curve where small changes in voltage result in large changes in gain. In order to identify the appropriate voltage, a series of single ion measurements are performed as a function of multiplier voltage and the voltage at which the single ion count rate begins to plateau is selected.

9 If the top curve does not plateau nicely, then the calibration should be done at a voltage that results in a calibrated gain of 3×10^6.

- Depending on the plateau shape, **repeat the procedure.** The blue bar should not be close to the right axis.
- Quit (press Q) and return to Mass Spectrum mode.
- Toggle chopper (shift T) and note the air beam signal (AB).
- AB signal should change according to Calibration Change measured above (*e.g.*, for a change of -60%, the air beam count rate should be 2.5 times higher than before setting new gain.
- Choose the mass for multiplier calibration (*e.g.*, m/z 42 or 46) in the m/z selection window (press F6 from TOF window). Make sure that the sliding window is set to 2 instead of 4.
- Set the chopper to "Beam closed," or close the inlet so that only background ions are used for the calibration.
- Turn off the filament.
- Set threshold manually for selected m/z (right click on threshold and use 🡅 and 🡇 to raise and lower the threshold).
- Turn filament on low setting (~ 0.05 mA) and check the number of ions; there should be 200-1000 ions/second – if too few ions are present, turn up the emission current or lower the threshold; if too many ions are present, lower the emission current.
- Go to the average single particle signal screen (press "Insert" in TOF mode).
- If the multiplier voltage and scaling factor are set correctly, the ions per particle (IPP) should be 1 (these are not particles, only ions, so ions/ion = 1).
- If IPP ≠ 1, modify the multiplier voltage and/or scaling factor. The scaling factor marks the age of the multiplier.

For a new multiplier, select the mass for multiplier calibration (m/z 46 sliding window = 2) and set the threshold as described above, then follow the steps below:

- Set the multiplier voltage to a low value (~ 1500 V) and go to the single ion window (press "Insert" in TOF mode). Ions/particles should be ~ 1; if not, go to the Multiplier & Chopper tab and change the scaling factor until the ions/particle in the single ion window ~ 1.
- Increase the voltage by 500 V and repeat this process several times.
- Fit the gain vs. multiplier voltage values to the gain equation. Scaling factor of multiplier gain = 1, by definition, at the time of calibration.

$$\text{Gain} = 1 \text{ at start} \bullet 10\text{^}(C_1 + C_2 \bullet U_{mult} + C_3 \bullet U_{mult}^2)$$

where U_{mult} is the voltage of the multiplier in kV and C_1, C_2, and C_3 are constant coefficients.[10]

[10] At low multiplier voltages the measurements are very noisy, because the weakly amplified single ion signals are not much more intense than the electronic noise. Therefore, only the data measured at higher multiplier voltages should be taken.

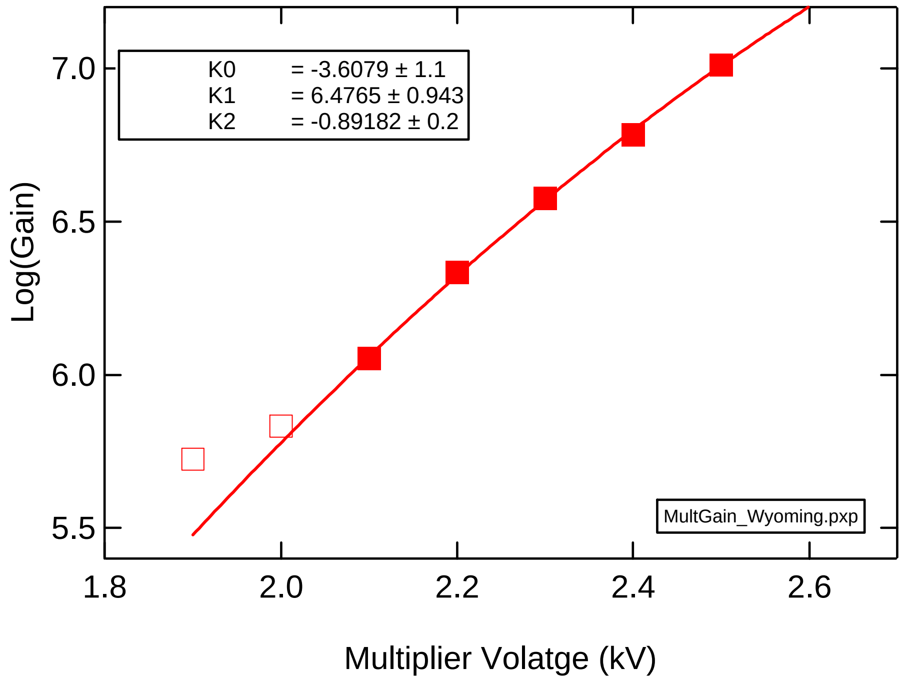

Figure 19: Multiplier Gain Curve

Quadrupole Mass and Resolution Calibration

The shape and position of mass spectral peaks must be calibrated to ensure that the AMS is performing properly. The resolution of the quadrupole mass spectrometer (QMS) determines the peak shape and spacing between peaks. It is important to have the optimum resolution because signal can be artificially lost or gained if it is set incorrectly. The QMS is set to an operating resolution of approximately 1amu to maximize ion throughput (signal).

Peak Shapes in Quadrupole Mass Spectra

The QMS is a mass filter, which can be passed by ions of a mass range, depending on the voltage settings of the mass spectrometer electrodes. This range, for which the mass filter is open, defines the resolution of the mass spectrum. To distinguish ions of different mass number, the mass resolution (m/Δm) at the mass of the ion has to be at least the value of this mass in amu (m/Δm = 100 for resolution of mass 99 and 100).

At high resolution, the mass line is a peak of asymmetric shape: It has a sharp tail on the right side (on the high mass side) and a longer, less-sharp tail on the left side (on the low mass side). Decreasing the mass resolution of the QMS makes the peak wider. It does not change the shape of the peak and the position of its right tail. The left, long tail walks away from the right tail and the maximum rises up. The peak shape can be changed by changing the value of the field axis, which is typically set at 14. The ideal peak shape is a "top hat" or a "flat top" shape so that the signal intensity for each amu can be determined by averaging over the flat section of the top hat. To maximize the ion transmission through the QMS, the resolution setting should be as low as possible. However, with too-low resolution the left tail of the peak ends in the peak of the next lower mass, which changes the intensity of this peak.

To summarize, the AMS should be operated with 1 amu resolution throughout the entire mass scan range. For optimum ion transmission, set the resolution as low as possible and as high as necessary (to get correct ion signals of the different masses).

In **MS Mode** of the AMS program the ion intensity at the single masses (also referred to as stick intensities in the mass spectrum) is measured as averaged ion signal, averaged over a window region (adjustable in the parameter window) in the center of the peak. The averaging width should be as large as possible to maximize signal to noise ratio, but it should not be wider than the flat top part of the peak to maximize signal intensity.

A window width of 0.4 amu centered about 0.5 amu to the left of the right edge of the peak is ideal for obtaining peak signal intensity. In the MS window this averaged ion signal is displayed as a box with height equal to the averaged intensity and width equal to the averaging window. The resolution of the mass spectra may be so low that the left tail of the peaks reaches the next peak on the low-mass side and alters the right tail of this peak. For correct ion signal calculation, it must not reach the integration box of the next lower mass peak.

In **TOF Mode**, the signal intensity at each selected m/z is determined at a single point on the peak. It is important that the maximum of the flat top section of the peak be used to determine the signal for each m/z. Since the quadrupole scan in TOF mode is slower than in MS mode, the peak shapes in the two modes differ slightly.

If the peak resolution has been set properly for the MS Mode as described above, the maximum of the peak for the TOF Mode is offset by approximately 0.6 amu to the left of the right edge of the peak. The value of the offset is usually the same for all masses and can be set for each selected mass in the m/z window (F6 from TOF window). In order to determine the best value for the offset in the TOF Mode, go to the m/z window and select m/z 28 (N_2^+). The top right corner of the window contains a mass spectrum in the region about the m/z = 28. The calibration is set properly when the mass peak in the middle of this window is located between the two black sticks rising into the window from the bottom. The black stick on the right denotes the selected mass and the right edge of the signal peak should line up with this stick. Since the TOF measurement for a particular m/z is only obtained at one point along the signal peak, the quadrupole should be set to the m/z value point that coincides with the maximum of the signal peak.

Peak Shape Calibration

The peak shape is a function of the quadrupole resolution, described as a linear function of amu as follows:

$$\text{Actual Resolution} = (\text{ResolutionSetting}) + 1\ (\text{Slope} \bullet \text{amu})$$

The Resolution Setting and Slope values are menu parameters that can be changed during calibration. The quadrupole resolution can be calibrated by a two-point calibration at 2 different m/z values. It is recommended to use a low mass (such as m/z = 28) for one calibration point and a high mass (m/z = 149, which is typically present as a background peak) to perform this calibration.

An automatic resolution calibration can be performed as follows:

- Click the "Calibrate MS" button on the top right corner of the MS window. This opens a calibration window with two graphs that display sections of the mass spectrum for the masses that will be used for the calibration.
- Vary the actual resolution setting for mass 1 and mass 2 so that the peak shapes look appropriate (as described on page 48), and the calculated average signal intensity at each mass is maximized.
- Changes in the actual resolution settings will automatically be used to determine the slope and intercept for the resolution calibration. These values are displayed in the Fit Results section.[11]

Peak Position Calibration

Once the peak shape has been set, the peak position can also be calibrated. The quadrupole mass (amu) positions can be described by a linear equation which relates the amu that is scanned to the bits output by the computer to the data acquisition (DA) board (the output bits are converted to an output voltage to the quadrupole using 16-bit resolution: 10V = 32768 bits).

$$\text{amu} = (\text{slope})(\text{DA board bits}) + \text{Offset}$$

The parameters for this linear relationship[12] are determined by a two-point calibration at two different m/z settings. The mass calibration should be corrected if the rectangular

[11] In this formulation, the Resolution Setting value is inversely proportional to the resolution of the mass spectrum

[12] Found in the Mass Spectrometer menu tab.

boxes that denote the averaging widths for the m/z values of interest do not line up with the maximum intensity sections of the peaks.

If a manual calibration is being performed, these menu parameters must be manually changed so that the 0.4 amu averaging window is centered on the flat top part of each m/z being used for the calibration. In newer AMS software versions, automatic quadrupole mass calibration can be performed as described on page 53:[13]

[13] Often it is only the offset in the m/z calibration that changes as a function of time. In this case, the offset in the mass scale can be moved by pressing the y and Y key while viewing MS peaks and their signal average boxes in the normal MS window.

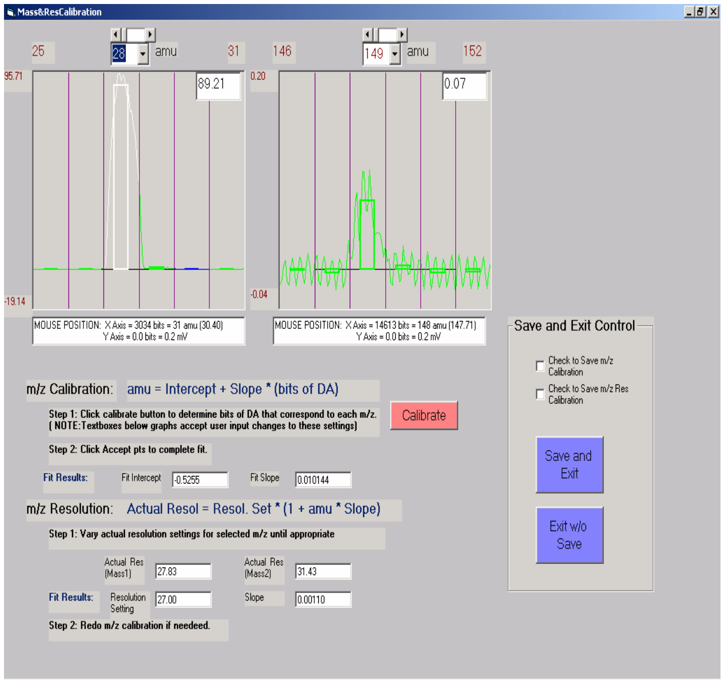

Figure 20: Screen: MS Calibration

PROCEDURE FOR MS CALIBRATION:

- Press the "Calibrate MS" button in the upper right corner of the MS screen. This window appears (Figure 20).
- Press the Calibrate button, and if the new positions are acceptable, click the Accept button. If the values are not acceptable, they can be changed to modify the suggested positions of the boxes before clicking the Accept button.
- Results of a linear fit to the 2-point calibration are displayed in the text boxes in the Fit Results section.[14]

[14] Changes in resolution and m/z calibration can affect each other so it is often necessary to repeat the calibrations until no large changes are observed.

- Before exiting, check ☒ Save m/z Calibration to save the fitted slope and intercept.

Ionization Efficiency (IE) Calibration

Ionization efficiency calibration, also called the mass or nitrate calibration, measures the ionization and ion transmission efficiency of ammonium nitrate. Ionization efficiency is the ratio of the number of ions made to the total number of available parent molecules for that ion species (*e.g.,* if the ionization efficiency is 1 x 10^{-6}, then 1 molecule in 1 million molecules is ionized).

In the IE calibration the product of ionization efficiency and ion transmission efficiency is determined for NH_4NO_3. For any given parent molecule, the total number of ions produced is determined by a sum of ion intensities of all its fragment ions.

If a precise fragmentation fraction for a given fragment ion is known, then the total number of ions from the parent molecule can also be expressed as the product of that fragment ion intensity and the inverse of the fragmentation ratio. The quantification of the AMS is based on the linearity of the ionization efficiency. Larger molecules have larger ionization efficiencies than smaller molecules, and the increase in ionization efficiency is linear with increasing molecule size.[15]

If the ionization efficiency can be determined for one molecule, the ionization efficiencies for all other species can be related to the measured ionization efficiency of the initial species.

Ammonium nitrate is used as the primary mass calibration species because the ionization efficiency, density, and shape are well known, and ammonium nitrate does not leave much residue to interfere with subsequent measurements. Ammonium nitrate vaporizes with close to 100% efficiency, so the ionization efficiency of NO_3^+ can be quantitatively

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[15] McLafferty and Turecek, 1993.

measured, and it is well-focused by the aerodynamic lens so that all the particles can be detected.

The fundamental assumption as described above is:

$$\frac{MW_i}{IE_i} = \frac{MW_{NO_3}}{IE_{NO_3}} \times f_i$$

where MW_i and IE_i are the molecular weight and ionization efficiency of a given species, MW_{NO3} and IE_{NO3} are the molecular weight and the ionization efficiency calculated for ammonium nitrate, and f_i is the calibration factor representing the relationship of the ionization cross-section of the species to that of ammonium nitrate.

The ionization efficiency for nitrate (IE_{NO3}) is calculated by determining the number of ions produced per particle of a select size as shown in the equation below:

$$\frac{\text{Ions Per Particle}}{\pi/6 \text{ x } d^3(\text{nm}) \text{ x } \rho(1.72\text{g/cm}^3) \text{ x } (1 \text{ x } 10^{-7}\text{ cm/nm})^3 \text{ x SF}(0.8) \text{ x } f_{NO3}(0.775)} \text{ x } \frac{MW_{NO3}(62\text{g/mole})}{6.02 \text{ x } 10^{23}\text{ molec/mole})}$$

where ions per particle is determined from the calibration (usually several hundred), *d* is the mobility diameter of the calibration particles (typically 350 nm), ρ is the density of ammonium nitrate, *SF* is the shape factor (<1 for non-spherical particles), f_{NO3} is the fraction of NO_3 in

$$NH_4NO_3\ (MW_{NO3}/(MW_{NO3} + MW_{NH4}))$$

and N_{AV} is Avogadro's Number.

To get the aerosol mass loading (*i.e.,* mass/volume) in μg/m^3 for a particular ion from the mass spectrum, the following calculation must be performed:

$$\left(\quad \right) \left(\quad \right) \left(\quad \right) \left(\quad \right)$$

$$\frac{\text{ions/sec(Hz)}}{\text{Flow(cm}^3\text{/s) x N}_{AV}\text{(6 x 10}^{23}\text{molec/mole}} \text{ x } \frac{MW_i}{IE_i} \text{ x } 1 \text{ x } 10^6\text{cm}^3/\text{m}^3 \text{ x } 1 \text{ x } 10^6\mu\text{g/g} = \frac{\mu\text{g}}{\text{m}^3}$$

$$\frac{MW_i}{IE_i} = \frac{MW_{NO3}}{IE_{NO3}} \times f_i$$

Or, using the substitution above:

$$\left(\frac{\text{ions/sec(Hz)}}{\text{Flow(cm}^3\text{/s) x N}_{AV}\text{(6 x 10}^{23}\text{molec/mole}}\right) \text{x} \left(\frac{MW_{NO_3}\text{(62g/mole)}}{IE_{NO_3} 1 \text{ x } 10^6}\right) \text{ x } \left(1 \text{ x } 10^6\text{cm}^3/\text{m}^3\right) \text{ x } \left(1 \text{ x } 10^6\mu\text{g/g}\right) = \frac{\mu\text{g}}{\text{m}^3}$$

When calculating the mass loading from TOF mode, the duty cycle of the chopper must also be taken into account.

Automated IE Calibration

PROCEDURE:

- In F6 window select m/z for calibration. Make sure that the species columns for the selected m/z values are filled in appropriately. If a new IE calibration is to be performed with NO_3, the first m/z selected must belong to the NO_3 species. If other species are to be calibrated, m/z fragments that correspond to those species must also be selected. If an accurate IE is to be calculated, **all** major m/z fragments formed from the species of interest must be selected.
- In addition to the species being calibrated, select m/z belonging to species AIR so that the air beam signal can be monitored at time of calibration.
- Using TOF acquisition mode, set thresholds for selected m/z values. Set the blue lines in the Single Particle graph so that they group the particles of interest of the desired size. The IE will be calculated for Region #2, which should contain the particles of interest.

- If a CPC is attached, compare the AMS count rate of the 350 nm NH_4NO_3 particles with that of the CPC. Typically the AMS should count 90-100% of the particles. Also, look at the average TOF traces and make sure that all of the AMS NO_3 mass at m/z 30 and 46 for the 350 nm particle is counted. If not, check the single particle threshold that has been set.
- Once the initial checks are complete, activate alternate MS/TOF mode and let the system average for a few minutes. After averaging is completed, press Shift M to bring up the calibration window.
- Fill in correct information about the calibration particles being used in the User Input section. Typical inputs for NO_3 calibrations are 350 nm pure NH_4NO_3 calibration particles. In this case, the density used in the $\mu g/m^3$ calculations is that of NH_4NO_3, but these calculations need to be corrected for the fact that m/z 30 and 46 detect only NO_3(MW=62) in NH_4NO_3 (MW=80). This is done with the Mass Fraction of Species entry which is 62/80=.775)
- If only NO_3 is being calibrated, then the mass fractions for species 2, 3, and 4 should be set to 0. If any other species are being calibrated (*e.g.* NH_4 from NH_4NO_3 or SO_4 in a mixed $NH_4NO_3/(NH_4)_2SO_4$ particle) then the appropriate mass fractions and species designations should be entered in User Inputs.
- Click "Calibrate Now." This action automatically starts the AMS in alternate TOF/MS mode and displays performance parameters. A TOF and MS file with the most recent run number will be saved in the C:/AMS/AMSData/NonAutoSave/ directory.
- Make a note of AB (both MS and TOF), IPP, TOF/MS μg, IE, and IE/AB.
- Save a screen picture.

Flow Rate Calibration

Knowledge of the precise rate of flow of gas into the AMS is required to normalize the measured particle mass to the volume of sample taken. This allows one to report particle mass loadings in the conventional units of $\mu g/m^3$, where µg comes from the mass spectrometer measurement and the m^3 represents the volume of air sampled.

The flow of sample into the AMS is fixed by a critical aperture (~100 µm diameter) mounted upstream of the aerodynamic lens. The pressure drop across this aperture is large enough that a choked-flow condition exists (ambient pressure to ~1 torr). Under these choked-flow conditions the volumetric flow through this orifice is constant. The acquisition program continually monitors this flow rate by measuring the pressure at the entrance to the aerodynamic lens. [16]

There is a unique relationship between lens pressure and flow and lens transmission properties. In this system an absolute pressure gauge is used to monitor the lens inlet pressure, and from calibration the volumetric flow can be extracted. This relationship between pressure and flow is based on the Poiseuille Equation, relating the volume flow rate of a viscous gas through a laminar flow element. In this case the laminar flow element is the aerodynamic lens tube which has an entrance pressure of ~ 1 torr and an exit pressure of <10m torr.

[16] Note that this critical aperture plays a more fundamental role in fixing the lens inlet pressure, which defines the focusing and transmission properties of a particular lens system.

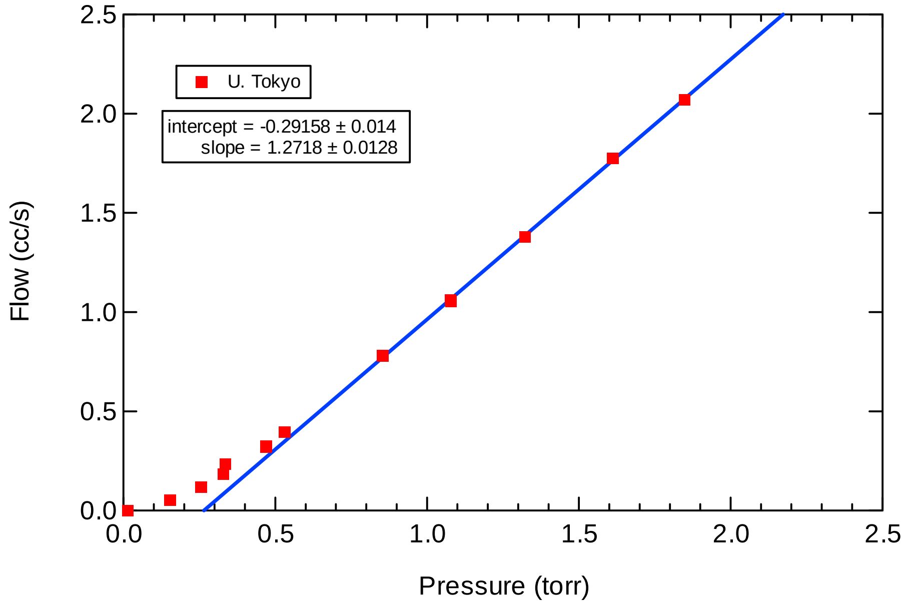


Figure 21: Flow Curve

FLOW CALIBRATION PROCEDURE:

Replace the critical aperture with a variable leak (a needle valve) and record the lens inlet pressure for a series of different flow rates. For the calibration, the volumetric flow is measured using a soap film flow meter or equivalent.

This approach uses a mass measurement device (the absolute pressure gauge) to report a volumetric flow. For this reason, it is important to record the ambient pressure and temperature at which the calibration was performed so that subsequent measurements at different ambient pressures and/or temperatures can be converted back to a volumetric flow.

Aerodynamic Lens Alignment

The aerodynamic lens forms low-divergence beam of particles. The particle beam passes through the skimmer, through the chopper and then hits the vaporizer at the ionizer assembly of the QMS.

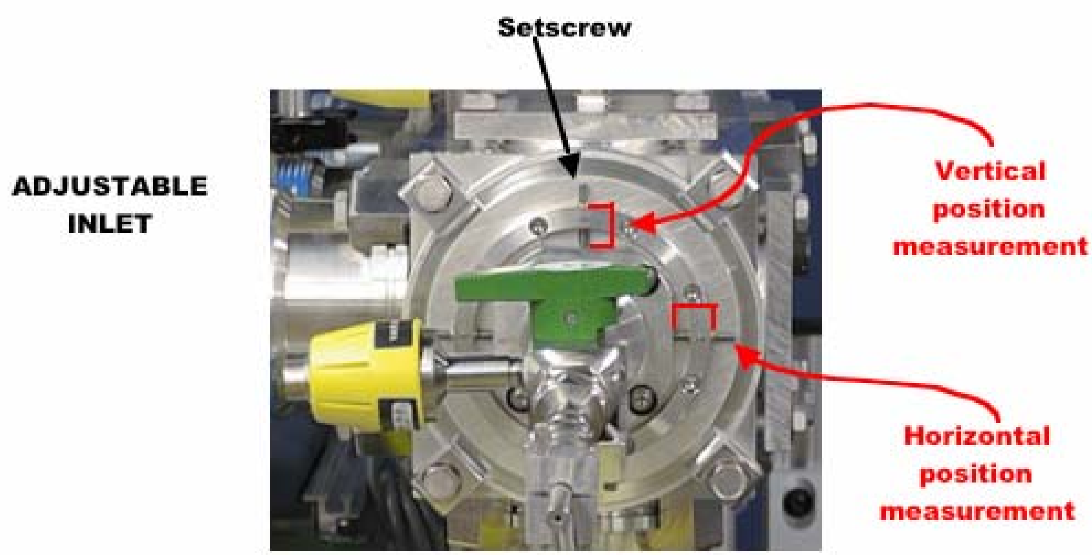


Figure 22: Lens Alignment

The lens position is important to getting the widest beam collection angle. Normally, the lens position should be adjusted after moving the instrument and then locked in place and not moved again unless there is a possibility it may have been knocked out of alignment.

ALIGNMENT PROCEDURE:

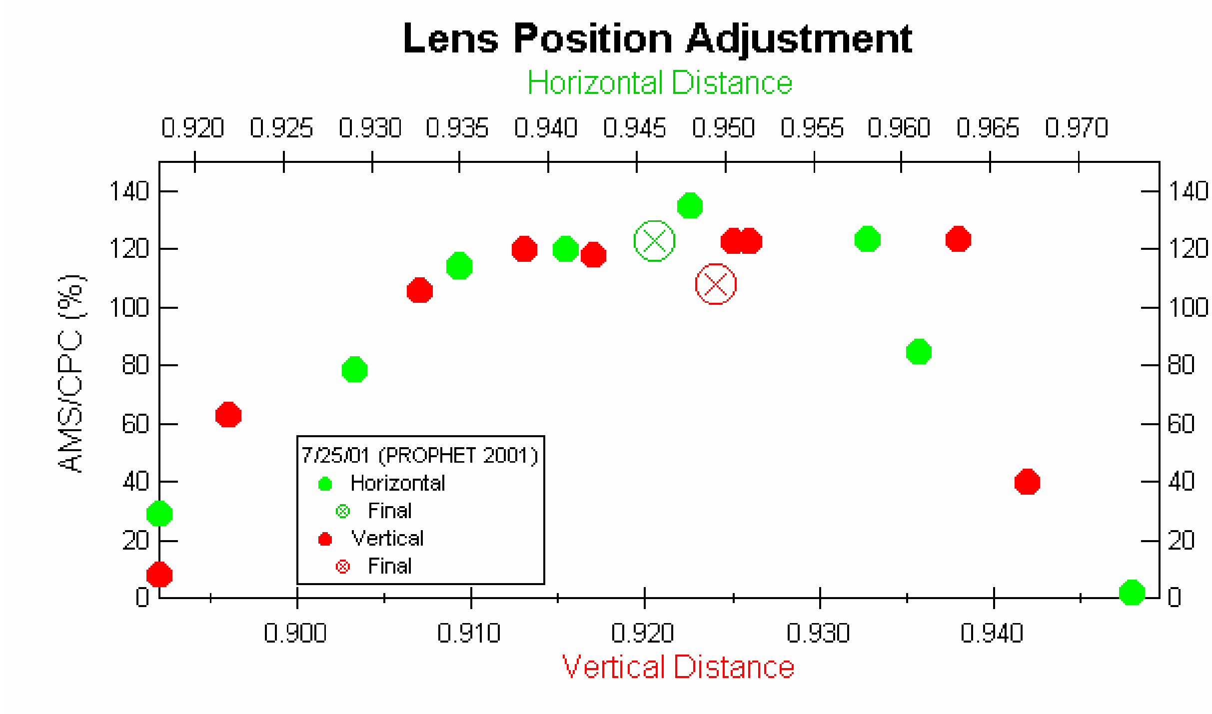
- First, set up a DMA to produce 300-350 nm particles and connect a CPC to the AMS. We know particles in this size range focus to ~ 0.5 mm diameter.

Make sure that the output of the DMA is constant by watching the number reported to the CPC and AMS before beginning the lens position calibration.

- Loosen the locking screws holding the lens plate so that the lens plate floats on its o-ring and loosen the *Ultratorr* fitting holding the lens in place.
- Press F6 in TOF mode to get to the m/z selection window.
- Set the AMS to detect m/z 30 and 46. Make a note of the IPP and AMS/CPC % at the initial position.
- Adjust the setscrews that hold the lens in place and move the lens, either horizontally or vertically, to center it (See next instruction). Refer to Figure 23.
- Measure the lens position carefully from the outside of the lens plate to the lens, ~ 0.9 inches (the lens position is easily changed when all the screws are loosened, so be careful not to change the position while measuring). Always measure from the same place, usually right next to the setscrew (See red bars on diagram).
- Again, make a note of the IPP and AMS/CPC % at this position.
- Continue moving the lens either horizontally or vertically and noting the IPP and AMS/CPC % until the performance of the AMS in that direction has been fully mapped.
- Move to what appears to be the best position (highest IPP and AMS/CPC= ~100%) and then continue mapping in the other direction.

- Again, find the best position and double-check the AMS performance (See Figure 23, page 63).

- Tighten the locking screws holding the lens plate and the *Ultratorr* fitting and then recheck the IPP and AMS/CPC, since tightening the lens plate can alter the lens position.



-

Figure 23: Lens Position Adjustment

Particle-Sizing Calibration

The AMS reports an aerodynamic particle size based on a particle velocity measurement. As described in the Theory of Operation section, particle flight times are measured using a beam-chopping technique. Particle size calibrations are performed by sampling particles of known size, usually polystyrene spheres (PSLs) and particles delivered from a Differential Mobility Analyzer (DMA), usually NH_4NO_3. The use of PSLs represents a primary standard, but the range of sizes is somewhat limited. Therefore, the DMA particles can be used to extend the range of measurements to smaller sizes.

For PSL measurements, the first step is to increase the vaporizer temperature to ~750-800C (1.3-1.4 amps). In the data acquisition program, select mass 104 (styrene parent mass) in the F6 menu. Particle TOF data is then recorded for a series of different size PSLs. Similarly, NH_4NO_3 particle TOF data is collected for a range of sizes, but in this case mass 46 is selected and a vaporizer temperature of ~600-650 is set.

Figure 24 shows typical particle TOF data for both PSL and NH_4NO_3 aerosol. Mass peak arrival times are indicated for two peaks. Particle velocity is calculated from these

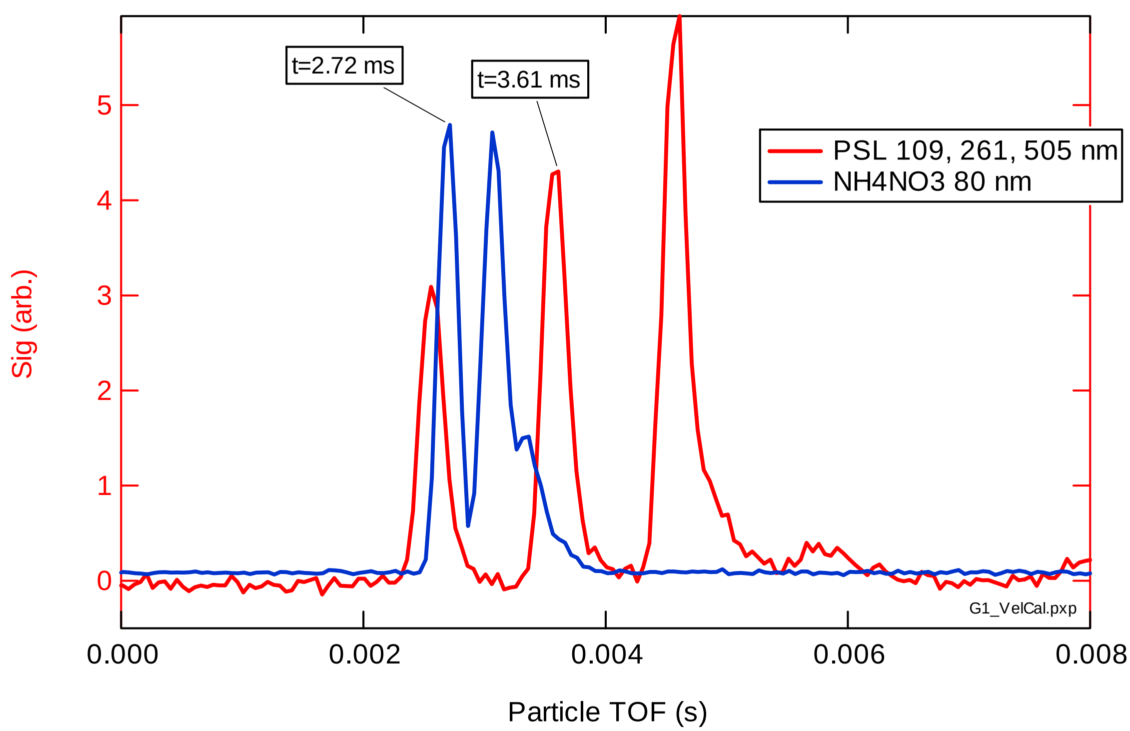


Figure 24: Particle Times of Flight

times, and the particle flight distance (0.395 meters for 255-xxx series chambers and 0.293 meters for the 215-xxx series chambers). In this example, the PSL data contains a mixture of 3 different PSL sizes and the NH_3NO_4 data shows the multiple charged diameters for a selected mobility diameter (Q=1) of 80 nm. The calculated velocities are plotted against particle aerodynamic diameter as shown in Figure 25.

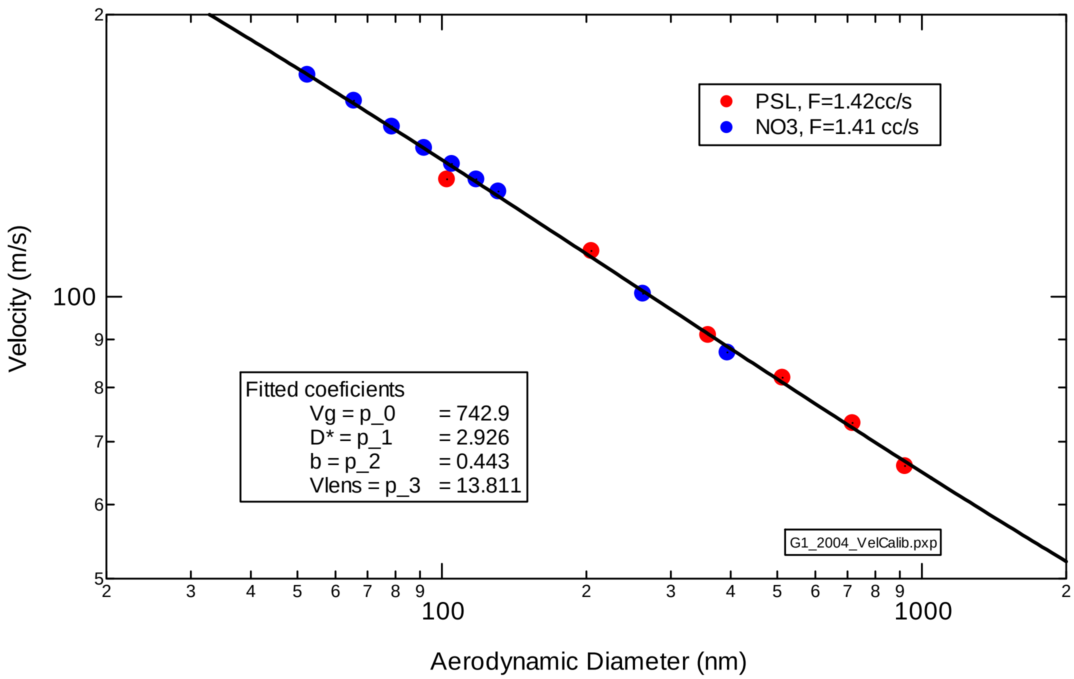


Figure 25: Velocities plotted against Particle Aerodynamic Diameter

Aerodynamic diameter is defined as the product of geometric diameter and density. For PSLs the density is 1.05 gm cm^{-3}; therefore, in the example shown, a size of 261 nm has an aerodynamic diameter of 274 nm and the velocity is 109.4 m s^{-1} (0.395/0.00361). NH_4NO_3 density is 1.72 gm cm^{-3}, so its aerodynamic diameter is the product of the mobility diameter and the density. The NH_4NO_3 velocity data is aligned with the PSL data by applying a shape factor,

Daero = Dmobility * density * shape factor.

The shape factor is interpreted as a density modifier and is usually in the range of 0.8 to 0.85. The assumption is that only 80 to 85% of the particle is solid NH_4NO_3, the remaining percentage is void volume. This is the origin of the shape factor that is applied to the NH_4NO_3 ionization efficiency. Note that the NH_3NO_3 DMA-generated particles extend the measurements to smaller sizes and the PSLs extend the measurements to larger sizes. It is important to cover as wide a range as possible in size since extrapolating the fitted curve beyond the data points can lead to significant errors.

The solid line in Figure 25 is a non-linear least squares fit to the combined PSL/NH_4NO_3 data set using the following empirical equation:

$$\text{Velocity} = Vgas_{lens} + [Vgas_{exit} - Vgas_{lens}] / [1 + (Daero/D^*)^b]$$

Where $Vgas_{lens}$ is the velocity of the gas in the lens, $Vgas_{exit}$ is the gas velocity at the lens exit, D* is an effective scaling diameter and b is the power dependence. The Vgas terms provide limits to the particle velocity for small and large size particles. A small particle cannot travel faster than the expanding gas ($Vgas_{exit}$) and a very large particle cannot go slower that the velocity of the gas in the lens ($Vgas_{lens}$). The product of the velocity calibration is the fitted coefficients, which, for this example, are shown in the figure. These coefficients are then entered into the data acquisition program menu under the Flow, Size & Mass Calib. tab. (See Figure 26)

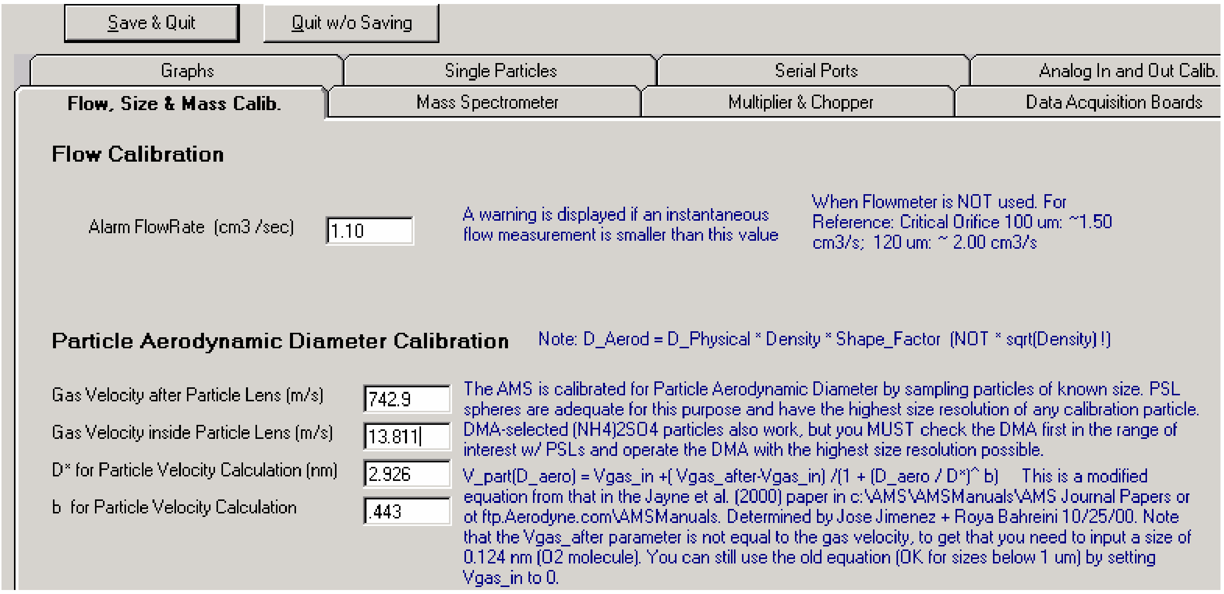


Figure 26: Screen: Data Acquisition Program Menu

Servo Motion/Position Calibration

This should be performed after transport and or manipulation of the chopper flange; it does not need to be performed at any other time unless there is a significant difference between the sizes of the MS and TOF air beam. [17]

[17] Alternatively, this can be done every couple of weeks as a precaution.

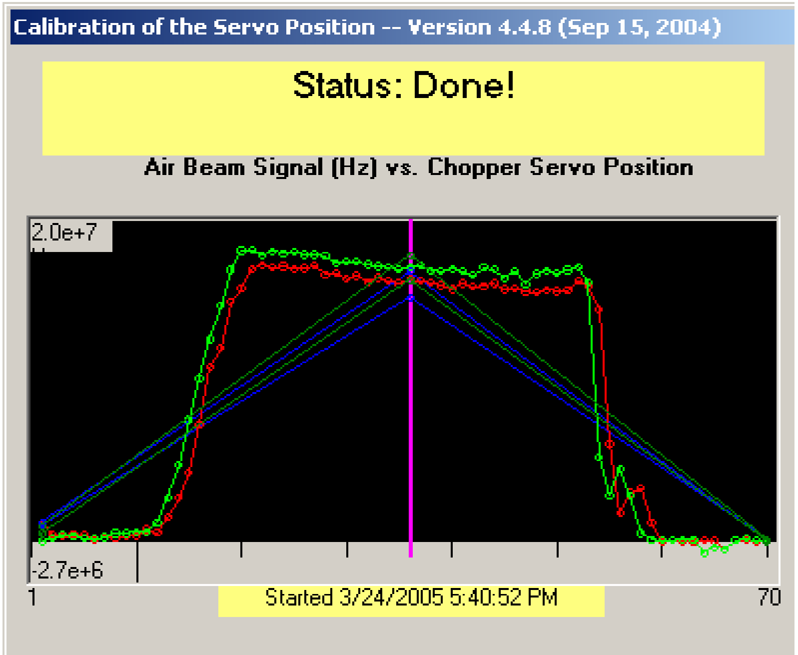


Figure 27: Screen: Servo Motion/Position Calibration

PROCEDURE FOR SERVO MOTION/POSITION CALIBRATION:

- Quit (press Q) to main menu and choose "Servo Adjust."
- Let it run and note shape of signal as the servo walks the chopper across the beam both forward (red trace) and backward (green trace).
  - There should be a "top hat."
  - If the top hat is positioned evenly over the initial servo positions (*i.e.*, the set chopped position is in the middle of the top hat and the completely open and closed positions are beyond the edges of the top hat, then the servo is adjusted correctly.
- Check hysteresis between the green and red curves. A large hysteresis could indicate that the servo will fail or the mechanical linkage has loosened up. Acceptable hysteresis is 2-3 steps.

- If the top hat is not positioned evenly, calculate center of top hat and note center number.
  - Quit (press Q) and choose the Multiplier & Chopper tab
  - Set the three positions based on the center of the top hat: center – 30, center, center + 30.[18]
  - Typical values: 10, 38, 65 (screen shows values 110 greater than these).
- Quit (press Q) and return to Mass Spectrum mode.
- Toggle chopper position (shift T) and note air beam signal (this should not change if servo was set correctly).

[18] First position must be made positive, to turn off single-stepping, even if there is no change of chopper position (this will eventually be automated in the code).

Acquisition and Analysis Software Overview

Data Acquisition Modes

The AMS is controlled and operated by a stand-alone software program that operates under Windows™ operating system. This program configures the instrument, interfaces with the mass spectrometer and performs data collection based on user-specified parameters.

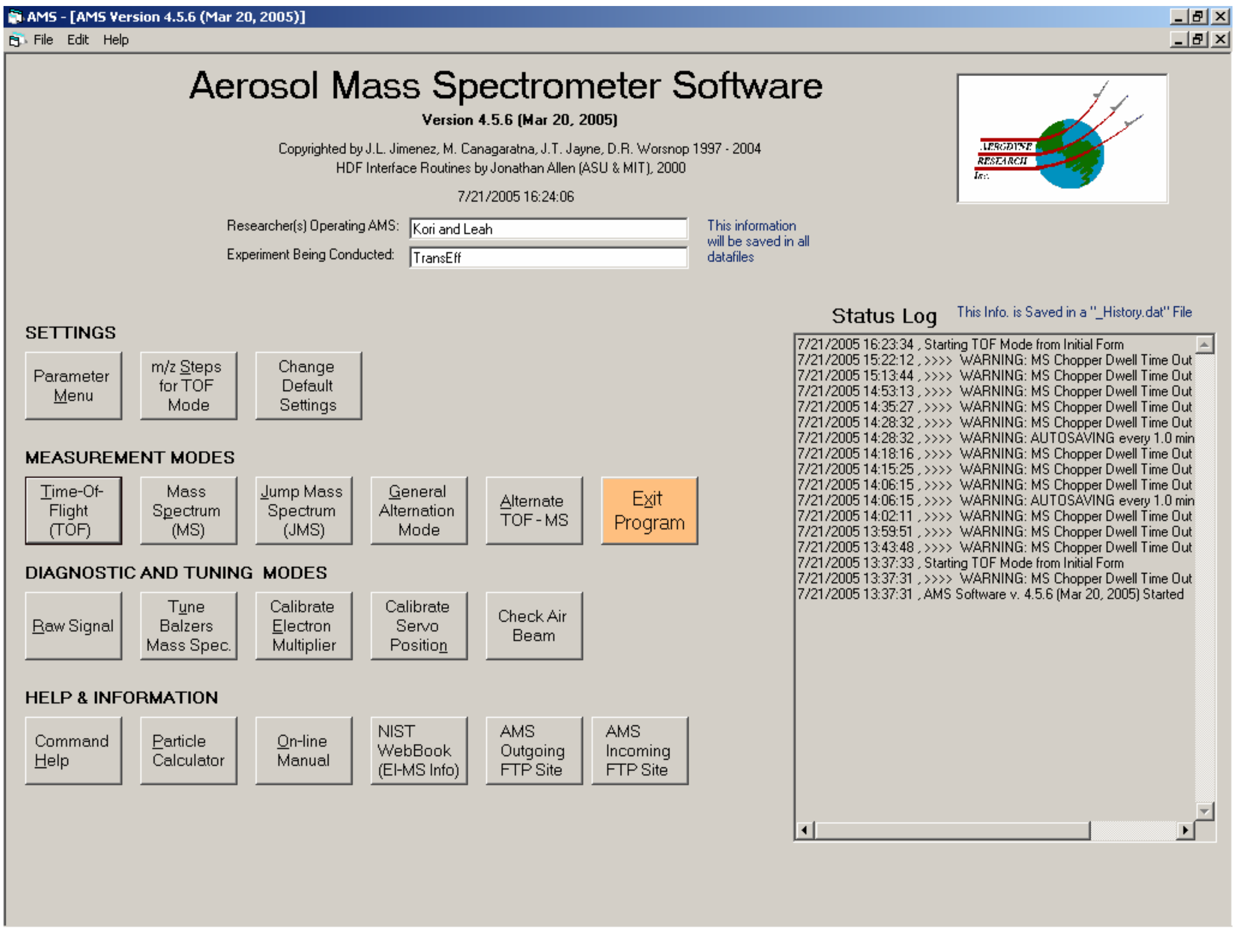


Figure 28: Screen: AMS Software

The following discussion provides a brief overview of the AMS data system. A more detailed discussion of the operating software is provided in a separate document, the *AMS Data Analysis Software Manual*.

The AMS is configured to operate in several modes. The standard modes are referred to as the TOF mode and the MS mode.

TOF Mode

In the TOF (time-of-flight) mode, chemically speciated size-resolved data is obtained. In this mode, the quadrupole mass spectrometer is programmed to "sit" on one of several pre-programmed masses. Ion signal intensity is monitored as a function of the rotational phase of the chopper. This measurement of particle flight time is used to determine particle aerodynamic diameter. In TOF mode, the chopper is positioned so that the beam is "chopped." While this allows particle size to be measured it also reduces the overall particle throughput by the ratio of the chopper duty cycle. For example, a 2% chopper will block 98% of the particles. Several different spectrometer settings are usually selected to characterize different chemical species (sulfate, nitrate, ammonium, organics, etc.). In TOF mode, the spectrometer is step-scanned at 3Hz over the different pre-selected masses.

MS Mode

In MS mode, more complete information on chemical composition is obtained, but particle size information is not measured. In MS mode, the chopper assembly is removed (by computer control) from the beam path allowing all the "focused" particles to reach the detector. Here, the mass spectrometer is repeatedly scanned over a predetermined range (typically from 0-300 amu) providing an ensemble average composition for the particles that are vaporized and detected. The scan rate in the MS mode is 1 ms/amu, so a 0-300 amu scan occurs at a 3 Hz rate. Typically, the data acquisition software is set up to alternate between the MS and TOF modes at a user defined interval (10-20 seconds) so there exists a TOF mode, a MS mode and an Alternate mode of data collection.

Alternate Mode

In Alternate mode, the data acquisition software writes data to the computer hard drive storage at user-defined intervals. Data save times in this mode are typically >30 seconds and represent the average composition determined over this sampling interval.

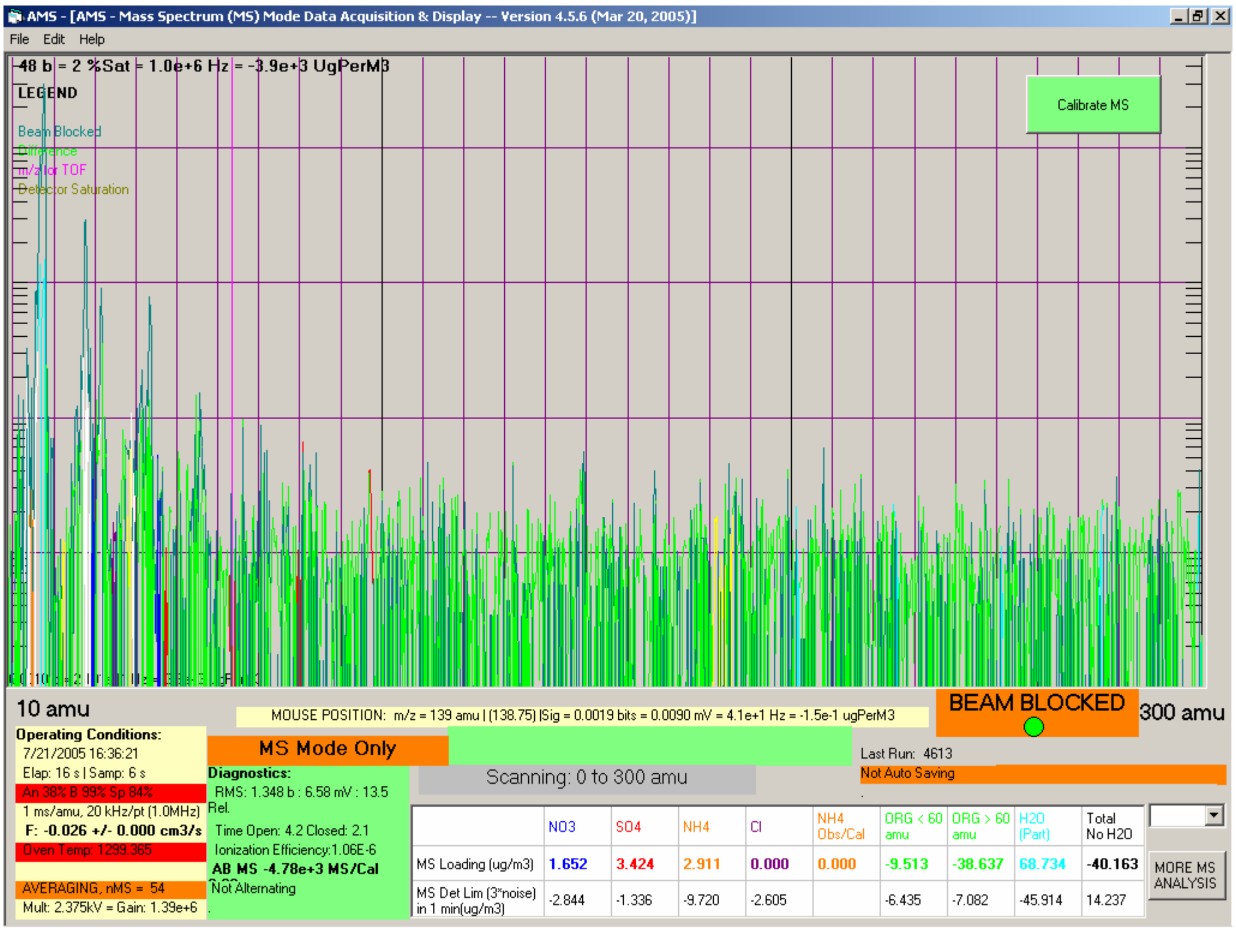


Figure 29: Screen: Example, MS Mode

Special Modes

There are several other modes of operation, which have evolved for special sampling purposes:

- 4-second mode
- Jump mode
- Selective scan mode
- Eddy correlation mode

These modes are explained in the *AMS Data Analysis Software Manual*. They have been developed to provide higher time resolution (up to 1 Hz), which is often required for measurements made on mobile platforms (aircraft, trucks) or for short-lived particle plume events.

Data Analysis Overview

The process of analyzing AMS data is separate from the data collection process. Data that is saved by the AMS acquisition program is in a format that can be directly read by IGOR, a data plotting and analysis software by Wavemetrics™. An IGOR data analysis procedure written by Dr. James Allan, School of Earth, Atmospheric and Environmental Sciences, University of Manchester, UK is available for all AMS users. A licensed copy of IGOR is delivered with each instrument. The data analysis program provides a rapid way to process AMS data. Detailed discussion of how to operate this software can be found in the *AMS Data Analysis Software Manual*. Functions are available to calculate time trends of particle mass loadings for selected chemical species, to analyze individual or group-average mass spectra. Individual or group-average particle TOF data can also be analyzed and images can also be displayed that show size distribution time series for the different masses that were monitored in TOF mode.

The analysis package also provides data diagnostic and data correction functions that help verify the quality of the data. One of the most important components of this analysis procedure is the "frag list." This feature is at the heart of the AMS analysis and provides a way of interpreting the mass spectra so that different chemical classes can be extracted from the spectra.

Interpretation of mass spectra can be complicated by the fact that different species can be detected at the same amu. Using the frag list makes it possible to separate the relative contribution or ion intensity that gives rise to a particular mass peak. The list is based on laboratory measurements of different aerosol types, known isotopic ratios, and ratios from the NIST database for electron impact ionization mass spectra for certain species. For some species it is simply a "best guess," based on comparison with data from other particle instrumentation. The frag list has developed over time and continues to evolve as our understanding of the data grows.

IGOR procedures are open source code, and other AMS users have contributed specialized routines for different applications. Since this is open source code, all users have the ability (and responsibility) to optimize the data processing for their individual applications. The majority of AMS users use this data plotting and analysis tool; however, it is not the only way to process AMS data. All AMS data files are written in ASCII format so they are easily imported into different data analysis and plotting software packages.

Maintenance

Almost all problems are related to items that are actively in use: pumps (pressure), airflow (inlet), and ion detection (vaporizer, filament, QMS, multiplier), so it is important to keep these parts clean and in good working order.

Cleaning

Keep the system clean and dust-free. After field deployments or extended operation (several months), the dust filters on the rack-mounted electronics should be removed and cleaned.

Leak Test

Typical pressure in the multiplier chamber is in the mid to low 10^{-8} torr region after pumping for 24-28 hours. Typical pressure in the detection region of the AMS is mid to low 10^{-8} torr after pumping for more than 24 hours.

The performance of the AMS depends critically on the vacuum level. On a "leak-free" system, leaks can arise over time from continued temperature cycling of the 1/2" Swagelok fittings used on the roughing stages of all the turbo pumps. These fittings use Teflon ferrules, which can soften and flow over time. Often a light tightening of the fittings will reduce leak rates to an acceptable level. If the vacuum system was vented and components removed and then refitted, the system should be leak-checked.

Leaks can be caused by loose bolts, bad o-rings on the flanges or chopper feed-thru, dirty o-rings or dirty/scratched metal surfaces.[19] Clean the o-rings and the flanges by blowing clean, dry air on them. Scratches can be removed by sanding the flanges with small-grit sandpaper (400 or higher). To locate the leak, use the following procedure if the pressure is low enough to start the QMS.

The recommended leak-checking procedure requires the use of a digital voltmeter (DVM). If a DVM is not available, set the acquisition program to operate in MS mode with a narrow scan range around mass 4 (helium). If a DVM is available, connect the output of the preamplifier to the DVM (the MS signal will be disconnected from the computer). Operate the acquisition program in the TOF mode with the TOF mass set to

[19] Use small quantities of vacuum grease on o-rings.

m/z 4 (helium). Increase the preamplifier gain to 10^{-9} A/V. Set the DVM to 100-200 mV scale. An acceptable leak rate will yield a signal of ~20-50 mV with this gain setting (also assuming the multiplier gain is ~$2x10^6$). Spray helium around the AMS at various points and monitor the DVM reading. To verify that helium is being detected, it may be necessary to open the sampling valve and sample a small amount of helium through the inlet.

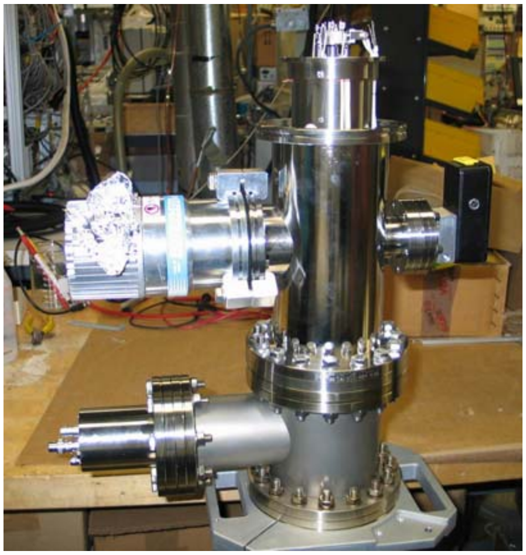
Filament replacement

The QMS has two filaments, only one of which is active at any given time. If one fails, the other can be used as a backup. Sooner or later, however, filaments need to be replaced. This procedure requires shutting down the vacuum system, venting and removing the quadrupole. The quadrupole should be removed with Pump #6 attached. The assembly weighs approximately 55 pounds. Follow the photographs below:

Figure 30: Filament Replacement

Removal of quadrupole from vacuum chamber: Remove four claw clamps and quick clamp at roughing port on Pump #6. Rotate quad assembly slightly to left before lifting up. Note hand position (See detail in Figure 31). This especially important when reinstalling.

Figure 31: Detail, Quadropole Removal: Hand Position



Mount the quadrupole assembly "upside down" as shown, using the supplied bench-mount clamp. This orientation facilitates filament replacement.

Figure 32: Filament Replacement: Quadrupole Mounting

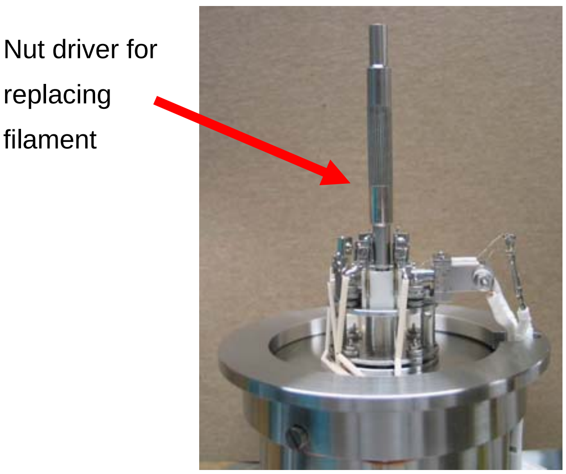


Figure 33: Filament Replacement

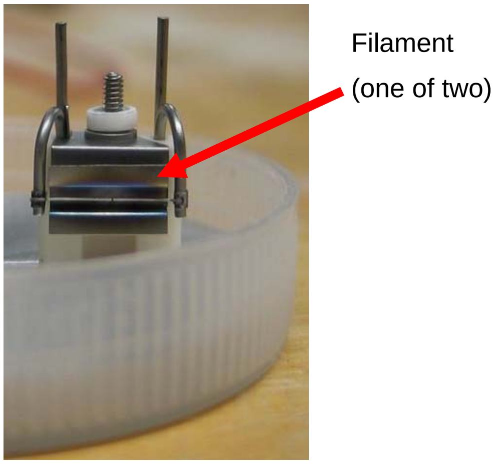
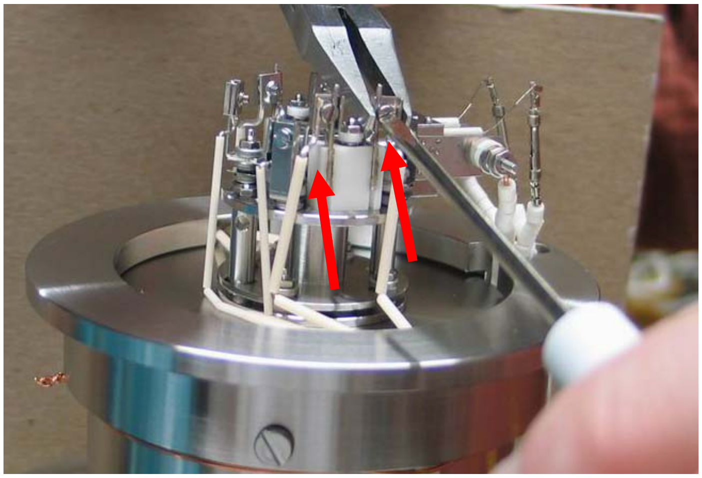


Figure 34: Replacement Filament Assembly



Loosen two screws connecting filament leads. Use clean needle-nose pliers to support filament post. Do not torque against unsupported post.

Figure 35: Filament Replacement

Replacement of Chopper Servo

The servo that positions the chopper wheel will eventually need replacement after continued use, typically 1 to 1-1/2 years of use. The procedure and important issues concerned with the replacement of this unit are as follows:

The servo should be replaced with a *Hitec Model HS-81 Micro*, available at most RC hobby shops, or see http://www.servocity.com/html/hs-81_micro.html.

The servo has three leads, black (GND), red (3.25-3.5V power) and yellow or white, the control signal. The servo is controlled by a pulse-width modulated signal that originates from the AMS software/NI boards applied to this line. This signal is a 50 Hz TTL pulse with a variable high pulse period of ~1-2 ms. Duration of the pulse sets the rotational position of the servo.

PROCEDURE:

1. Vent the system and remove the chopper flange. Note the alignment mark that is scribed on the outer diameter of the chopper flange and the vacuum chamber. When reinstalling, realign the flange to this alignment. Note that removing the chopper flange will alter the current velocity calibration.
2. Place the entire flange assembly, with the cable attached, on a nearby surface so that the chopper wheel and slide assembly are free to move.
3. Disconnect the servo 3-pin electrical connector and plug in the new servo. Be sure to match up the black/red/yellow (or white) colors on the connector between the mating connectors.
4. Run the AMS program in the MS toggle mode so that the command for servo movement is issued (toggling between block and open position) and observe that the new servo is actually moving. If it is not, see page 90.
5. After confirming that the new servo operates, go to the menu and set the chopper "Chop" position to 35. This is the electronic center position for the servo. Close the menu and run the program in the TOF mode. This will set the servo to the new center position. At this point, disconnect the 10-pin circular connector on the chopper flange. When disconnecting this cable carefully observe the servo output shaft and verify that the servo position did not "jump" when power was removed. If it did, repeat this step.
6. The next step is to remove the failed servo unit. Pay close attention to how this mechanical linkage is attached, *i.e.* which side the mechanical linkage is on. First, remove the servo output arm that couples the rotary motion of the servo to linear travel on the slide assembly – this is the small white 4-arm cross attached with a

Phillips screw. Remove the servo arm by gently prying it off the servo body. Note that the output shaft has splines to align the output arm.

7. Also, at this stage check the movement of the ball joints' couplings. The plastic fitting over the ball joints should move freely. Too much friction here can cause premature failure, as the servo has to work harder than needed. If the joints appear tight, use a sharp razor blade to slice the plastic coupling that fits over the ball to allow it to expand. It appears that, over time, these plastic couplings shrink while under vacuum and lead to this "high friction" condition.
8. Mount the new servo unit; secure it with the two #2-56 machine screws and tie the servo cable to the side of the assembly.
9. Next, replace the servo output arm on the output shaft. The important issue here is getting the correct rotational position. Since the servo was set to the electronic center (step 5), the output arm should be placed on the shaft in such a way that the slide assembly is at its halfway point (note that the slide has 1/2" of total travel). Do not replace the Phillips screw that holds the output arm in place at this time.
10. Reconnect the cable to the flange and operate the program in the TOF mode. Using the "g" and "G" keystrokes to position the chopper in the block/chop and open positions, observe the assembly for smooth and un-hindered movement. Make sure that the slide has not "bottomed" out in either the block or open positions and that, in the open position, the wheel is not touching the flange. If there are problems in this step, either the output arm was replaced in the wrong position or the mechanical linkage (the #2 threaded rod with the ball joint ends) needs adjustment. Make any necessary adjustments. If the assembly operates properly, replace the Phillips screw to secure the output arm. Fine adjustment is made when the system is operating using the "Calibrate Servo Travel" procedure described in the *AMS Data Analysis Software Manual*.

11. Several measurements should be verified before reinstalling in the vacuum. Refer to the drawings on the following pages. These drawings show the beam location with reference to the vacuum side flange surface. The two reference dimensions shown in the figure are 2.450" and 2.150".

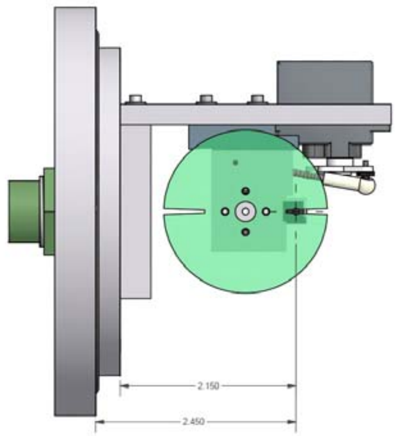


Figure 36: Servo in Beam Block Position

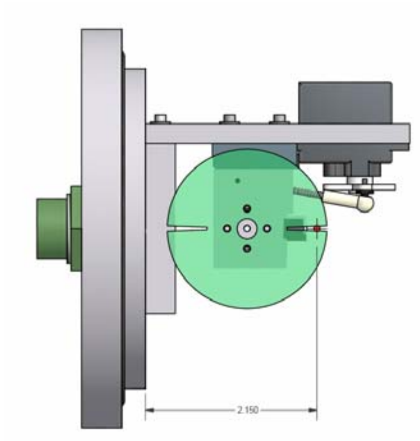


Figure 37: Servo in Beam Chop Position

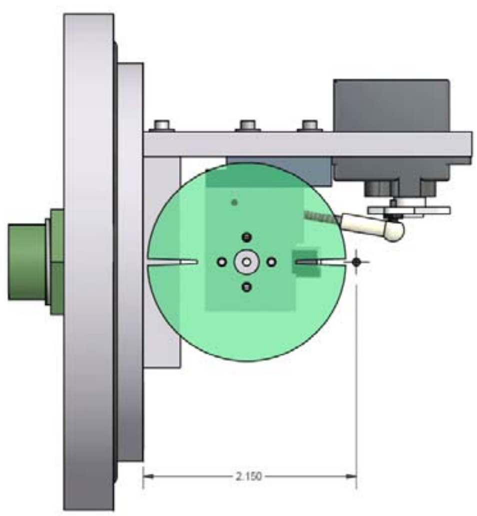


Figure 38: Servo in Beam Open Position

Multiplier replacement

The electron multiplier on the quadrupole should be considered an expendable item and it is recommended to carry a spare multiplier. The useful life of the multiplier will range from several months to several years depending on its use. Since the multiplier is operated at high gains ($>10^6$) and large signals are monitored (up to 10^7 Hz, the air beam) the useful life is rather short. Replacing the multiplier requires that the vacuum system be shut down and vented. The recommended replacement multiplier is the ETP Model AF140, although the Balzers SEV217 can also be used. The ETP is recommended since it has a much longer shelf life than the Balzers, due to different active surface technologies. Discussed here is the replacement of the ETP multiplier. The multiplier is mounted on a 6" conflat (copper gasket style) flange. Before starting this procedure be sure to have a new replacement copper gasket for the 6" flange.

After venting, remove the eight 5/16 diameter bolts holding the 6" conflat multiplier flange in place. Carefully remove the conflat flange. Note the orientation of the alignment stud on the mating flange. The replacement ETP multiplier will have two signal leads; one of them will need to be cut/removed as indicated in the figure. The AMS uses lead A; therefore, cut lead B.

Multiplier housing on
6” conflat flange

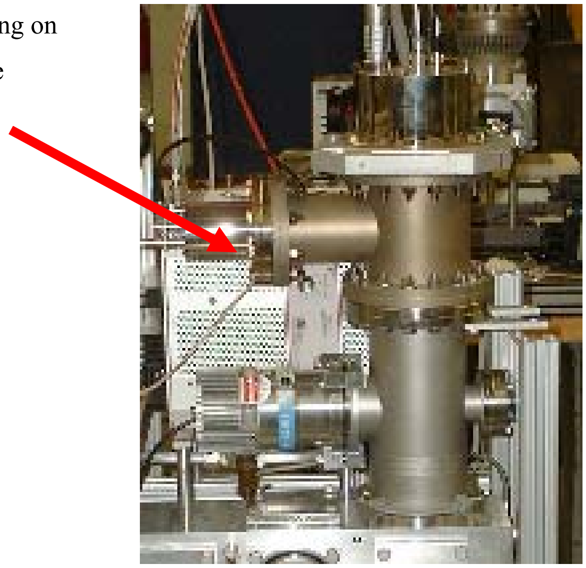


Figure 39: Electron Multiplier Replacement

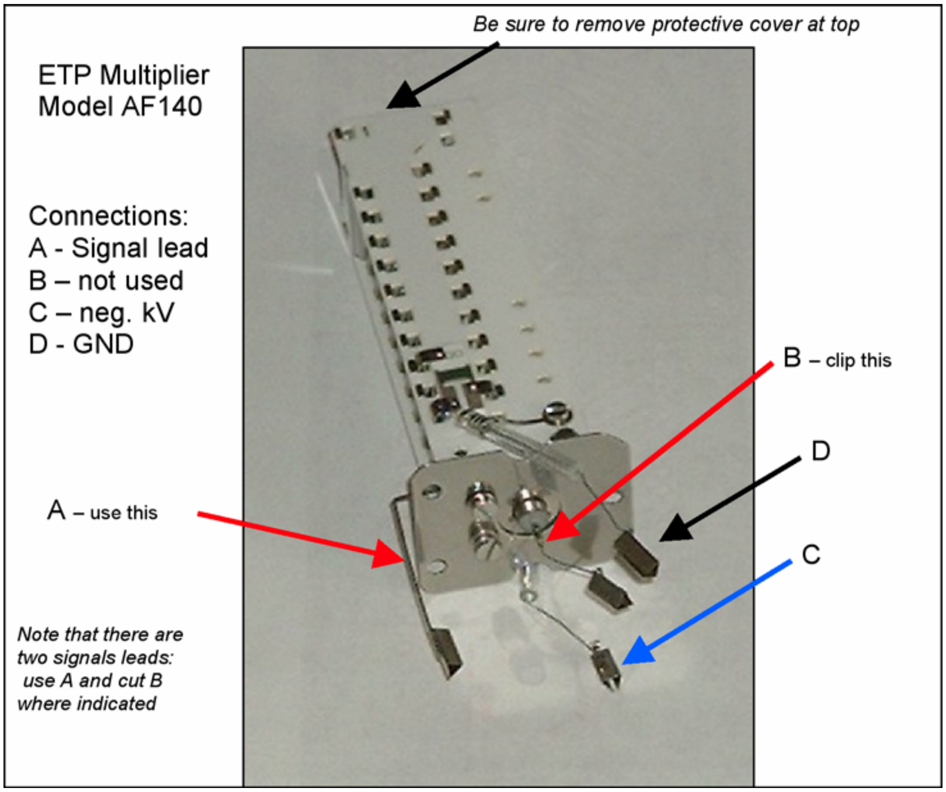


Figure 40: Electron Multiplier Replacement

Electron multiplier replacement. ETP multiplier shown.

Troubleshooting/Diagnostics

No ion signal.

POSSIBLE CAUSE	RECOMMENDED ACTION
Filament failure.	• Look through rear window – is filament lighted? • If not, switch to alternate filament via QMG422 interface.
Multiplier voltage not present.	• Check computer manual switch on Electronics Box. • Is computer providing command voltage? Check DAC1 from NI 6024E (slow) board.
Preamplifier problem	• Increase range switch on preamp from 1 x 10^{-6} A/V. If there is a signal response, preamp is probably OK. • Is the vaporizer temperature display correct? (This circuit shares preamp power supply). If not, there maybe a problem with preamp power supply. Check +/-15V to preamp on DB9 cable [P7 (GND), P8 (-15V), P3 (+15V)]. • Terminate preamp input and measure output voltage. If > 1 – 2 volts, preamp is bad. Try terminating multiplier output with a ~10K resistor by bypassing the preamp, then looking for the ion signal.
Vacuum Interlock Protect circuit activated.	• Ensure that turbo pump P6 and all other pumps are at full speed.
National Instrument cables (and/or boards) not properly seated.	• Disconnect and re-connect NI cables at computer and BNC board.

Troubleshooting, continued….

Low ion signal.

POSSIBLE CAUSE:	RECOMMENDED ACTION/COMMENT:
Deflection voltages are wrong or not present.	• Verify that all voltages are being sourced from Balzers IS420 ionizer module. • Remove chrome cover on quadrupole feed-thru flange, check and reference voltages on pins to Figure 41: Balzers Cross-Beam Ionizer Voltage Table on page 94.

No variation in ion signals during quadrupole auto-tune procedure.

POSSIBLE CAUSE	RECOMMENDED ACTION/COMMENT
Serial interface is not working if "shift-B" window does not display ACK when an ionizer command is issued.	• Check serial cable connection from computer to QMG422. • Verify that the DB9 cable is "null-modem" type. • Check that QMG422 baud rate is set to 19200. This baud rate is hard-coded in the acquisition program. • Check that serial cable is connected to the COM port selected in acquisition program.
Serial interface OK, but quad auto tune does not adjust vaporizer bias voltage.	• Check to see that Comp/Man switch is set to Comp on front of EC box.
Serial interface OK but still no variation of ion signal.	• Remove chrome cover on quad feed-thru flange, check and reference voltages on pins to Figure 41, page 94.

Troubleshooting, continued….

Chopper wheel not spinning.

POSSIBLE CAUSE	RECOMMENDED ACTION/COMMENT
Switch is in "computer" position.	• Put switch in manual position.
Voltage that drives chopper motor.	• Remove the 10-pin circular connector on chopper cable at the chopper flange. Measure between pin B (GND) and pin A. You should observe a voltage of ~ .8V that varies with "Chopper Speed" trim pot adjustments. Verify presence of 12V signal between pin B (GND) and pin C. If both voltages are present and motor is not spinning, the motor may have failed.
Chopper servo travel out of range, jamming chopper wheel on chopper flange and preventing wheel from rotating freely.	• Look through Lexan flange for evidence of chopper wheel touching chopper flange.

Chopper servo not moving.

POSSIBLE CAUSE:	RECOMMENDED ACTION/COMMENT:
Software selection of NI board used to drive servo not selected/is wrong.	• Check which board is selected to control chopper servo, both in the software and on the BNC termination panel.
Cabling and software selection of board used to control servo is OK but still no servo motion.	• Monitor output of CTR0 (the servo drive signal) from BNC2110 or BNC2090 board on an oscilloscope and run program in MS-Toggle mode. You should observe a 0-5V(TTL) pulse at 50Hz with a changing duty cycle as program switches between "beam open" and "beam closed." Pulse width should be ~ 0.8 to 2 ms. • On chopper circular connector at chopper flange, pin B to pin E should be 3.2-5V (servo power) and pins B to H should be the variable duty cycle pulse to position servo.

Troubleshooting, continued….

No MS signal.

POSSIBLE CAUSE	RECOMMENDED ACTION/COMMENT
Filament and/or multiplier shut off.	• Check vacuum interlock and status of pumps #4, 5 & 6. • Verify that the multiplier switch is in "computer" setting. Alternatively, place multiplier switch in manual setting. • Check the orange LED on the RF box. If it is not flickering, there is no mass ramp. Use a scope to check signal from DAC0 on the fast board (NI6110E0). • Increase gain on preamplifier. If this causes increased noise on the computer display, the preamp and data board are most likely operating. If not, check to make sure the preamp is getting power. Refer to Preamplifier Problem, page 88.

Filament does not light.

POSSIBLE CAUSE	RECOMMENDED ACTION/COMMENT
Pump speed, poor cable connection or broken filament.	• Check status of vacuum interlock. Pump #5 should be at 100% speed. • Make certain that the interlock cable between Electronics Box and PCB is connected. • Check filament continuity, refer to Figure 41.

Troubleshooting, continued….

Filament does not stay lit, turns on but shuts off.

POSSIBLE CAUSE	RECOMMENDED ACTION/COMMENT
Pressure too high	• Check status of turbo pumps: power and speed
Filament Protect level set too low.	• On QMG422, press EMISSION then select E-PROT and set a few tenths of an amp higher.

No or low Air Beam signal.

POSSIBLE CAUSE	RECOMMENDED ACTION/COMMENT
Pinhole disc clogged.	• Check lens pressure. Disable MS Toggle Mode and verify that there is a mass spectrum. If spectrum is present, investigate chopper movement. If no mass spectrum, see No MS signal., page 92.

Air beam decreasing but ionization efficiency is OK.

POSSIBLE CAUSE	RECOMMENDED ACTION/COMMENT
Pressure in TOF chamber (pump #3) is high	• Check for leaks in TOF chamber. • Check status of turbo pump #3: power and speed

ID	Color	Connection	V ID Set volt.	Measured Voltage
0	Black	Deflection Inner	V6 226 (/10)	-22.5
1	Brown	Ion Reference	V1 78	-77.9 (to ground)
2	Red	Extraction	V5 238	-237
3	Orange	Filament #2	---	-68.1
4	Yellow	Filament #1	---	-69.6
5	Green	Filament (common)	V2	-68.2
6	Blue	Focus	V3 11.25	-11.2
7	Purple	Deflection Outer	50 (/5)	-9.9
8	White	Field Axis	V4 12.5	-12.4

All voltages are measured relative to Ion Ref (VI), except where noted. Filament #1 is on at 0.25 mA (actually, 2.5 mA is what it is). Filament #2 is off.

Figure 41: Balzers Cross-Beam Ionizer Voltage Table

Safety

The ARI Aerosol Mass Spectrometer should be operated using generally accepted safety precautions. The instrument should be plugged in to a standard AC outlet (115 – 240 VAC) for power, and appropriate precautions should be taken when working with any AC powered mains voltage. The AMS also uses high voltage (,4kV) for both the electron multiplier and voltage on the quadrupole rods. Care should be taken when measuring voltages at either the quadrupole flange (see Figure 41: Balzers Cross-Beam Ionizer Voltage Table) or inside the AMS electronics box where the multiplier voltage originates. The instrument should be protected from water, and the instrument should be operated only by those trained in its operation. Operators are advised to wear protective eyewear, to be familiar with caution or warning symbols on the equipment, and to be familiar with the guidelines for safe use of Class III lasers if the AMS has a light-scattering module (see http://www.osha-slc.gov/dts/osta/otm/otm_iii/otm_iii_6.html#4).

Emergency shut-down procedures are described on page 38. The instrument should be used only for the purpose for which it is intended, including attention to the specific model that is appropriate for a particular location or application. For example, only certain models are designed to be used in mobile applications, such as on a boat, truck or airplane. ARI personnel are available for consultation on safety issues by calling 978-663-9500.

Glossary

AB – Air Beam.

Aerodynamic diameter – the product of geometric diameter and density.

amu – Atomic mass units.

CPC – Condensation particle counter.

Data Diagnostics – Plots important amu ratios that are used to optimize fragmentation waves for accurate mass concentration calculations.

Delta Analysis – Analyzes the organic components of the measured mass spectra

The program allows the user to highlight various delta groups within a mass spectrum.

The user can calculate the delta pattern for any range of carbon numbers. The program generates curves for the delta signals as a function of carbon number, allowing for quick and easy identification of discrepancies within the delta patterns.

DMA – Differential mobility analyzer.

EM – Electron Multiplier.

F – Female connector.

Fragmentation waves – Mechanisms for interpreting mass spectra.

FWHM – Full width at half maximum intensity (of Gaussian curve).

GND – Electrical ground.

IE – Ionization Efficiency.

QMS – Quadrupole Mass Spectrometer.

Hysteresis – The lagging of an effect behind its cause, as when the change in magnetism of a body lags behind changes in the magnetic field. (*American Heritage Dictionary of the English Language*, 4th ed., 2000).

IPP – Ions per particle.

M – Male connector.

NIST – National Institute of Standards and Technology.

PSL – Polystyrene latex spheres.

Servo – A small device with a positionable output shaft, used in robotics.

TOF – Time-of-Flight.

TTL – Transistor-Transistor Logic.

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