Field Optimization of Ion Trap Performance

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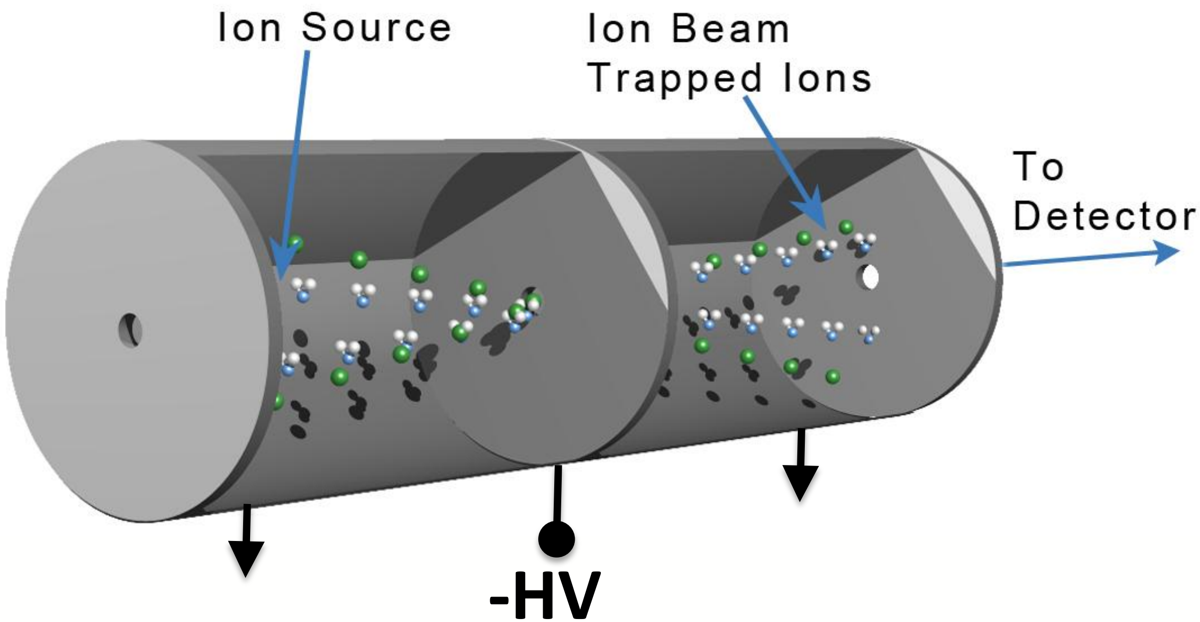
Outline – Autoresonant Ion Trap Mass Spectrometry (ART MS)

1. Electrostatic Ion Trap
2. AutoResonant Ion Ejection
3. ART MS Advantages for Field Work
4. Unit-to-Unit Variability
5. Instrument Characterization and Quality Control
6. Electron Coupling Efficiency (ECE)
7. Initial Potential Energy Distribution of ions (IPED)
8. Excited Potential Energy Distribution of ions (EPED)
9. Field tuning of ART MS sensors
10. Autotune Procedure (software)

Ion Storage - Electrostatic Ion Trap

A simple electrode structure is devised from two grounded metal cups and a central plate biased at negative high voltage (-HV).



1. Ions are created inside the cylindrical volume by electron ionization.
2. A purely electrostatic potential well confines the ion trajectories inside the electrode structure in oscillatory motion.
3. The ion trap operates at saturated charge capacity.

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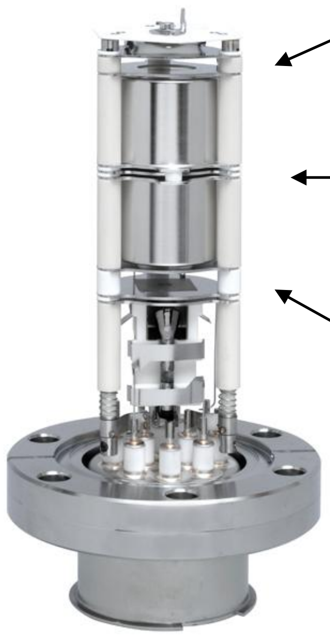
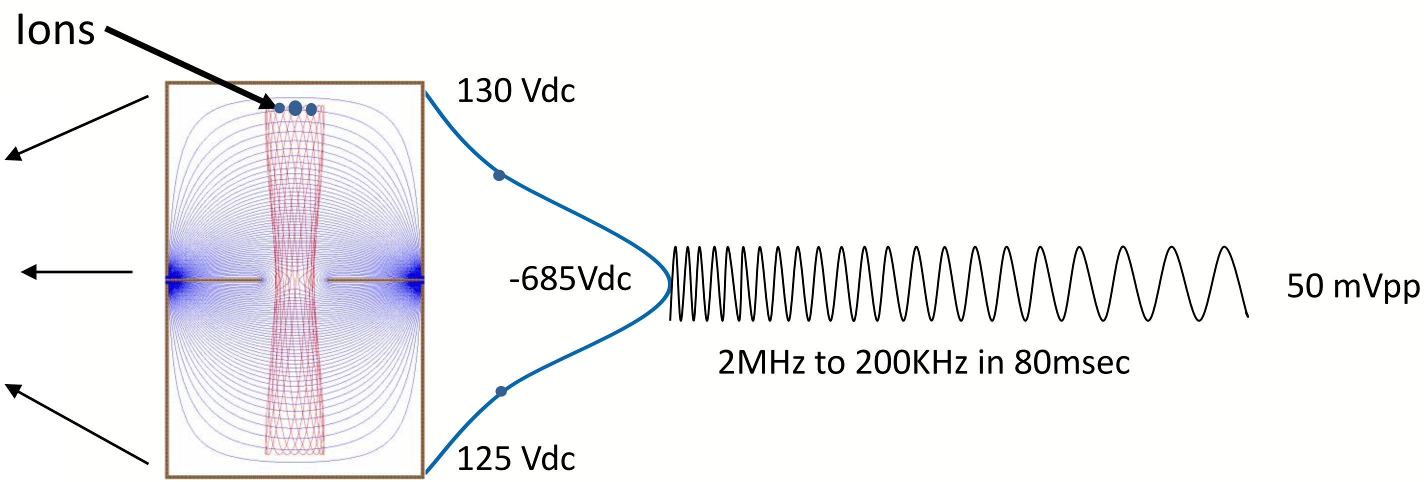
Ion Ejection - Autoresonance



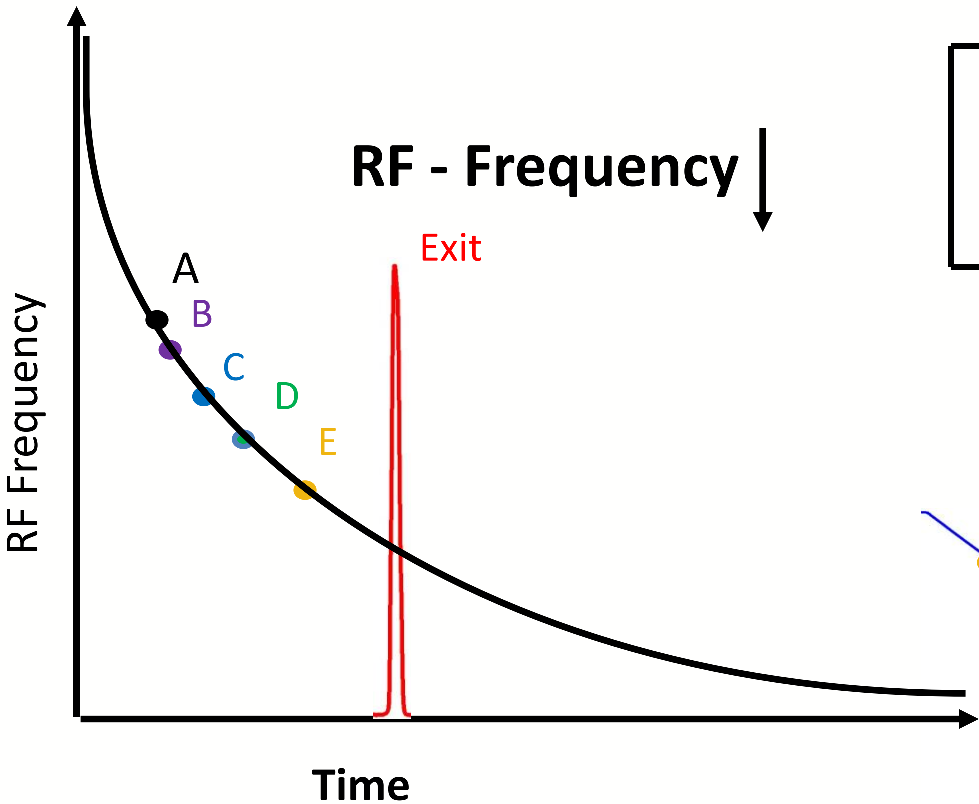


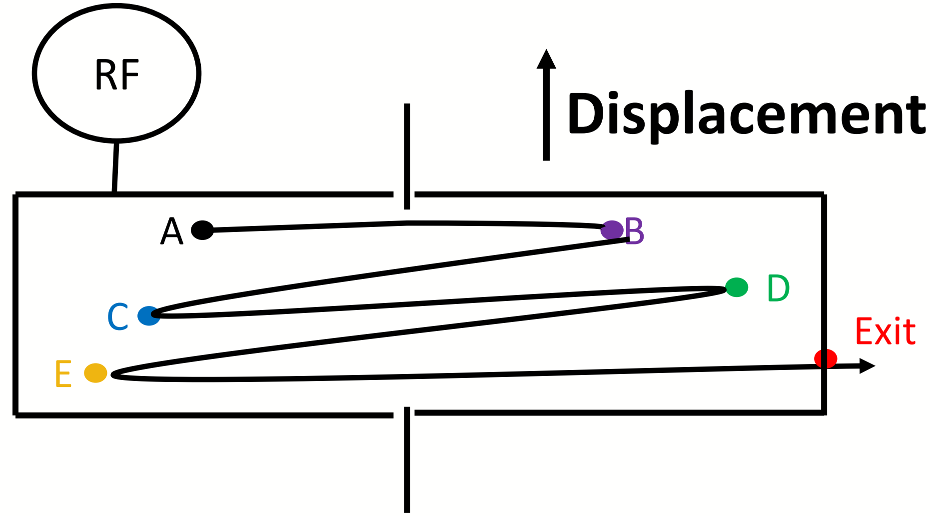
Electrostatic Ion Trap: Ions confined by purely electrostatic fields oscillate at a resonant frequency inversely proportional to $\sqrt{m/z}$

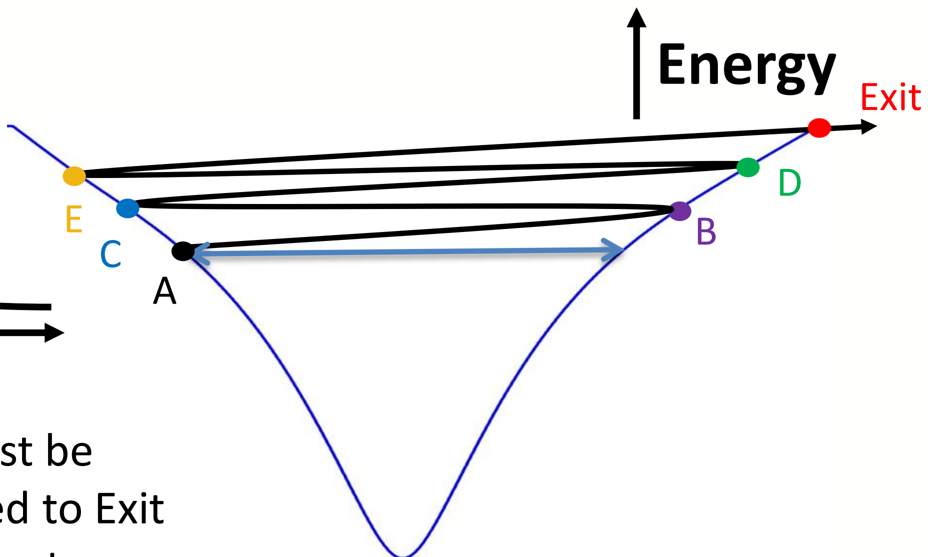
Where, m is mass, z is the total charge of the ion

Autoresonance: RF frequency scan pushes ions when scan frequency matches ion's resonant frequency

Displacement / Energy / Frequency Triangle







RF Amplitude and Frequency Sweep Rate must be carefully controlled so that all ions are allowed to Exit the trap – Mass dependent frequency sweep rate = mass independent ejection efficiency.

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ART MS: Well Suited for Field Work

Low Power	**<7.5W**	**Electrostatic ion storage and small amplitude RF excitation (c.a. 50mV)**
Small Size	Scalable	Trap scalable to very small volumes. Single Board Implementation.
Light Weight	Scalable	Assembly scalable to just a few parts
Relaxed Mechanical Tolerance	Low precision	Compatible with shaky conditions
Remote Interconnect	Up to 20 meter cable (50 meter tested)	Sensor can operate far away from the electronics. **No sensor-electronics matching**
Fast Measurement	<100msec scan time	Fast single scans allow detection from fast moving platforms
Unlimited Mass Range	Tested to 600 amu	If you can vaporize it then you can measure it
Simple Interface	USB and Analog out	Compatible with simple data acquisition setups
Field Calibration and Tuning	**Single mass-axis calibration**	**Autotune procedure**

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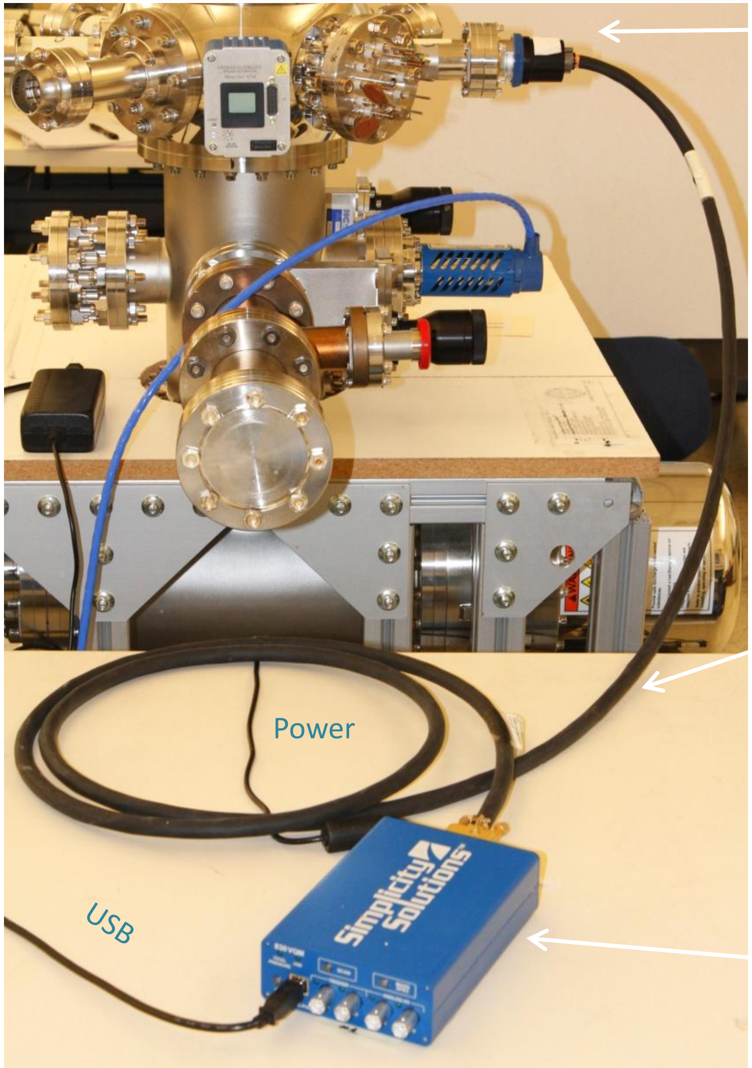
Remote Interconnect Advantage



ART MS Gauge

Interconnect cable (0.37m, 1m and 3m std., 20 meter optional)

Controller (Single Board)

- No RF matching required between gauge and controller.
- One controller can operate multiple units – i.e. economic advantage.
- The controller can be located far away from the gauge- protect electronics & personnel from harsh environments.

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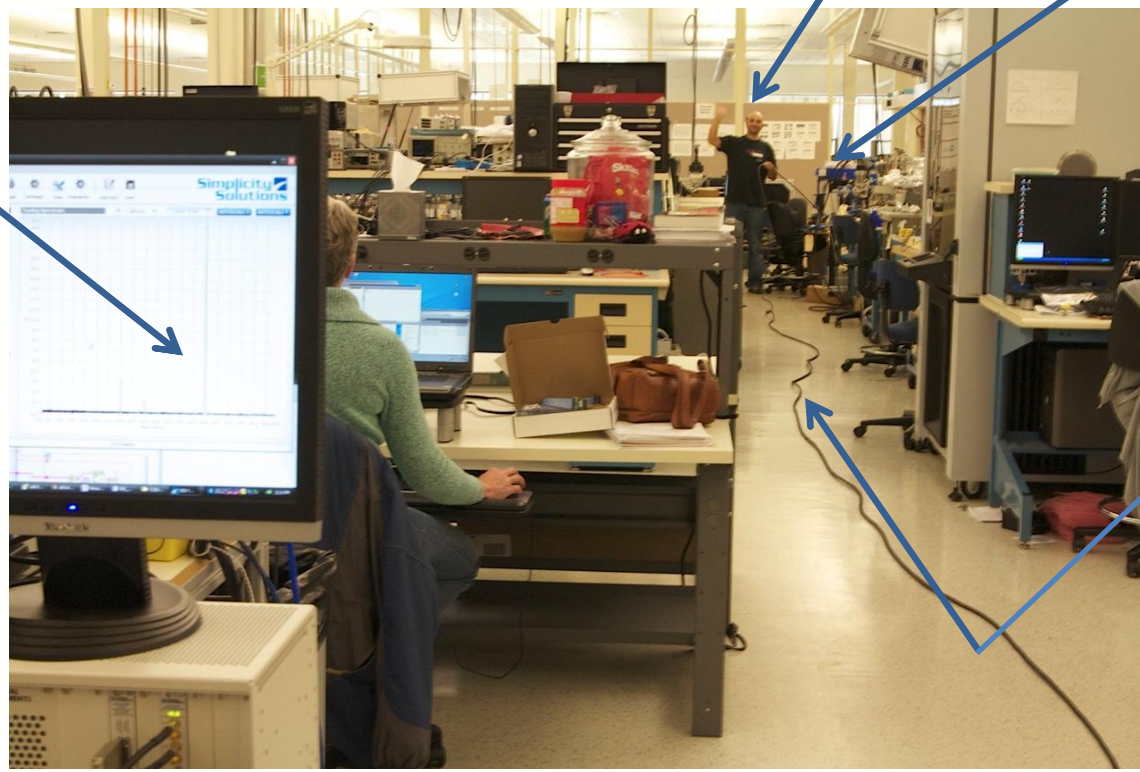
20 meter Interconnect Cable

Remote VQM Controller & Computer

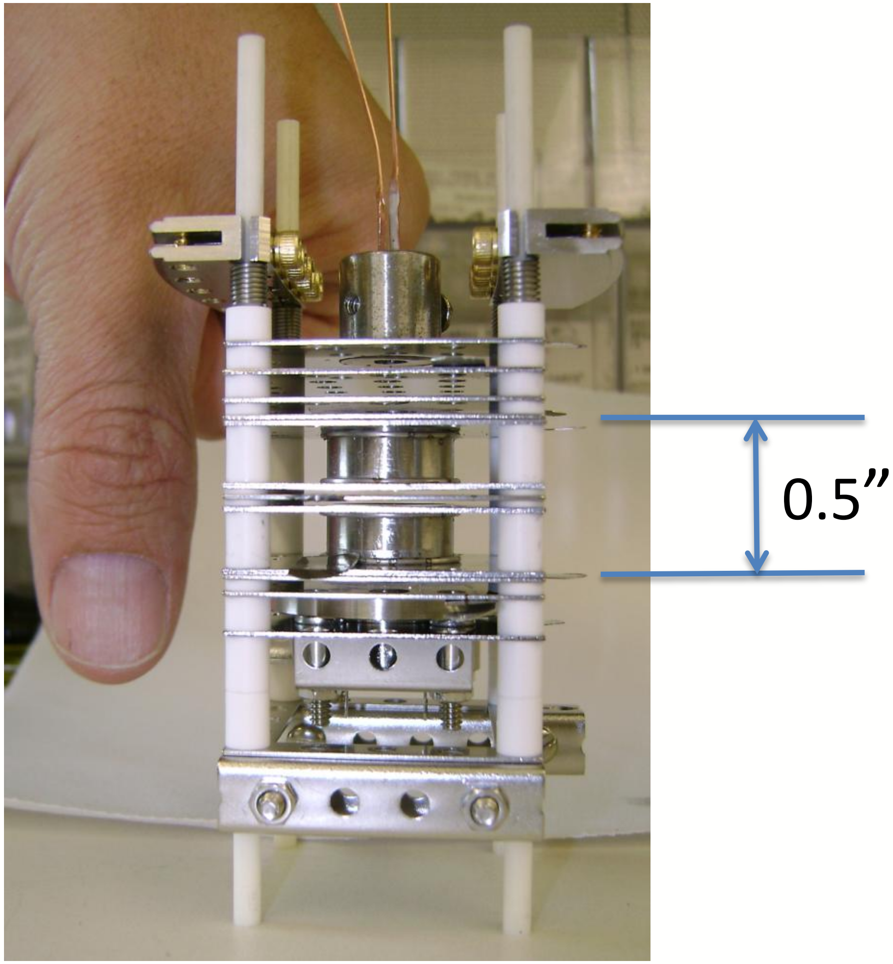
Jeff

VQM Gauge



20 m cable

Small Dimensions



Prototype built in our Lab

Resolving power = 60X

Low mechanical tolerance requirements. Most prototypes built out of eV Parts, Kimball Physics.

Manufacturing Specifications and QC

Each ART MS instrument manufactured at Granville-Phillips is subjected to a rigorous manufacturing process which includes:

Step 1- Verification Mechanical Assembly

Step 2- Verification of Instrument Specifications.

Specifications for each mass spectrometer include:

Resolving Power
Sensitivity
Dynamic Range
Peak Ratios

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Unit-to-Unit Variability

- It was realized early on that, even under strict mechanical controls, there are unit-to-unit variations in spectrometer performance (factory)
- Variations in instrument perfrormance are also experienced after Filament Assembly replacements (field).
- An investigation was launched to understand root cause for unit-to-unit variations and to develop a process that provides customers with consistent product performance.
- Investigation led to:

Under-the-Hood Performance Test Procedure (factory)

AutoTune Procedure (field)

Under-the-Hood Performance Tests (Factory)

In order to fully characterize an ion trap and provide a consistent product it became necessary to break down the ion trap functions into its basic components:

How many ions are formed inside the trap (ECET)?
What is the initial energy of the ions stored in the trap (IPED)?
How many ions are stored inside the trap (RFOT)?
Can the ions gain enough energy from the RF Signal (EPED)?
Are enough ions being ejected from the trap (RFOT)?
Does the detector have enough sensitivity (EMVT)?

Specification tests are only initiated if Performance tests are passed!

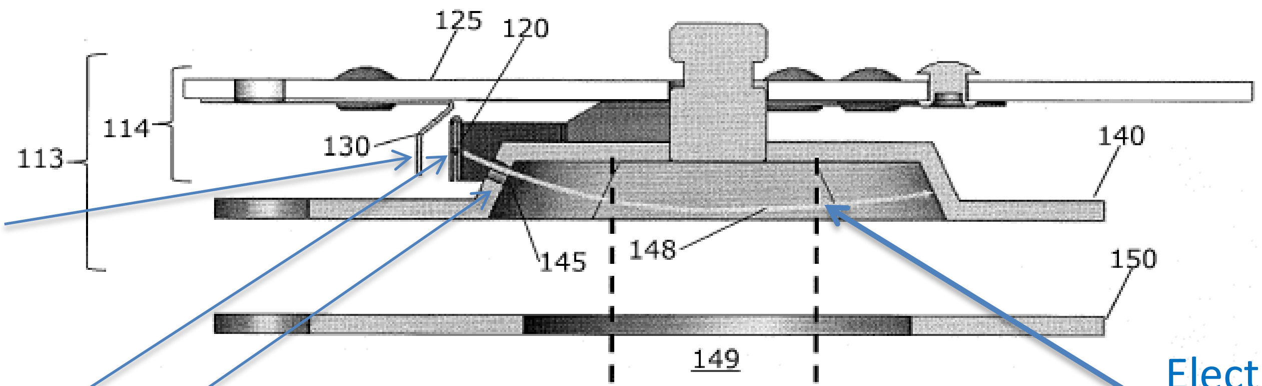
How many Ions are made inside the trap?

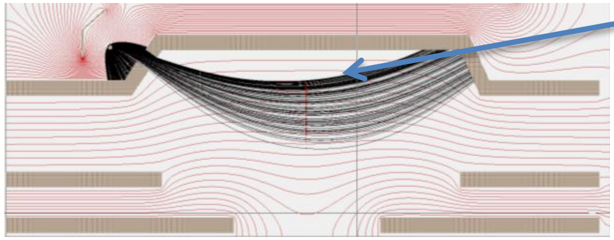
- Every spectrometer built at the factory must include an ionizer that makes enough ions.
- A low rate of ion production translates into problems related to:
  1. Ionizer alignment
  2. Yttria coating quality
  3. Spot welds quality
  4. Dynamic range predictor

- Ion formation is tested through the: Electron Coupling Efficiency Test (ECET) performed at the factory for each unit.

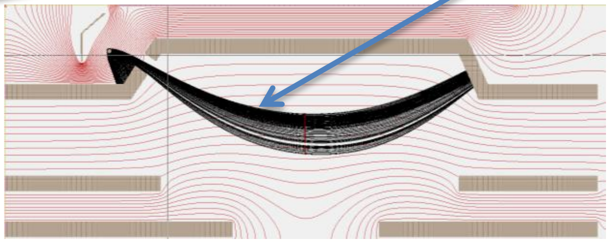
Off-Axis Ionization Source

Repeller Plate controls the coupling of the electron bean through the entry slit and into the ionization region.





- Poorly focused electron beam.
- ECE is low.
- Ions are spread out over large area.



- Properly focused electron beam: V-Repel = ECE_Max.
- ECE is acceptable.
- Ions are spread over a narrower area – leads to more defined initial conditions.

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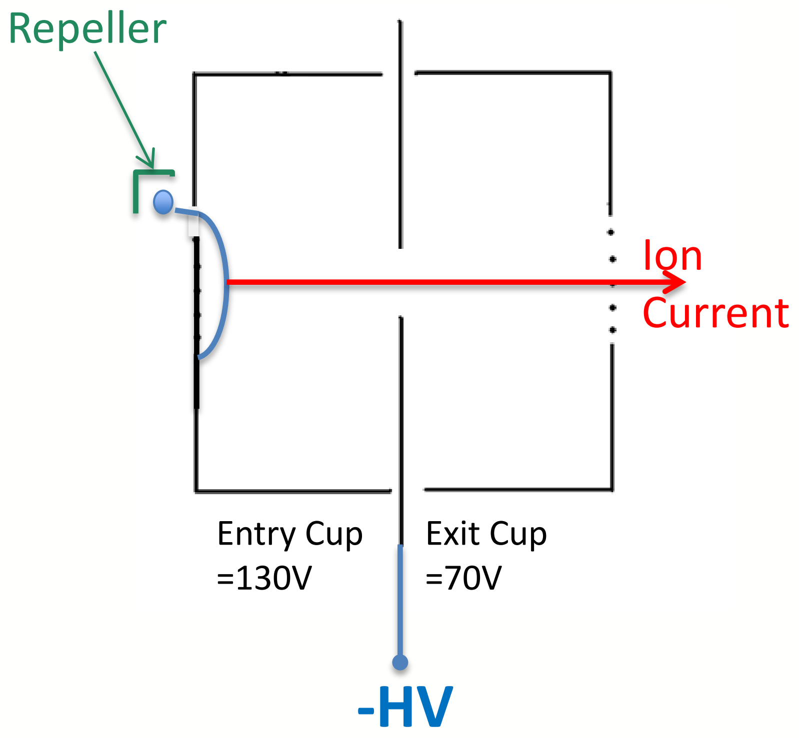
Electron Coupling Efficiency (ECE) Tuning

The repeller plate bias can be Tuned in the field to provide optimal electron coupling efficiency (ECE)

ECE Tuning finds the repeller plate bias voltage that provides the optimal coupling of electrons from the filament through the entry slit into the ionization region:

1. The exit plate bias is lowered so that all ions formed inside the trap are ejected through the exit grid
2. The Ion Current ejected from the trap is measured as a function of repeller bias voltage.



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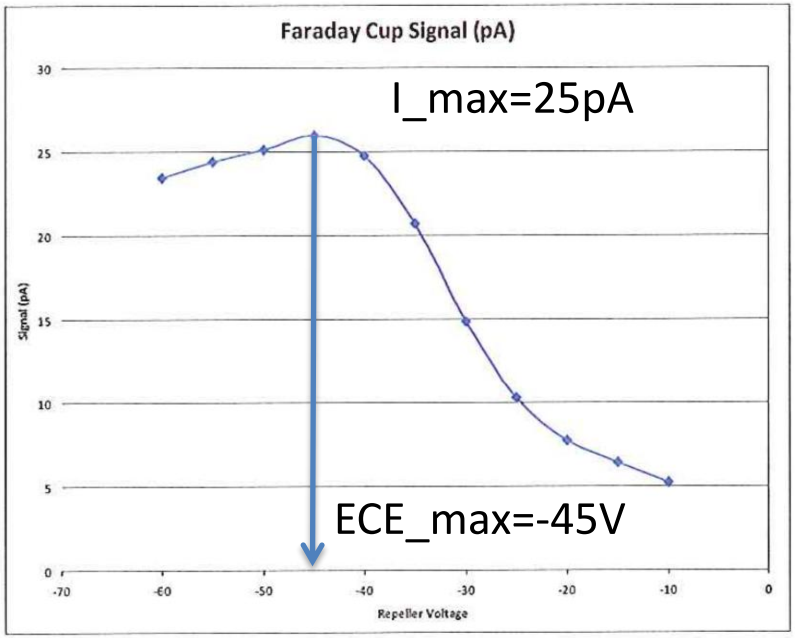
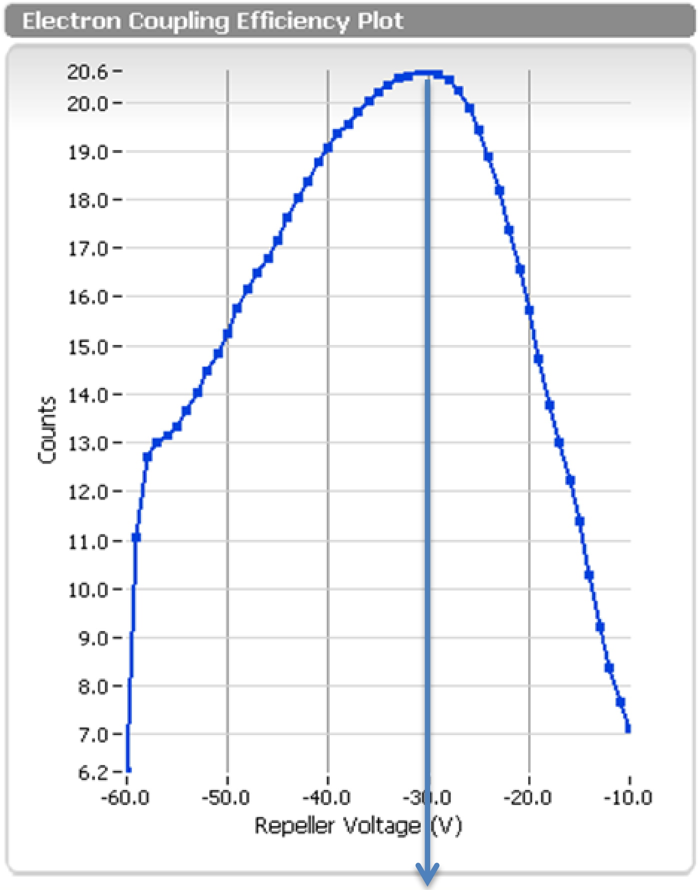
Electron Coupling Efficiency Test

1. Trap is operated as Extractor Total Pressure Gauge.
2. Pressure is set to 2E-7 Torr of pure Nitrogen
3. Ion Current is measured as a function of Repeller Voltage
   - ECE_max must be between -10 and -60 V
   - I_max must be between 20 and 30 pA.



ECE Tuning Output



ECE_Max = -30V

- The Repeller Bias is set at ECE_Max
- Users can repeat this procedure in the lab or out in the field at any time.

Initial Energy of ions stored inside the trap?

Every Instrument manufactured must make/confine ions in the proper energy range.

Ions produced with the wrong initial energies translate into problems related to:

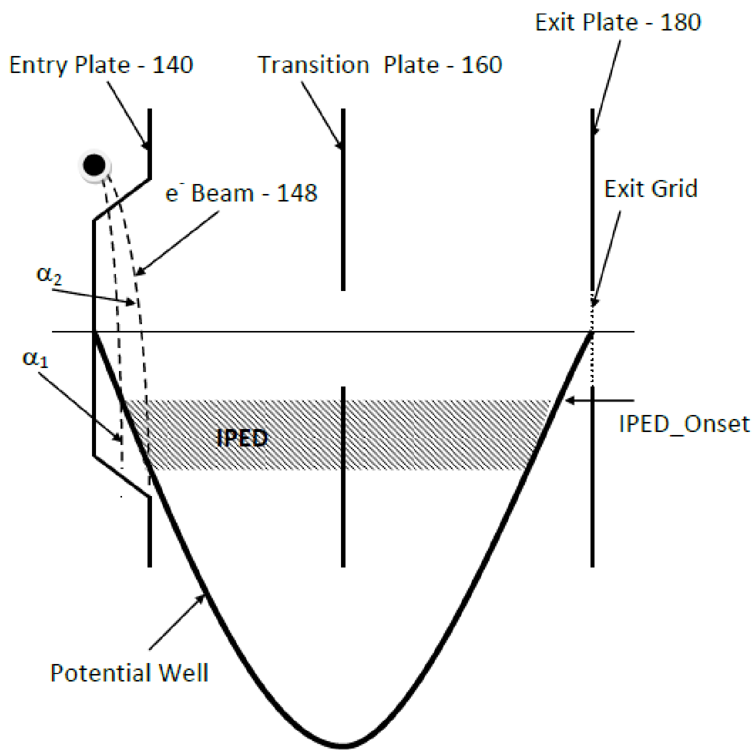
- Repeller-Filament-Slit alignment
- Resolution predictor
- Trap parameter bias setting.

Initial Ion Energy is tested with the Initial Potential Energy Distribution Test (IPEDT)

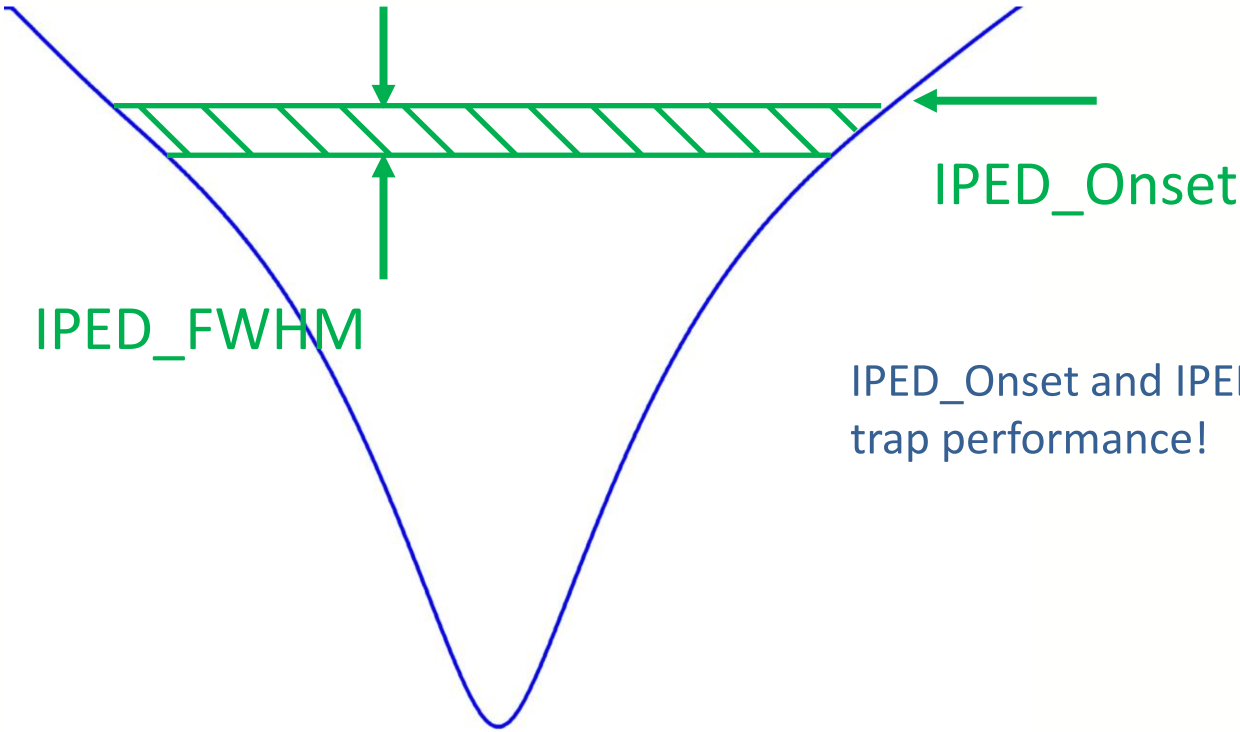
Ion Energetics in Off- Axis Ionization Source

- Off-axis Electron Ionization also defines the Initial energetics of the stored ions.

- The initial energetics of the ions defines the performance specifications of the spectrometer.



Initial Potential Energy Distribution



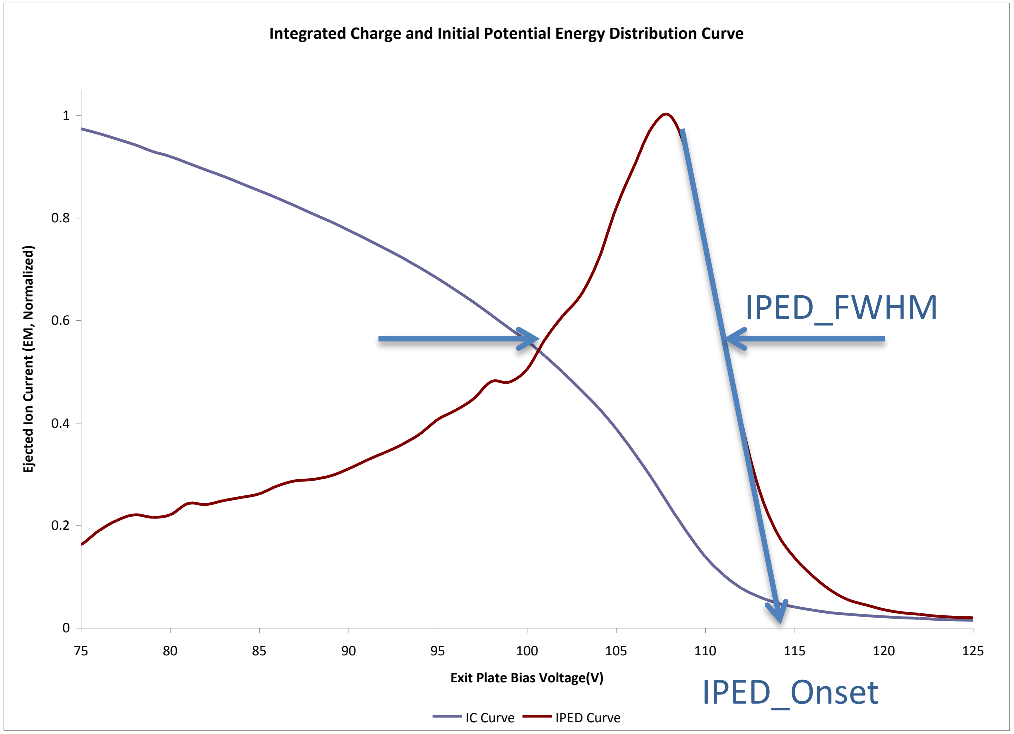
IPED_Onset and IPED_FWHM affect ion trap performance!

IPED Test

Initial Potential Energy Distribution Test (IPEDT):

1. Initial Potential energy is measured and plotted.

2. The Highest Potential Energy (IPED_Onset) and the Energy spread (IPED_FWHM) are measured.



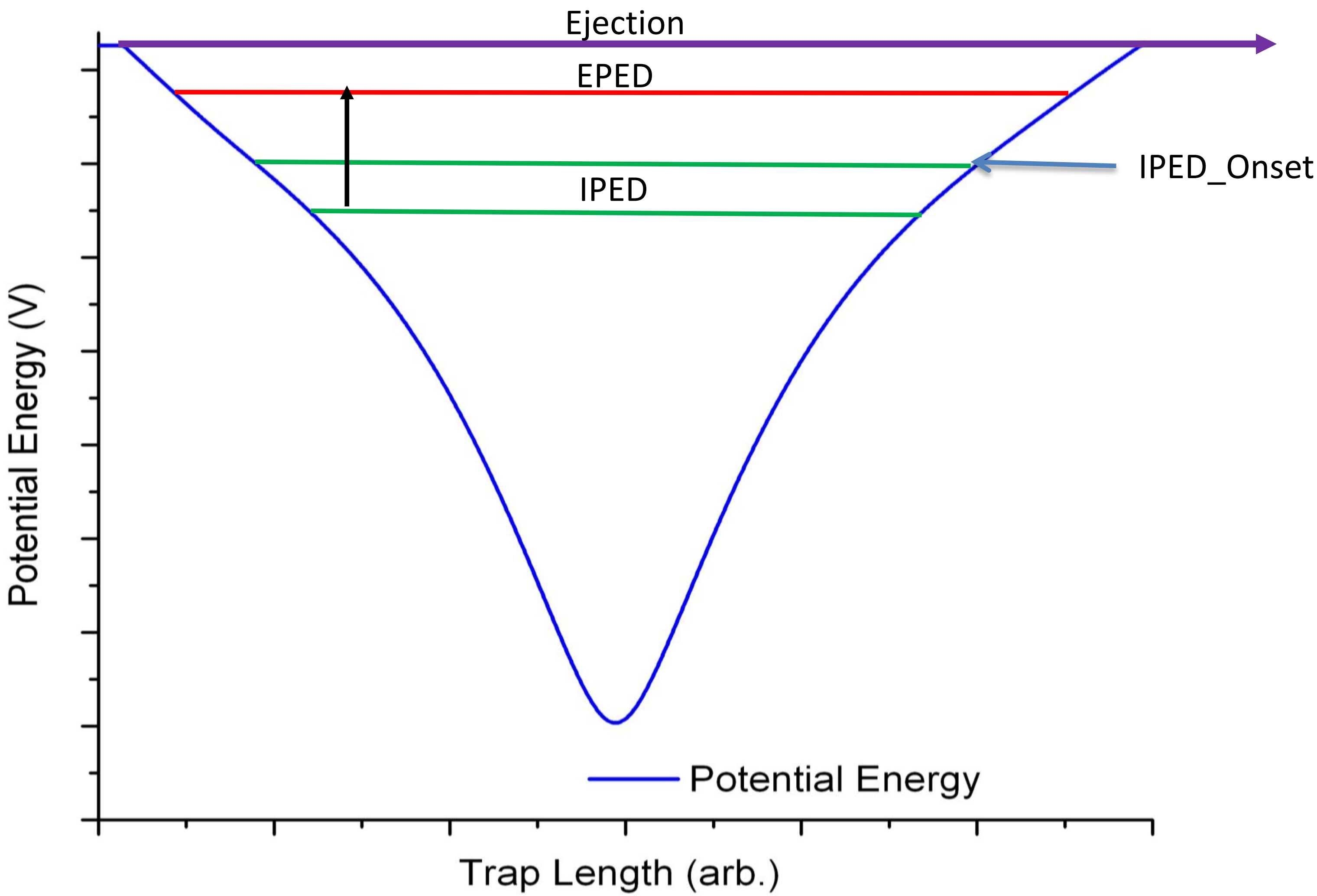
Do the stored ions Gain enough Energy from the RF Signal?

- Every Instrument manufactured must consistently energize the confined ions.
- Failure to properly energize the ions translates into problems with:

  1. RF delivery
  2. Cable quality issues
  3. Spot welds
  4. Electrode Stack up.
  5. Resolution and Dynamic range predictor

- The test developed to test the energy gain is the Excited Potential Energy Distribution Test (EPEDT)
- The trap parameters are adjusted based on the results of this test.

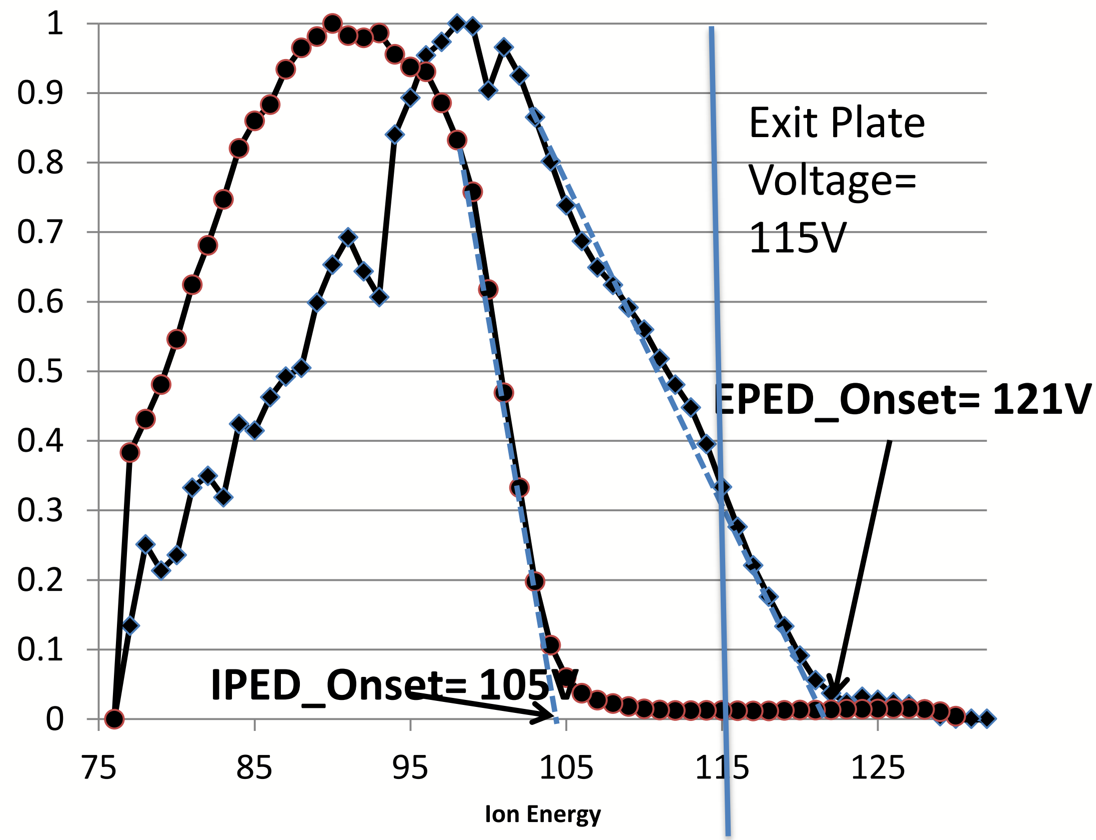
Excited Potential Energy Distribution



EPED Test

Excited Potential Energy Distribution Test (EPEDT):

1. Excited Potential energy is measured and plotted.
2. The Highest Potential Energy (EPED_Onset) and the Energy spread are measured.
3. Typical energy gains are >16V



EPED_Onset – IPED_Onset= 16V

Exit plate voltage is set to: IPED_Onset +10V

EPEDT – RF dependence

As expected the amount of energy gained by the ions during the RF sweep depends on the RF amplitude.

The exit plate potential needs to be adjusted if the RF Signal is changed.

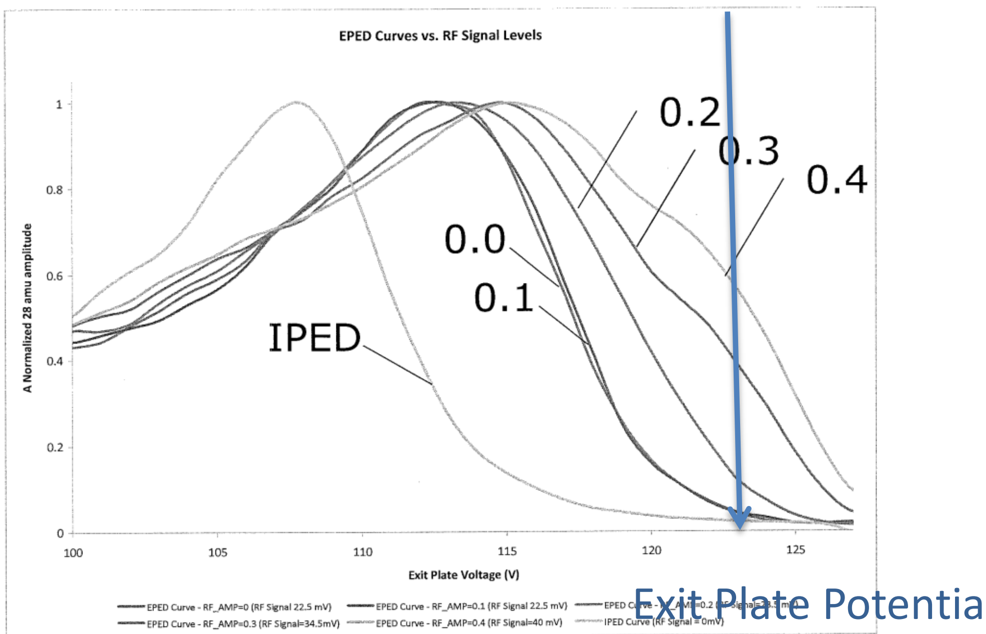


FIG. 17

Under-the-Hood Testing

- A series of tests are performed at the factory that break down the performance of the trap into its basic constituents.
- If the tests are passed the unit is tuned (trap settings adjusted) and tested against its specification requirements.
- If one or more of the tests fails then the unit is fixed based on the test information and returned to the production floor for retesting.
- Under-the-hood performance testing provides a consistent product offering and higher product yields.

Autotune Procedure

The methodology developed for Under-the-Hood Performance testing can be used in the lab or out in the field to optimize ion trap performance.

The same procedure was also incorporated into the software to provide Autotuning in case performance changes are seen with time or after a filament replacement.

The software automatically checks ion trap performance and adjusts that ion trap settings accordingly to restore full operation according to instrument specifications.

The software also reports any major problems that might be encountered.

Autotune Procedure (Field)

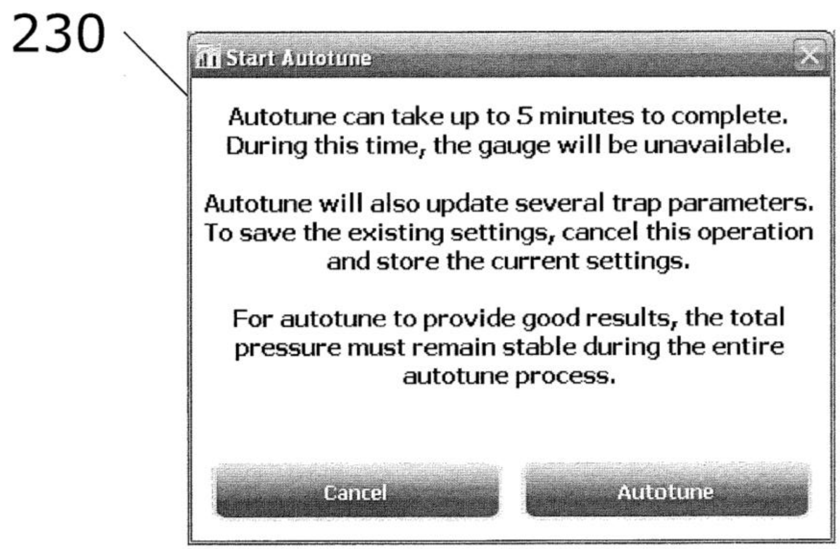


FIG. 2B

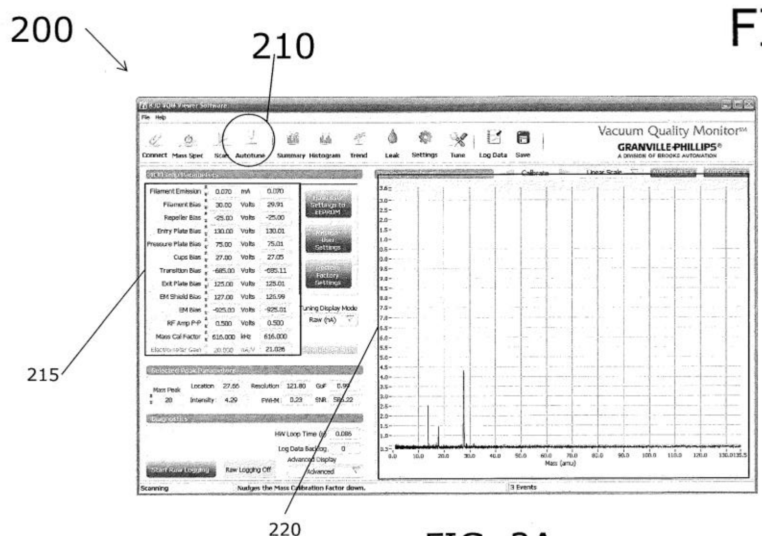


FIG. 2A

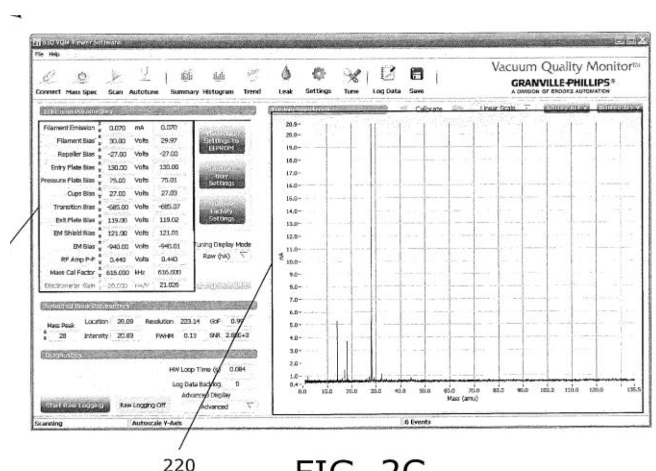


FIG. 2C

Conclusions

- A simple set of performance tests allows to evaluate ion trap performance prior to specification testing.
- Under-the-hood performance testing allows production of a very consistent product offering.
- Autotune procedure enables users to perform the same performance tests in the field and to adjust instrument settings to compensate against drift and filament replacement processes.
- Continuous product improvement depends on better understanding of ion trap principles of operation. Continuous learning process!

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Additional Information

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1. (WO2013066881) METHOD AND APPARATUS FOR TUNING AN ELECTROSTATIC ION TRAP

PCT Biblio. Data | Description | Claims | National Phase | Notices | Drawings | Documents

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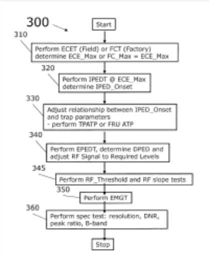
61/719,668 29.10.2012 US

Title
(EN) METHOD AND APPARATUS FOR TUNING AN ELECTROSTATIC ION TRAP
(FR) PROCÉDÉ ET APPAREIL POUR ACCORDER UN PIÈGE À IONS ÉLECTROSTATIQUE

Abstract:
(EN) An apparatus includes an electrostatic ion trap and electronics configured to measure parameters of the ion trap and configured to adjust ion trap settings based on the measured parameters. A method of tuning the electrostatic ion trap includes, under automatic electronic control, measuring parameters of the ion trap and adjusting ion trap settings based on the measured parameters.
(FR) La présente invention porte sur un appareil qui comprend un piège à ions électrostatique et des électroniques configurés pour mesurer des paramètres du piège à ions et configurés pour ajuster des réglages de piège à ions sur la base des paramètres mesurés. Un procédé d'accord du piège à ions électrostatique comprend, sous une commande électronique automatique, la mesure de paramètres du piège à ions et l'ajustement de réglages de piège à ions sur la base des paramètres mesurés.



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