TRACE DSQ

Automatic Tune

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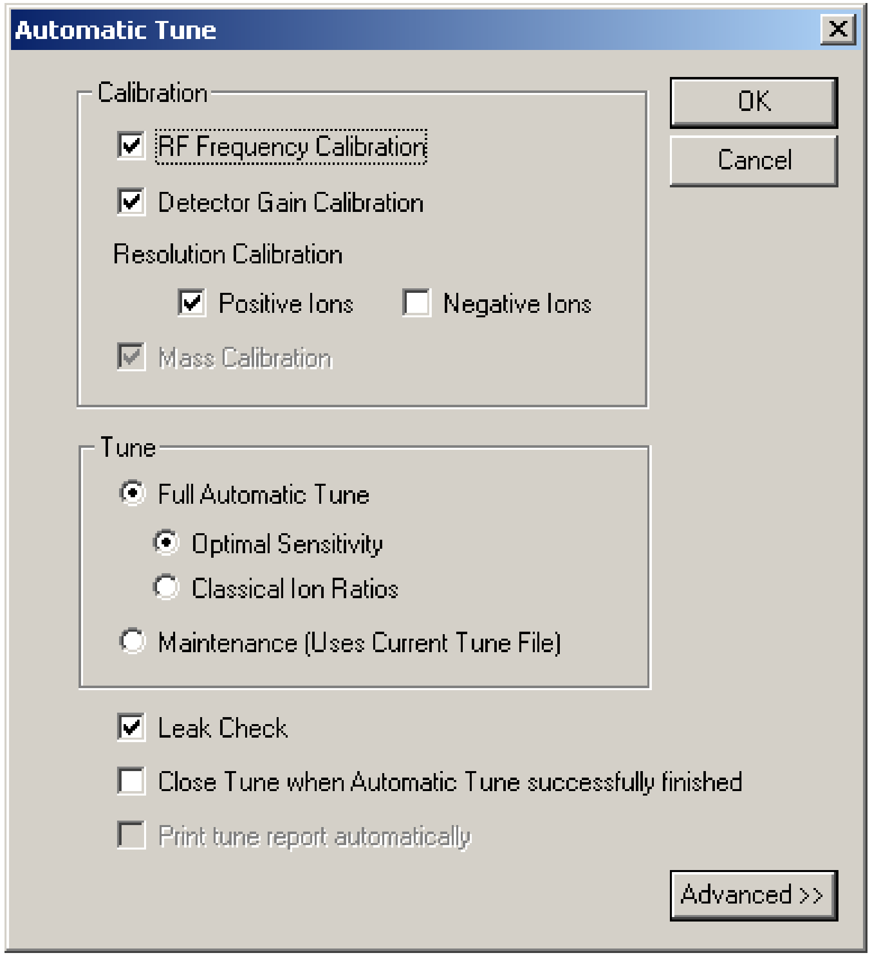
DSQ Product Support

March 2007

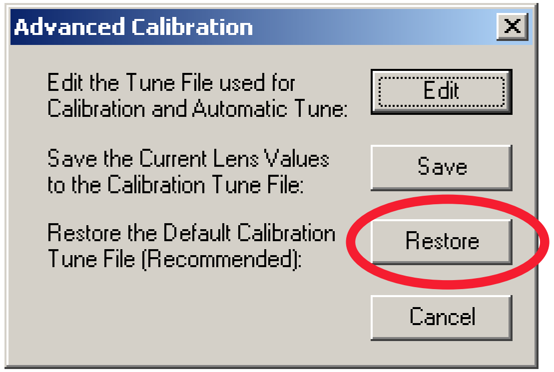
Automatic Tune
Tune and Calibration Selection



- Select Full Auto Tune to tune the instrument and generate a new autotune tune file. Full Auto Tune forces the RF frequency calibration to run when selected and does not tune the instrument if only RF Frequency is selected
- Maintenance Auto Tune allows generation of a report with the user's tune file and only changes the RF if the calibration is selected and the Detected RF is low.

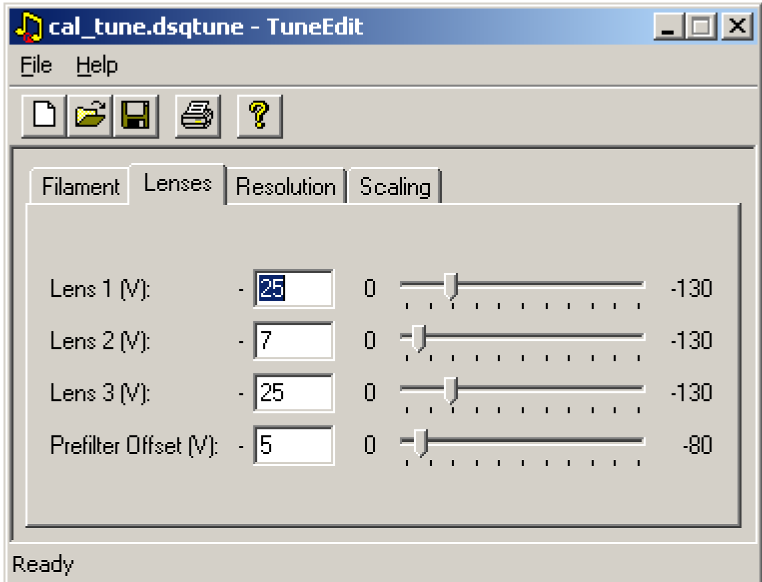
Analyze • Detect • Measure • Control™ Thermo ELECTRON CORPORATION

Advanced Calibration and Autotune Menu



The Advanced button on the Tune menu allows customer modification of the default values Calibration and Autotune routines use.

The Restore button resets these values back to the factory default values.



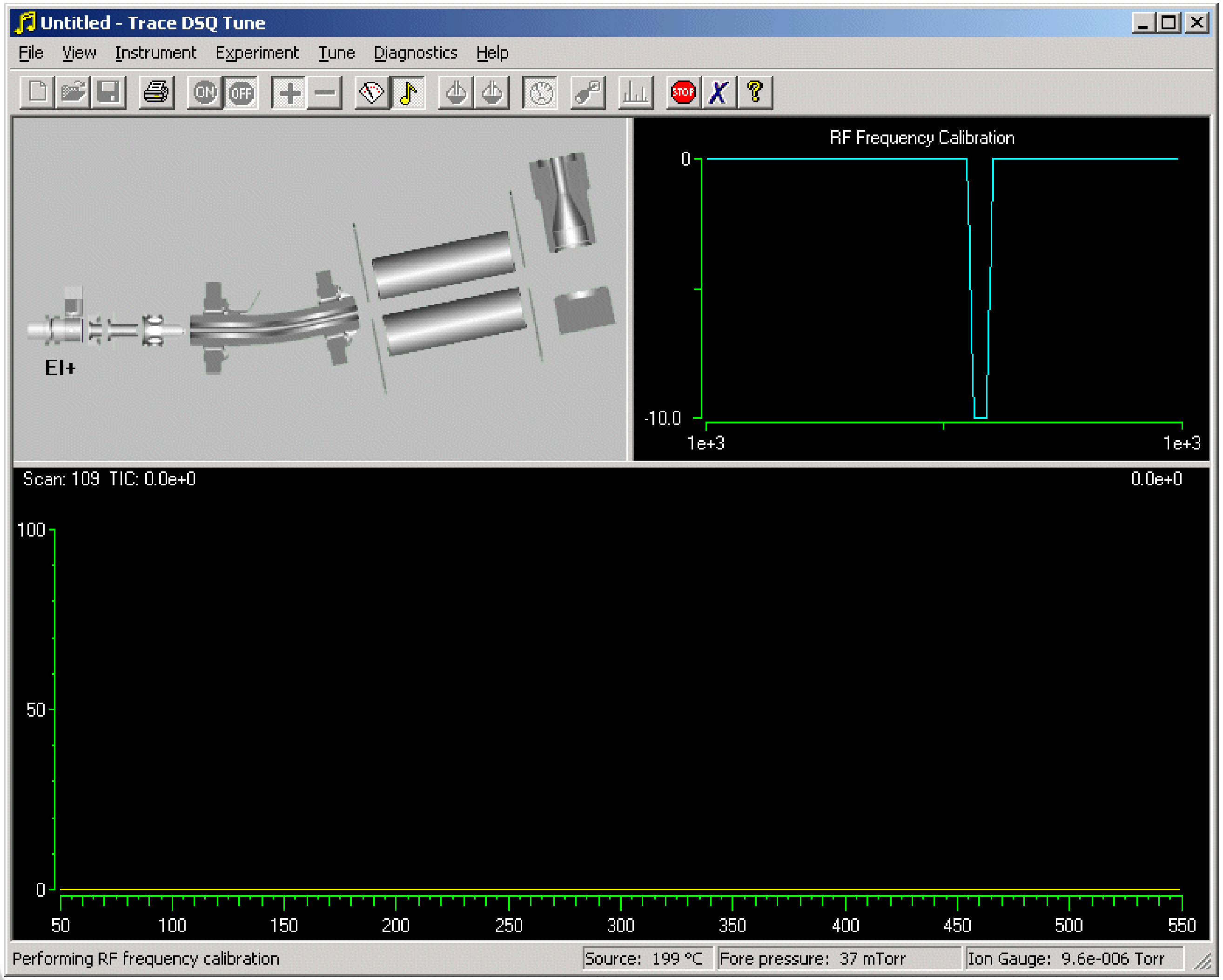
For some applications, setting the Prefilter offset value to 0 helps with low mass intensity, but may cause difficulty in passing Detector Gain.

You can use the Restore button to reset these values if source cleaning does not restore intensities.

Always check this in field visits for these kind of problem calls.

RF Frequency Calibration

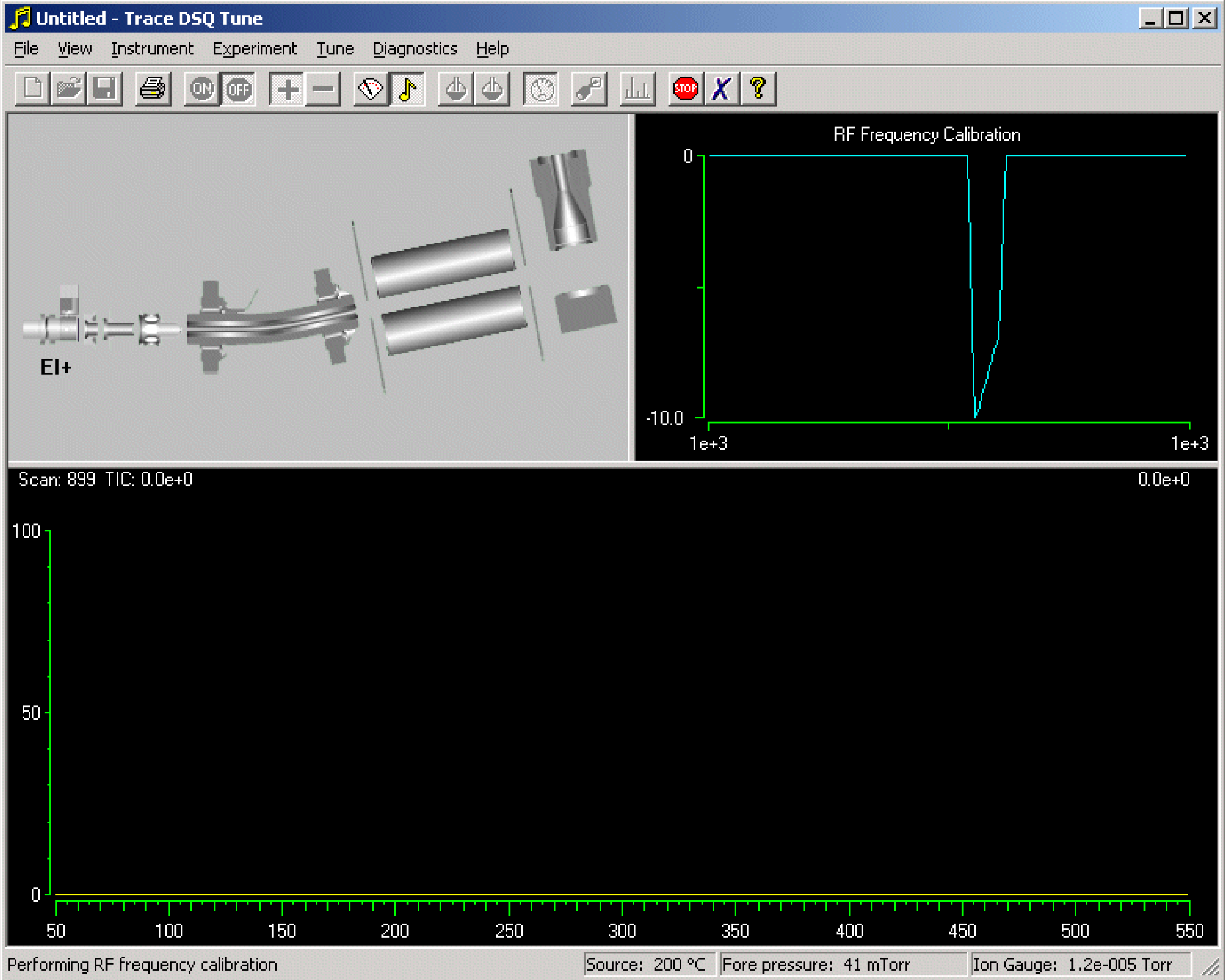
Measure Detected RF versus Frequency – Coarse



- Measure Detected RF at amplitude for maximum mass over a wide frequency range
- Discard all data points for which the RF generator reports SWR fail.
- The optimum frequency has detected RF closest to –10V

RF Frequency Calibration

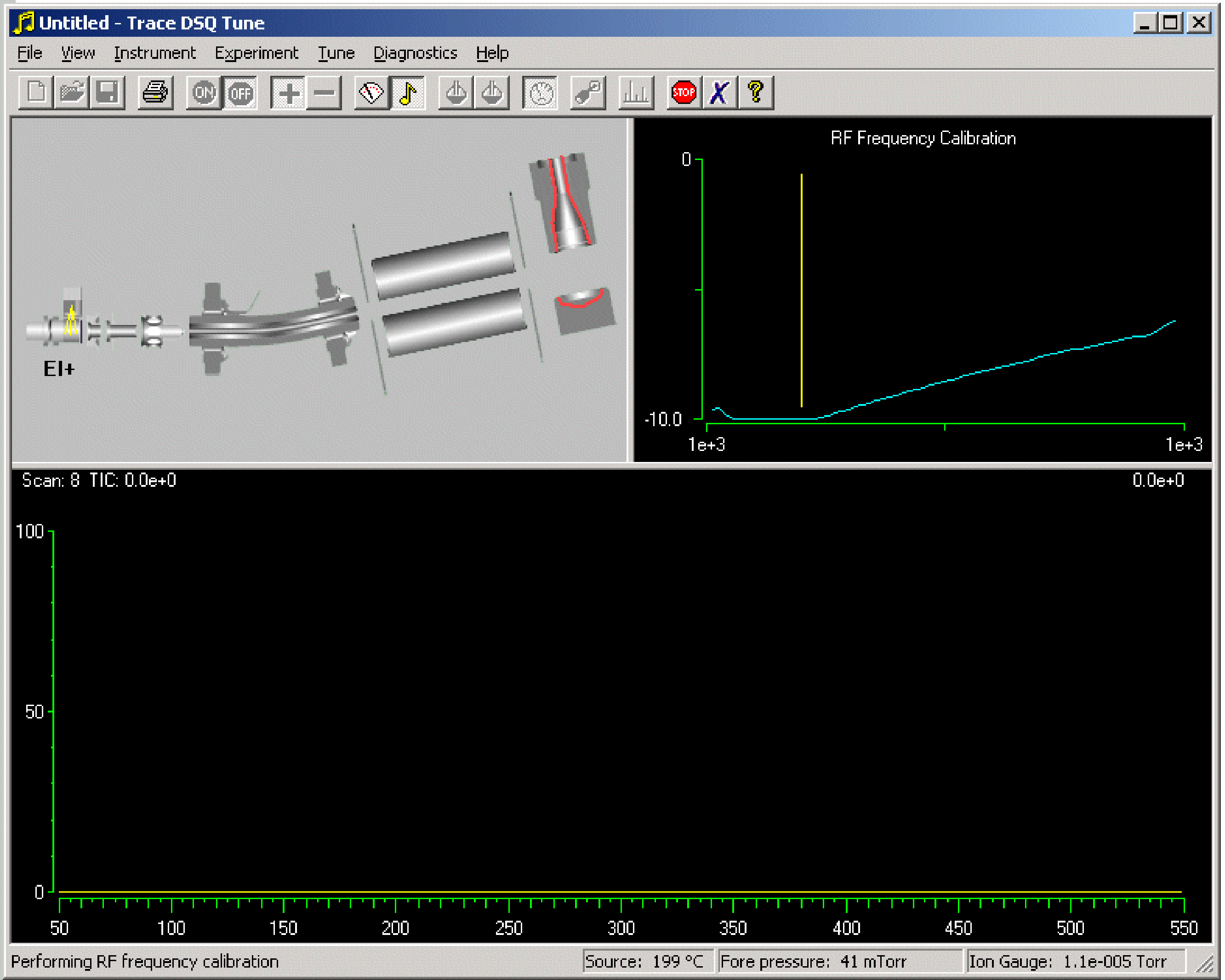
Measure Detected RF versus Frequency – Coarse



- Decrease amplitude for coarse frequency calibration until detected RF is not saturated at –10 V
- Keep frequency and amplitude for calibration over narrow range (fine)

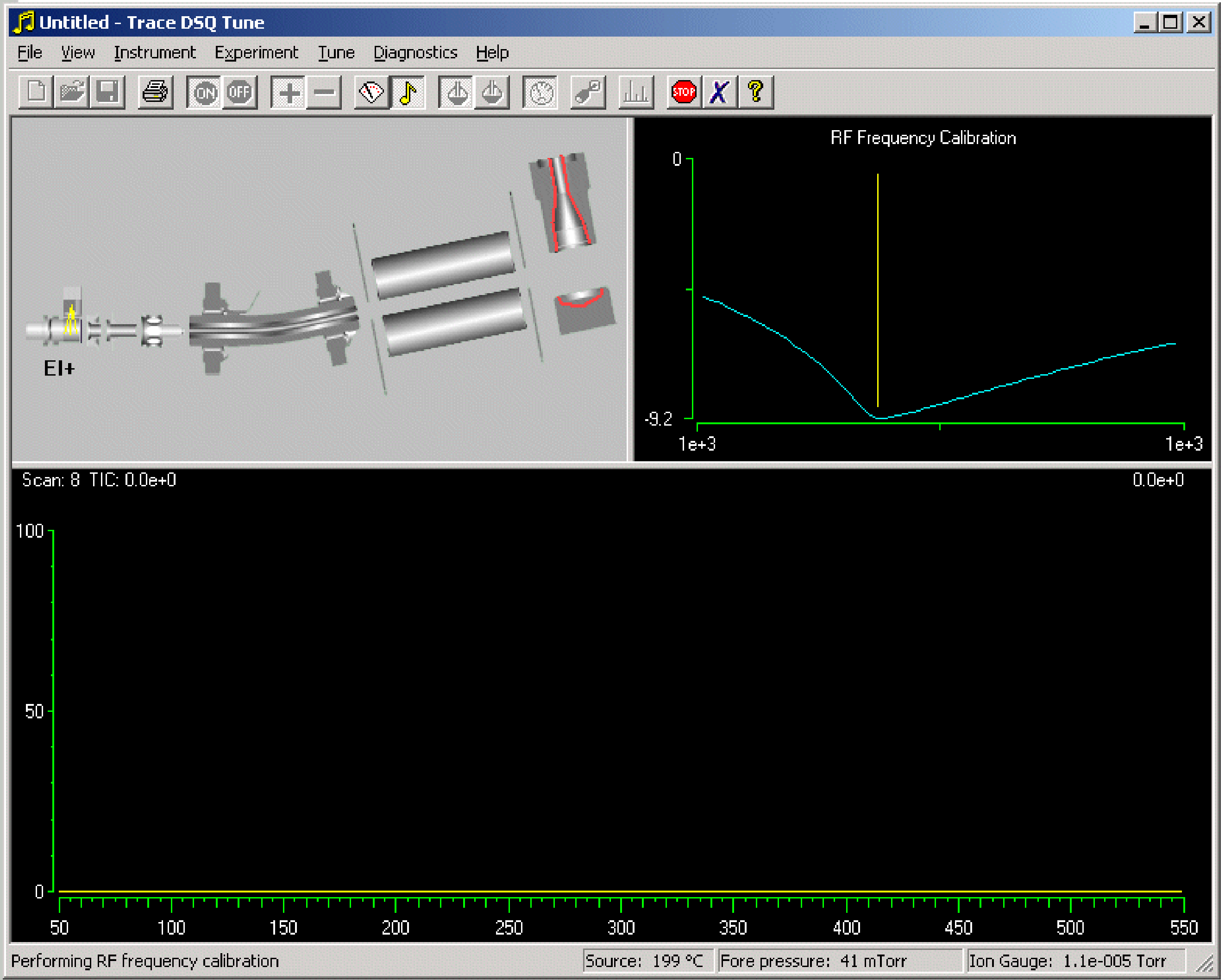
RF Frequency Calibration

Measure Detected RF versus Frequency – Fine



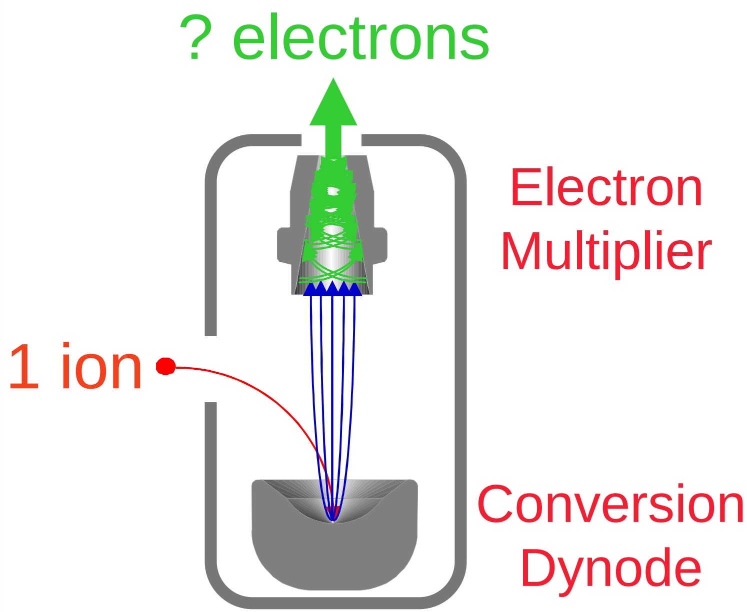
- Measure at amplitude and frequency from coarse calibration over a narrow frequency range (fine)
- Decrease amplitude until detected RF is not saturated at –10 V
- Keep frequency and amplitude for next fine check

RF Frequency Calibration
Measure Detected RF versus Frequency – Fine



- Repeat coarse and fine calibrations and report the average value
- Fail if optimum is not found at 1100 +/- 50 kHz
- Fail calibration if the difference between any two frequencies is
  > 0.5 kHz

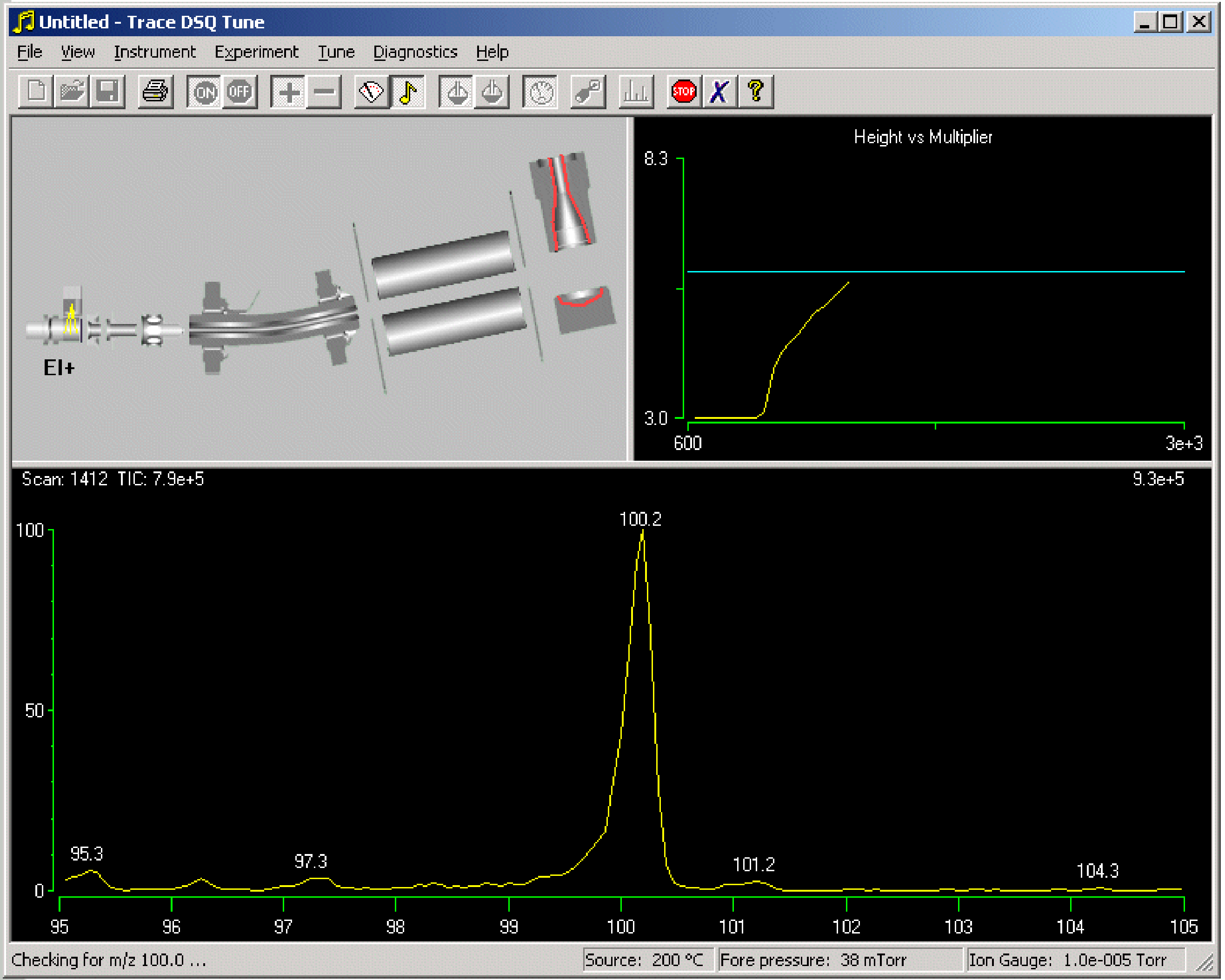
Detector Gain Calibration



$$\text{Gain} = \frac{\text{Number of electrons out}}{\text{Number of ions in}}$$

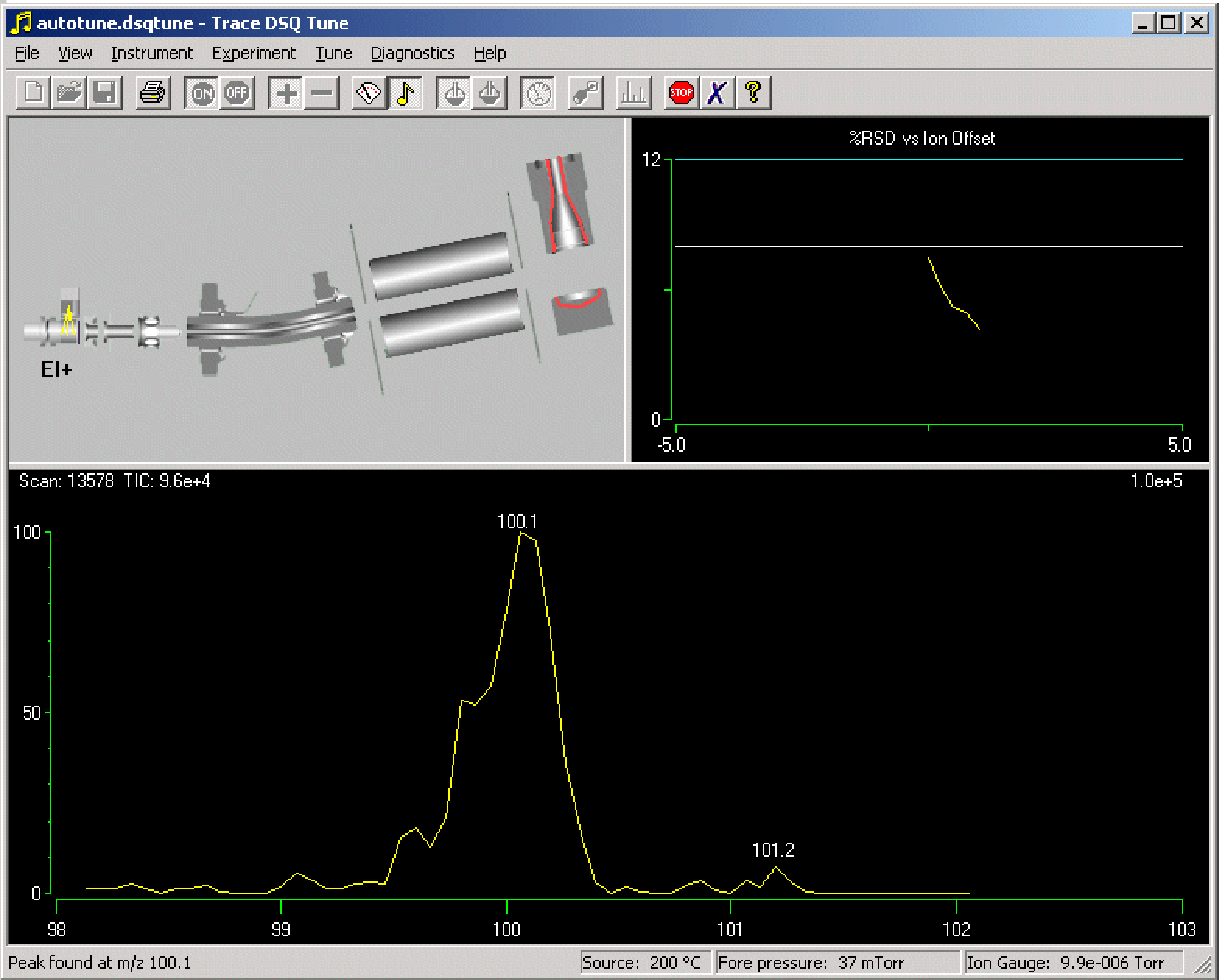
$$\text{Gain} = 300{,}000 \quad \text{(typical)}$$

Detector Gain Calibration
Find Initial Multiplier Setting



- Multiplier voltage is increased until m/z 100 reaches the desired intensity

Detector Gain Calibration
Adjust Ion Offset

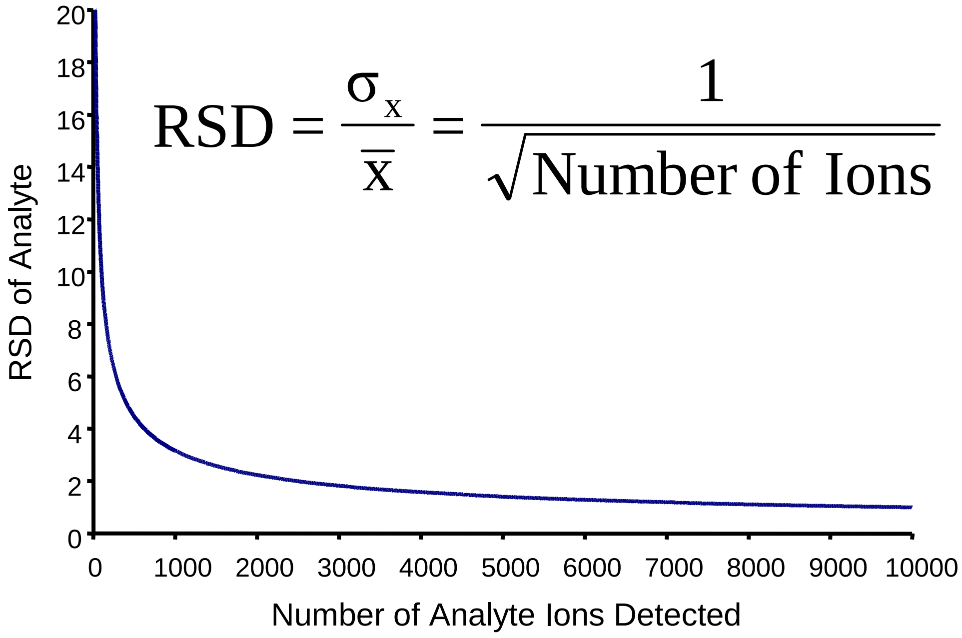


- Ion offset is adjusted so the desired ion flux is achieved
- It must be a low enough signal so we can accurately measure the number of ions
- How do we calculate the number of ions?

Detector Gain Calibration

Calculating the Number of Ions

- Detecting an ion produced from a molecule in the ion source is a random, discrete event
- Therefore, Poisson statistics governs the measurement
- Poisson says the precision is related to the number of ions



$$\mathrm{RSD} = \frac{\sigma_x}{\bar{x}} = \frac{1}{\sqrt{\text{Number of Ions}}}$$

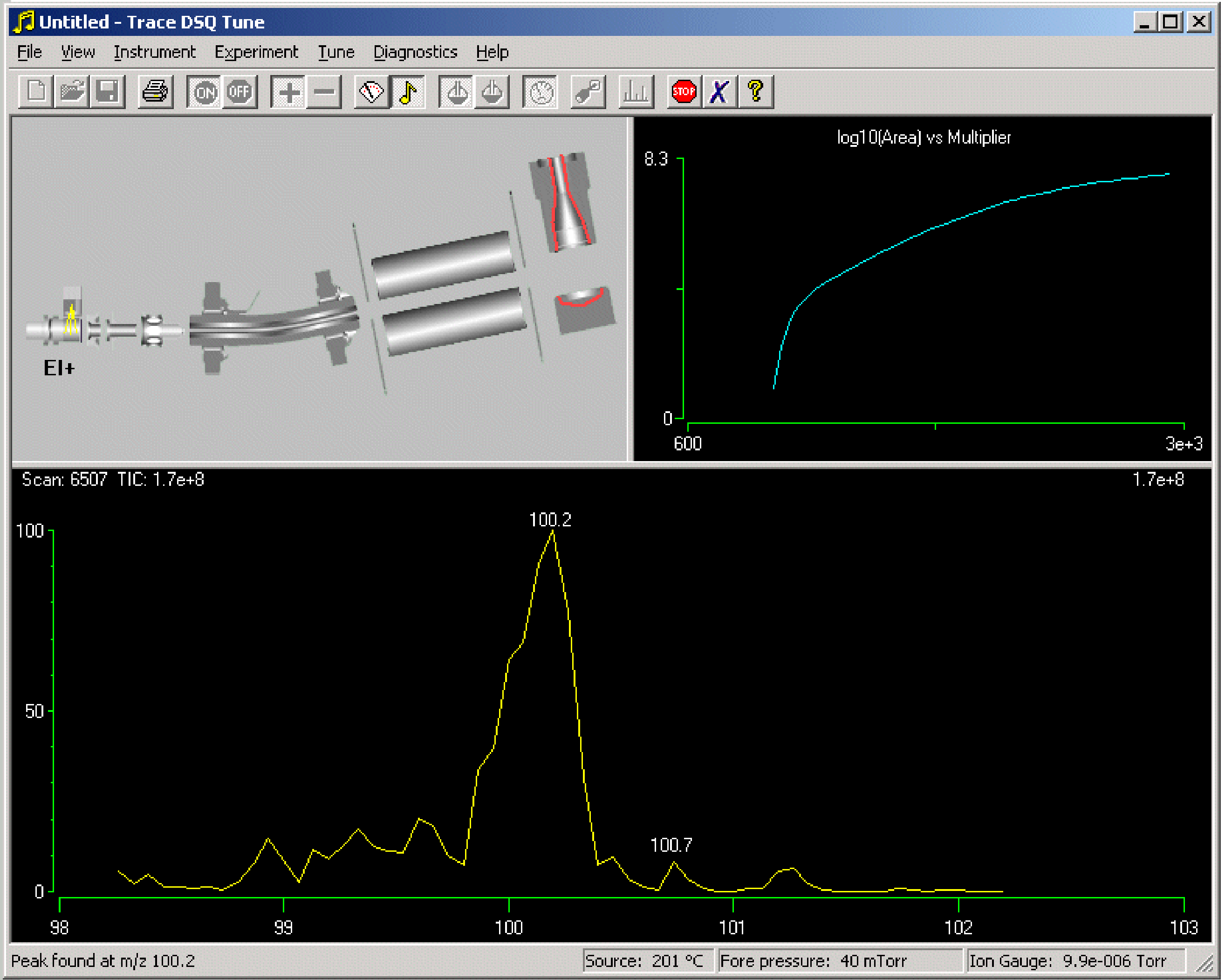
$$\text{Number of Ions} = \frac{1}{\mathrm{RSD}^2}$$

$$\text{Number of Ions} = \frac{1}{0.1^2} = 100$$

Fies, W.J. *Int. J. Mass Spectrom. Ion Processes* **1988**, 82, 111-129

Detector Gain Calibration

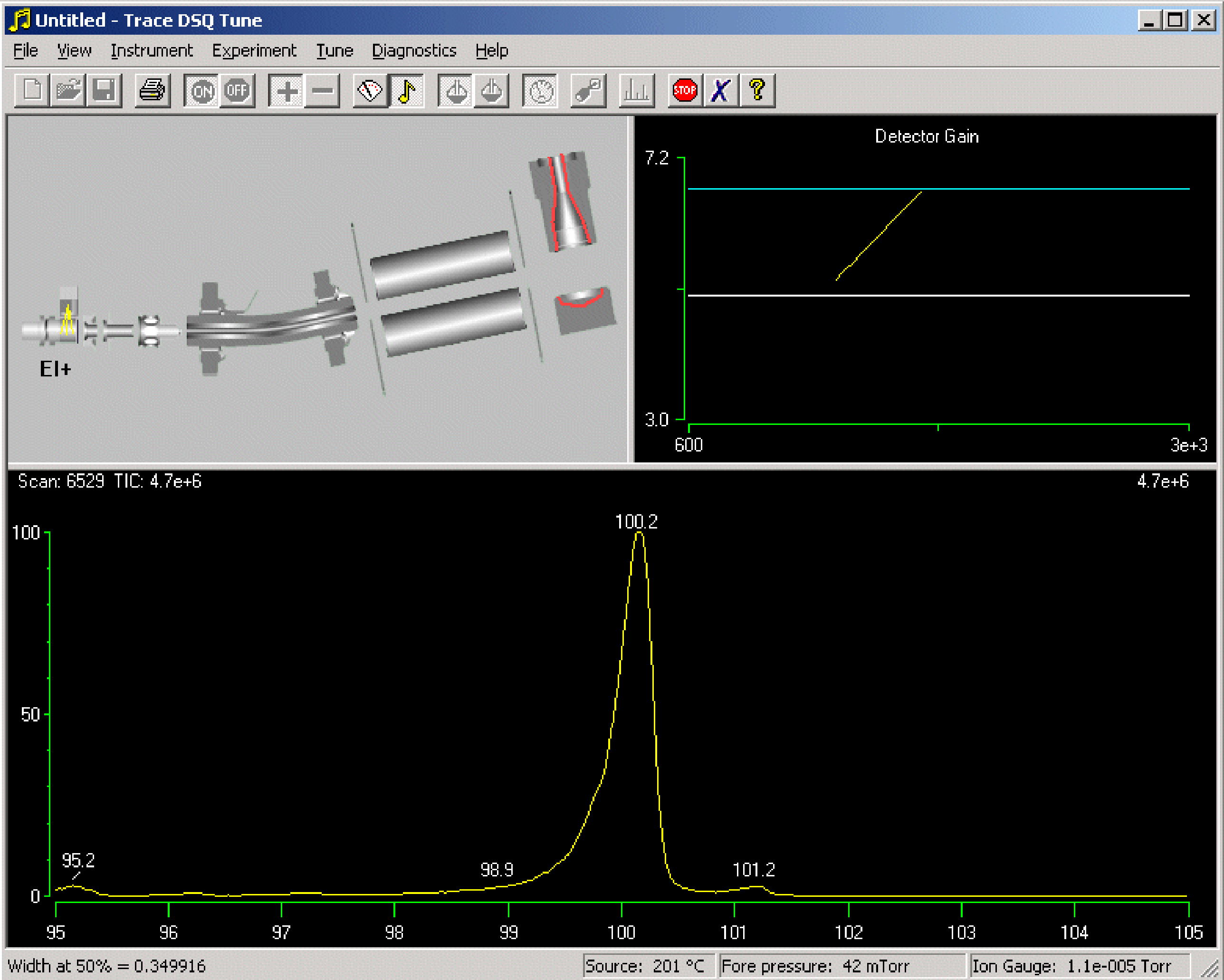
Measure Area & RSD for Each Multiplier Voltage



- Once we know the area & RSD, we can calculate the gain
- The gain at different multiplier voltages is stored in the calibration

Detector Gain Calibration

Plot Result of Gain vs Multiplier Voltage



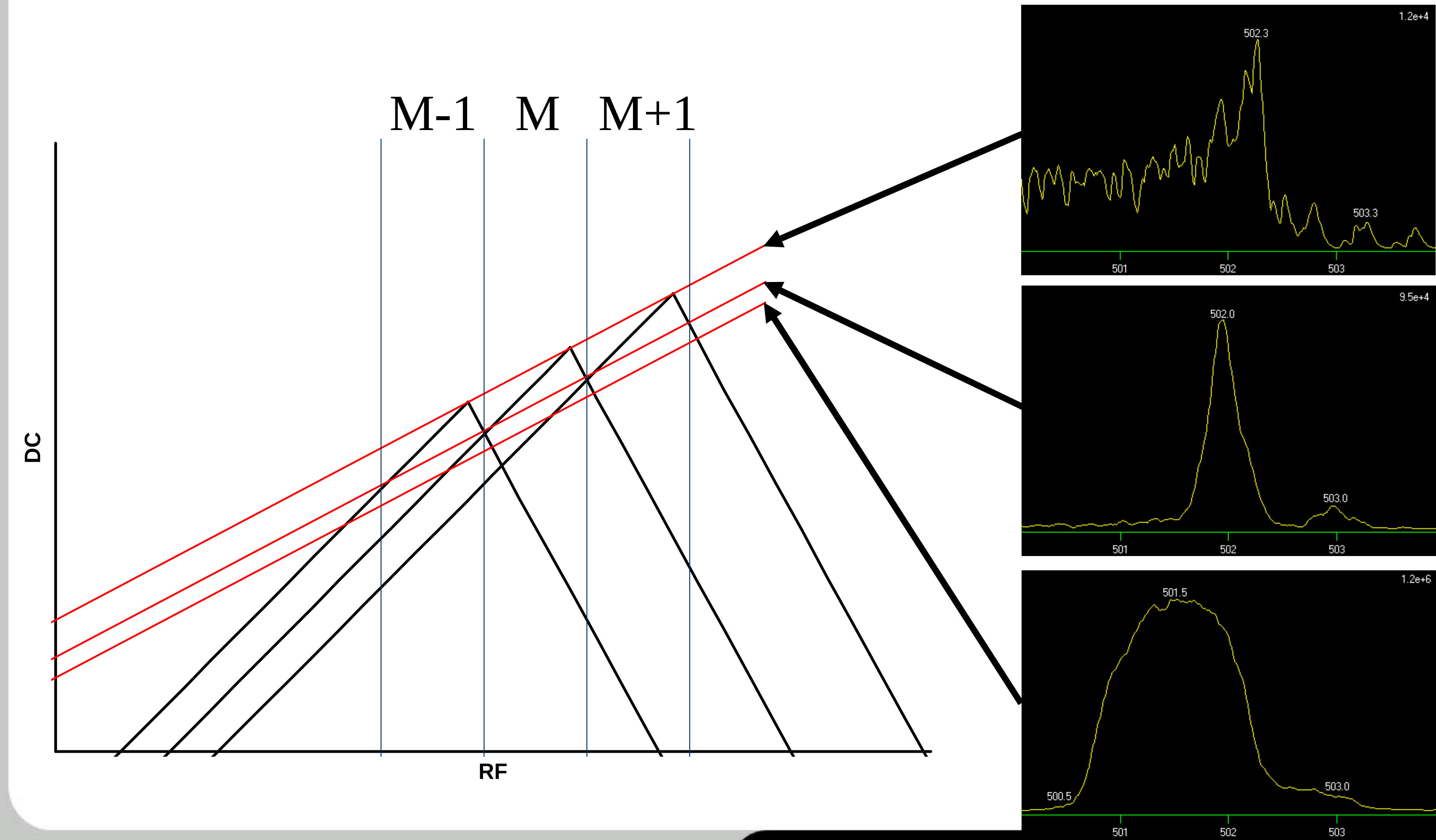
- Results of calibration are plotted. Horizontal lines represent $3x10^5$ and $5x10^6$
- This allows you to easily change the gain in your methods from $1x10^4$ to $5x10^6$
- Recommended gain is $3x10^5$
- Also shown is mass check for next calibration

Mathieu Parameters

$$RF = \frac{m\ r^2\omega^2}{e\ \ 2q_z}$$

$$DC = \frac{m\ r^2\omega^2}{e\ \ 4a_z}$$

Resolution Calibration
DC RF Scan Line



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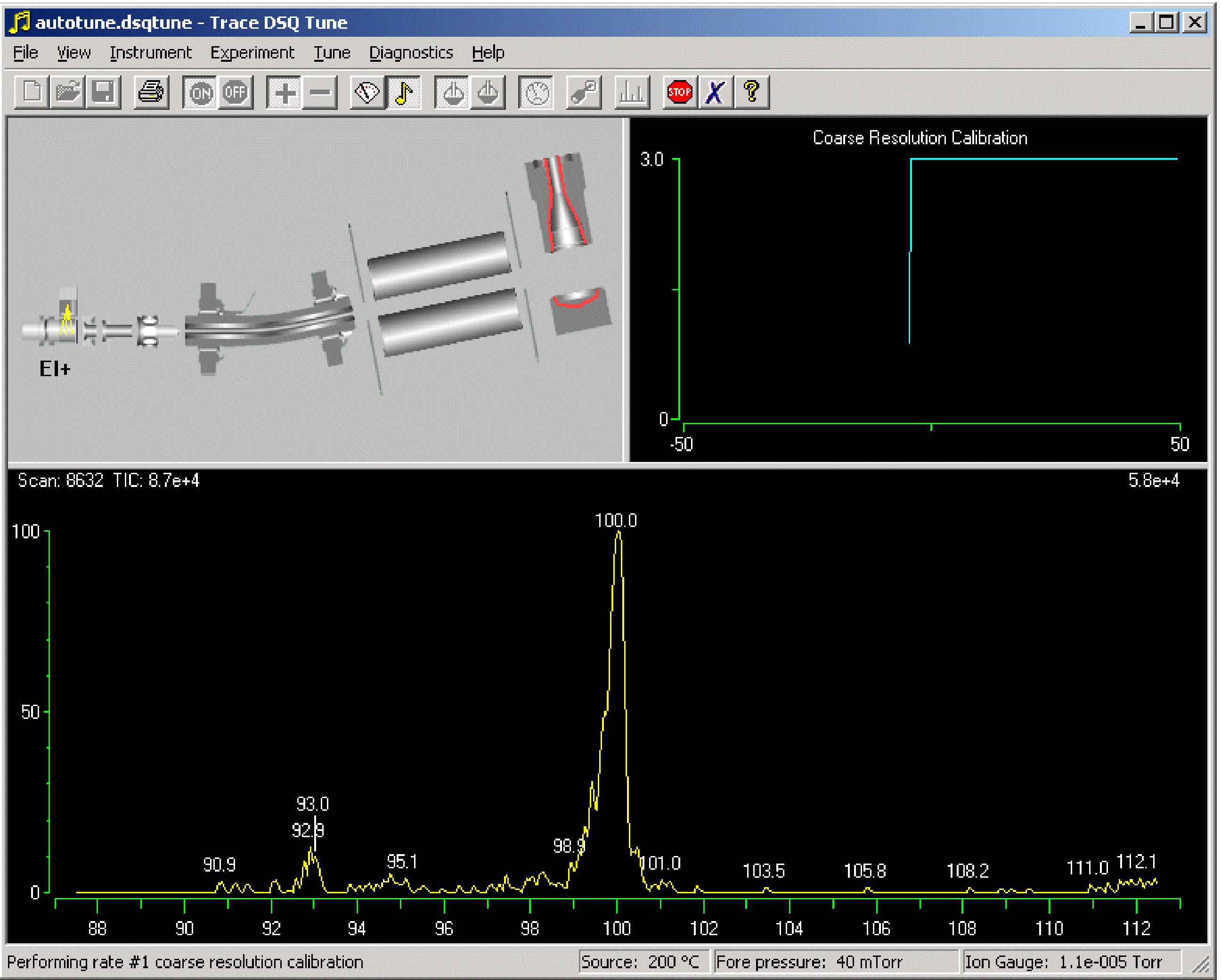
Resolution Calibration

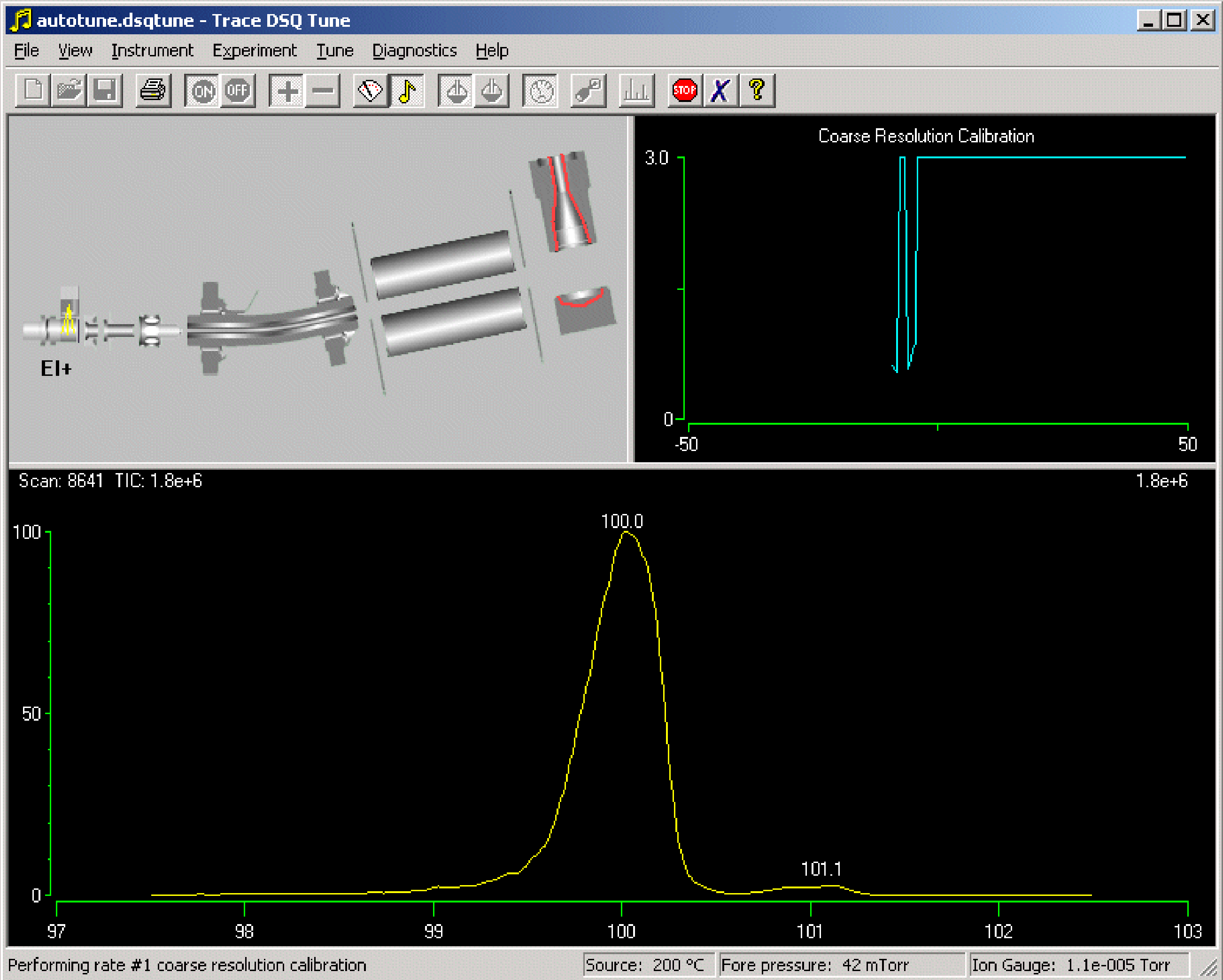
Plot Peak Width (20% height) vs Offset to DC (mV)



- Results of coarse calibration in progress showing precursors and over resolved ions as DC is adjusted from above the optimum DC scan line
- A wide mass range is scanned until the peak is identified

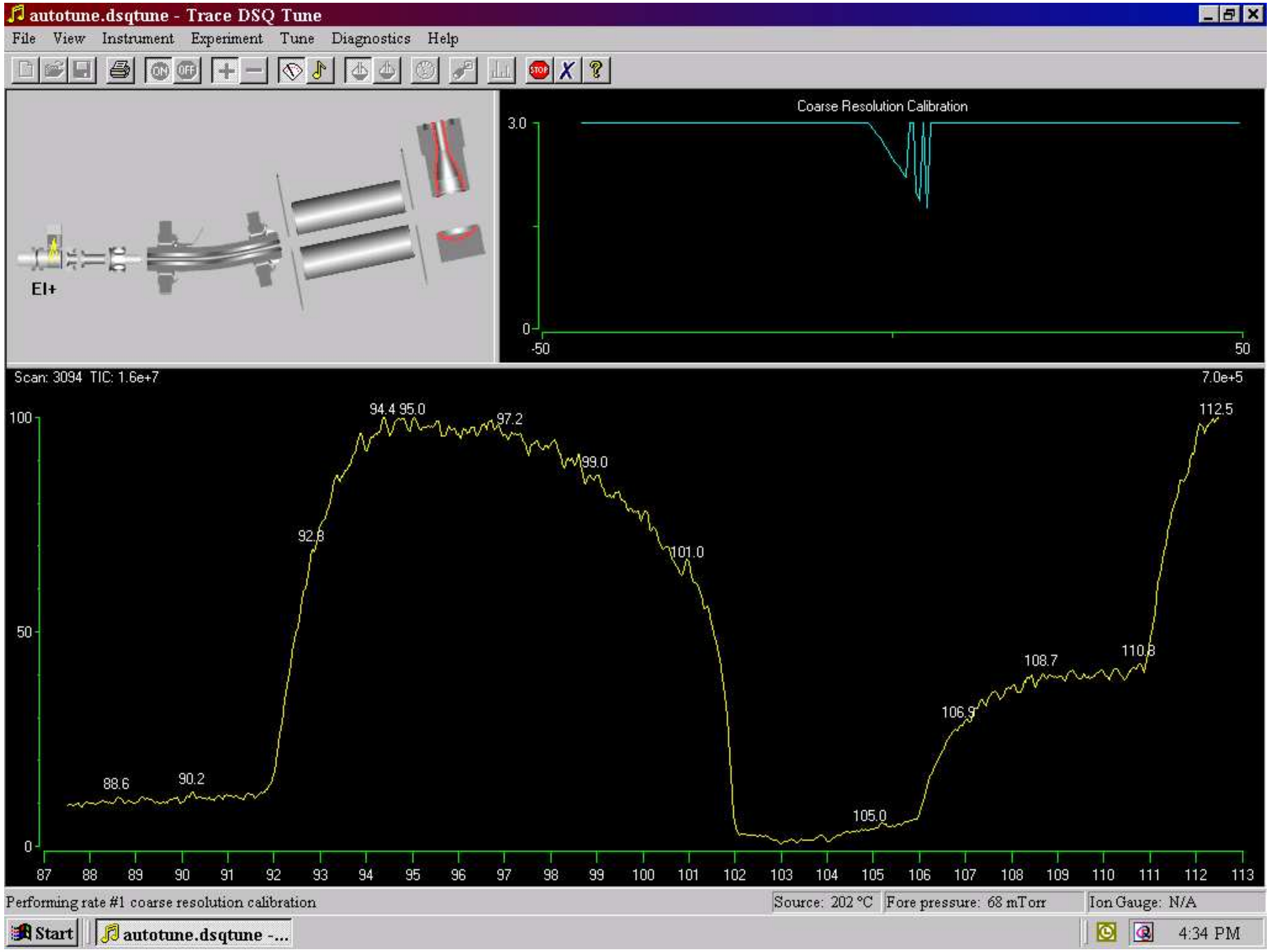
Resolution Calibration

Plot Peak Width (20% height) vs Offset to DC (mV)



- Results of coarse calibration in progress showing resolved ions as DC is adjusted through optimum DC scan line
- The peaks mass position is identified and centered in a narrow window for added speed

Resolution Calibration Failure Due to Low Signal Intensity



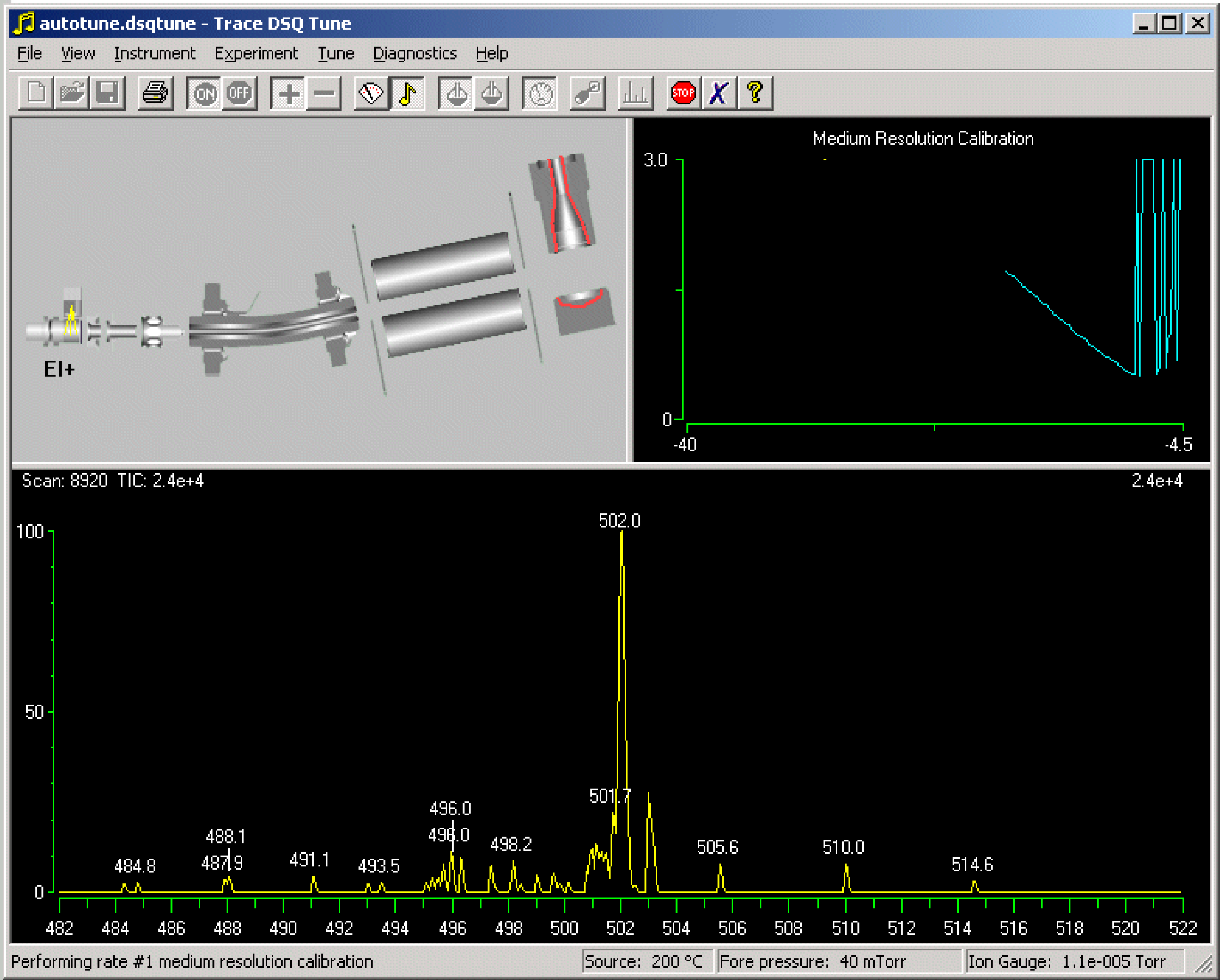
If the peak is low intensity, shown by the small drop in the indicator plot on the upper graph, the resolution calibration will fail.

Stop the autotune, raise the multiplier 50-100V and start.

This will also fail on a system needing source or prefilter cleaning.

Resolution Calibration

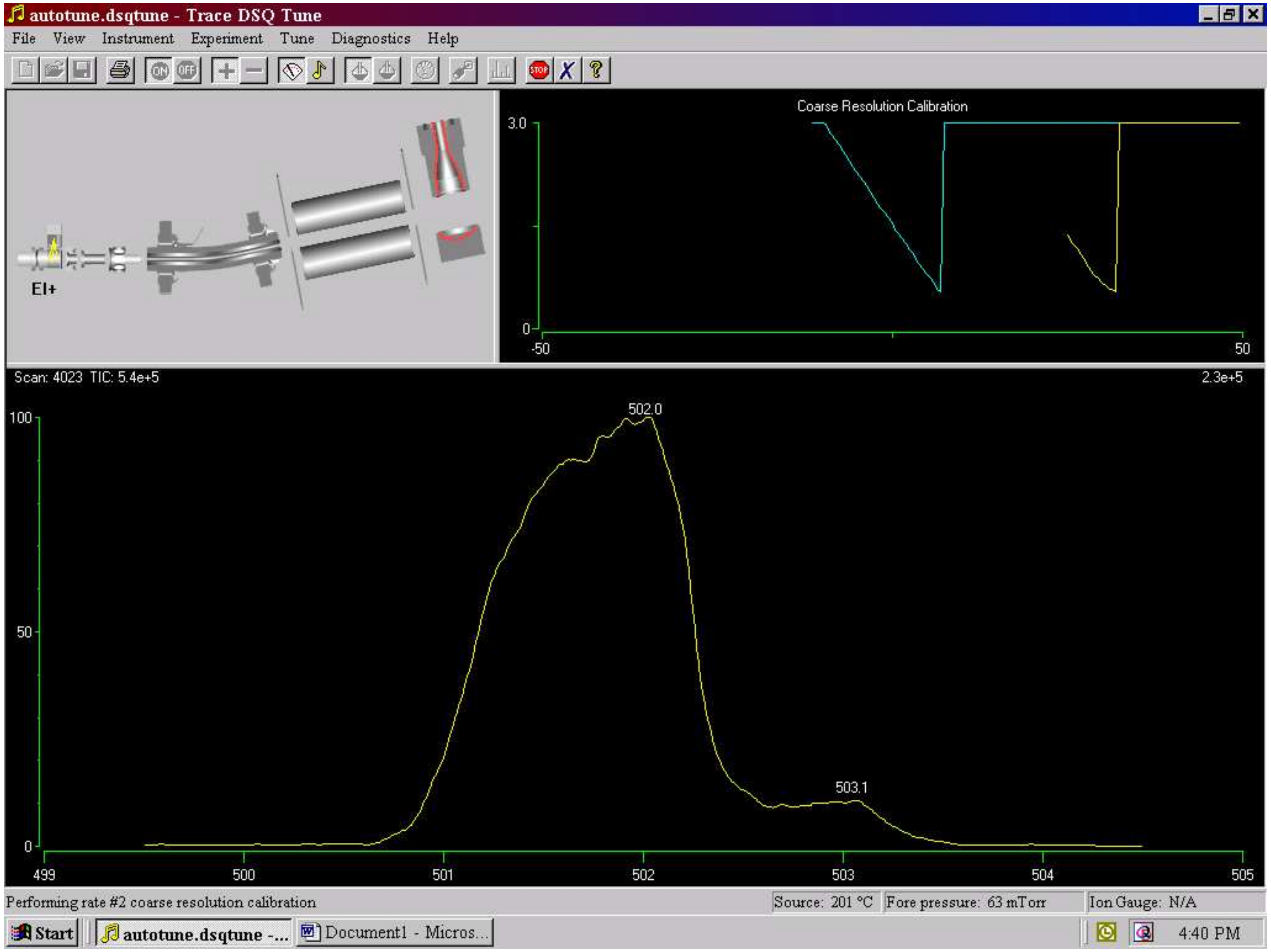
Plot Peak Width (20% height) vs Offset to DC (mV)



- Coarse calibration is completed for masses 100 and 502
- Results are used to perform medium calibration over narrower DC range
- This spectrum shows over resolved ions with precursors resulting from DC that is too high

Resolution Calibration

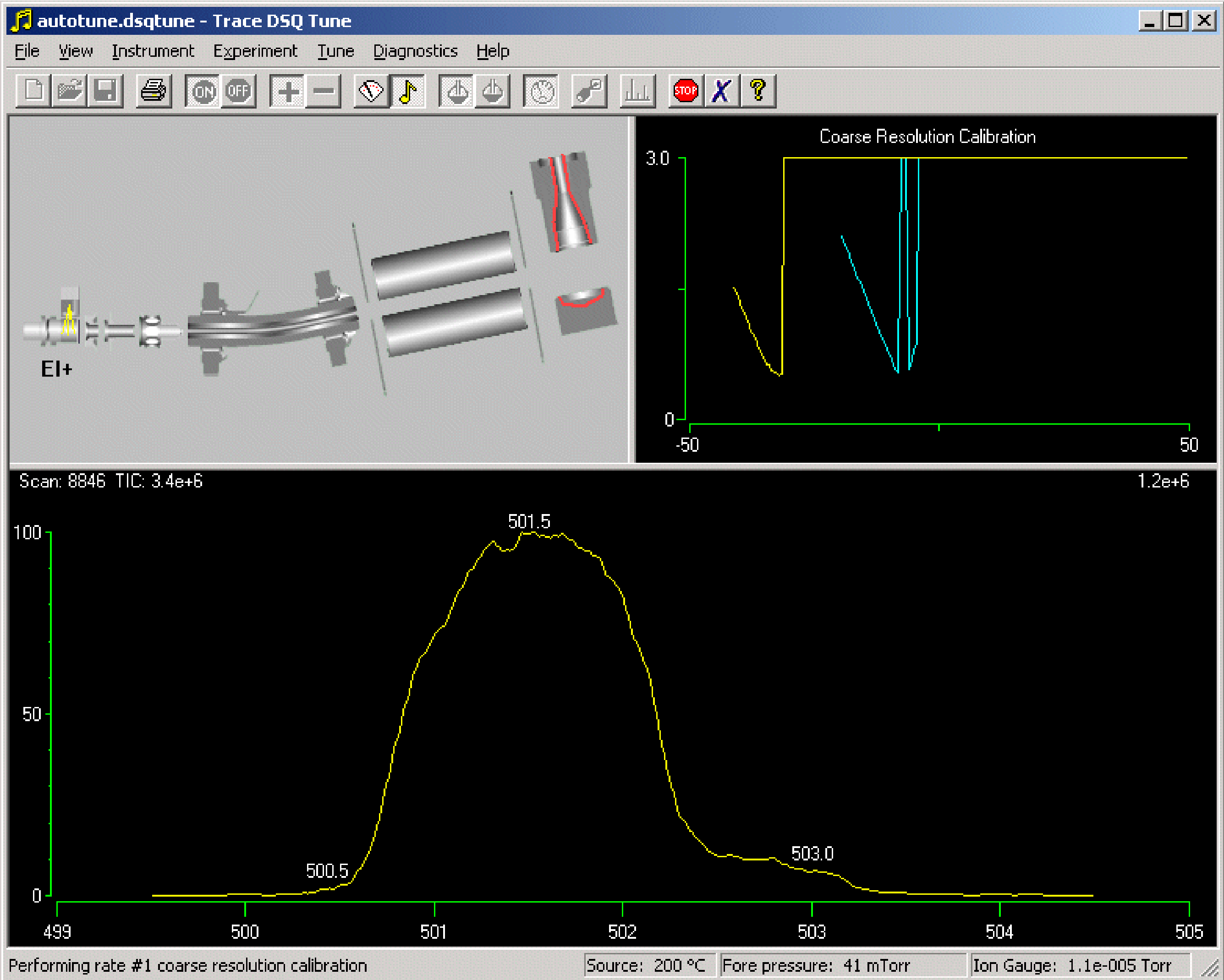
Plot Peak Width (20% height) vs Offset to DC (mV)



Successful tune for both mass 100 and 502.

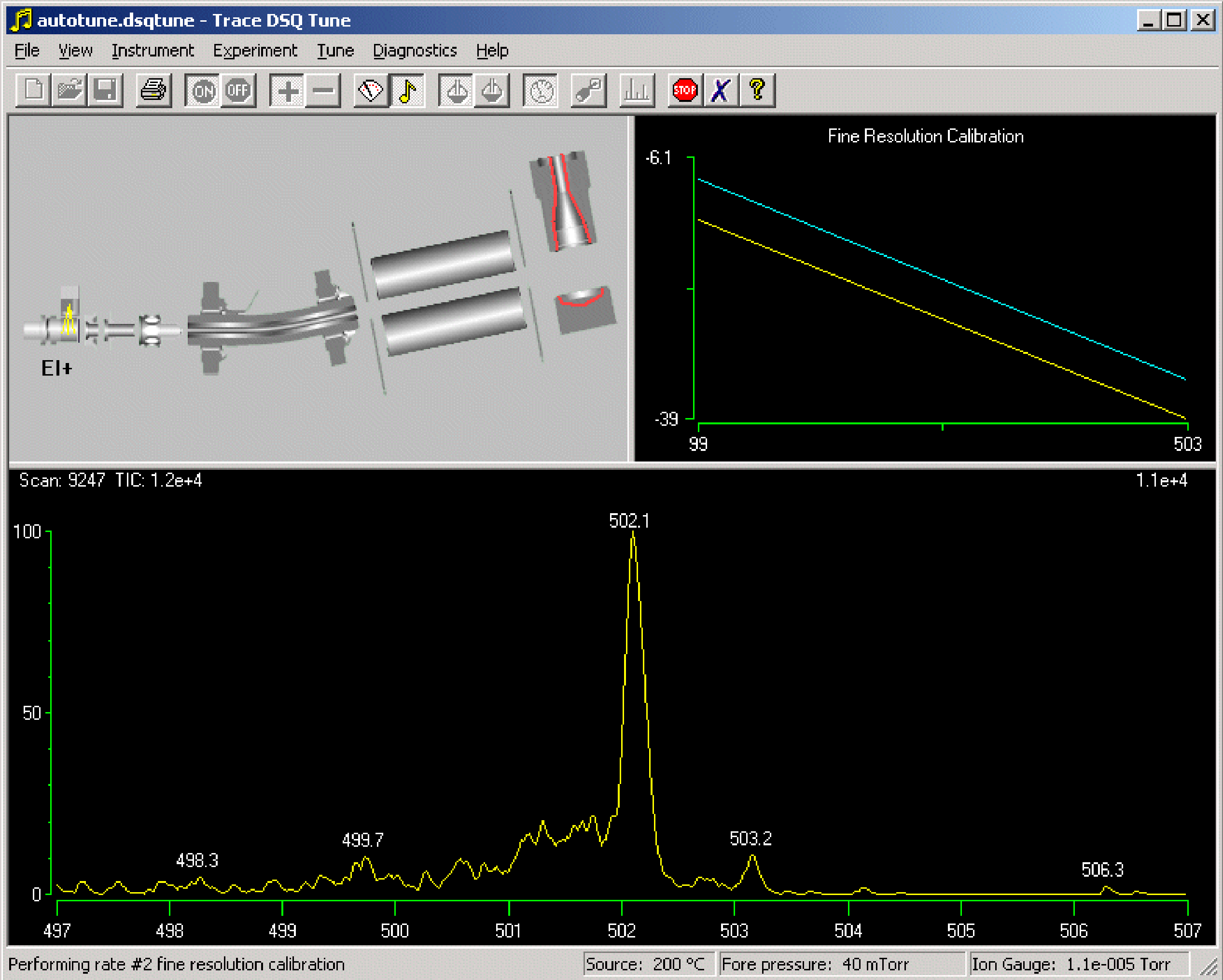
Resolution Calibration

Plot Peak Width (20% height) vs Offset to DC (mV)



Resolution Calibration

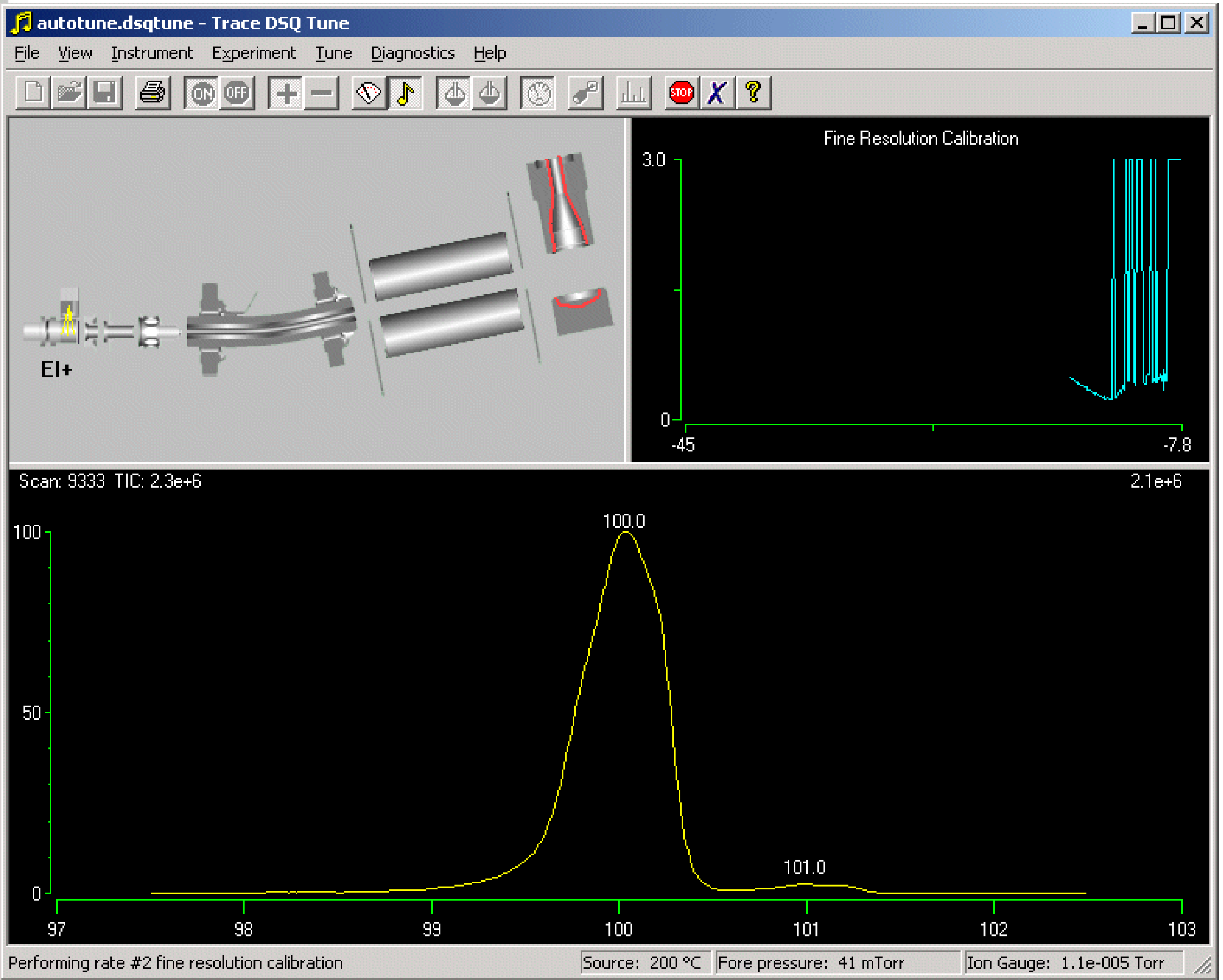
Plot Results from Rate 1 and Check Rate 2



- Results of Calibration are plotted as DC vs m/z for a fast scan rate (rate #1) at two peak widths
- The mass checks and calibrations are repeated at a slower scan rate
- Masses are narrow enough to begin with a fine calibration

Resolution Calibration

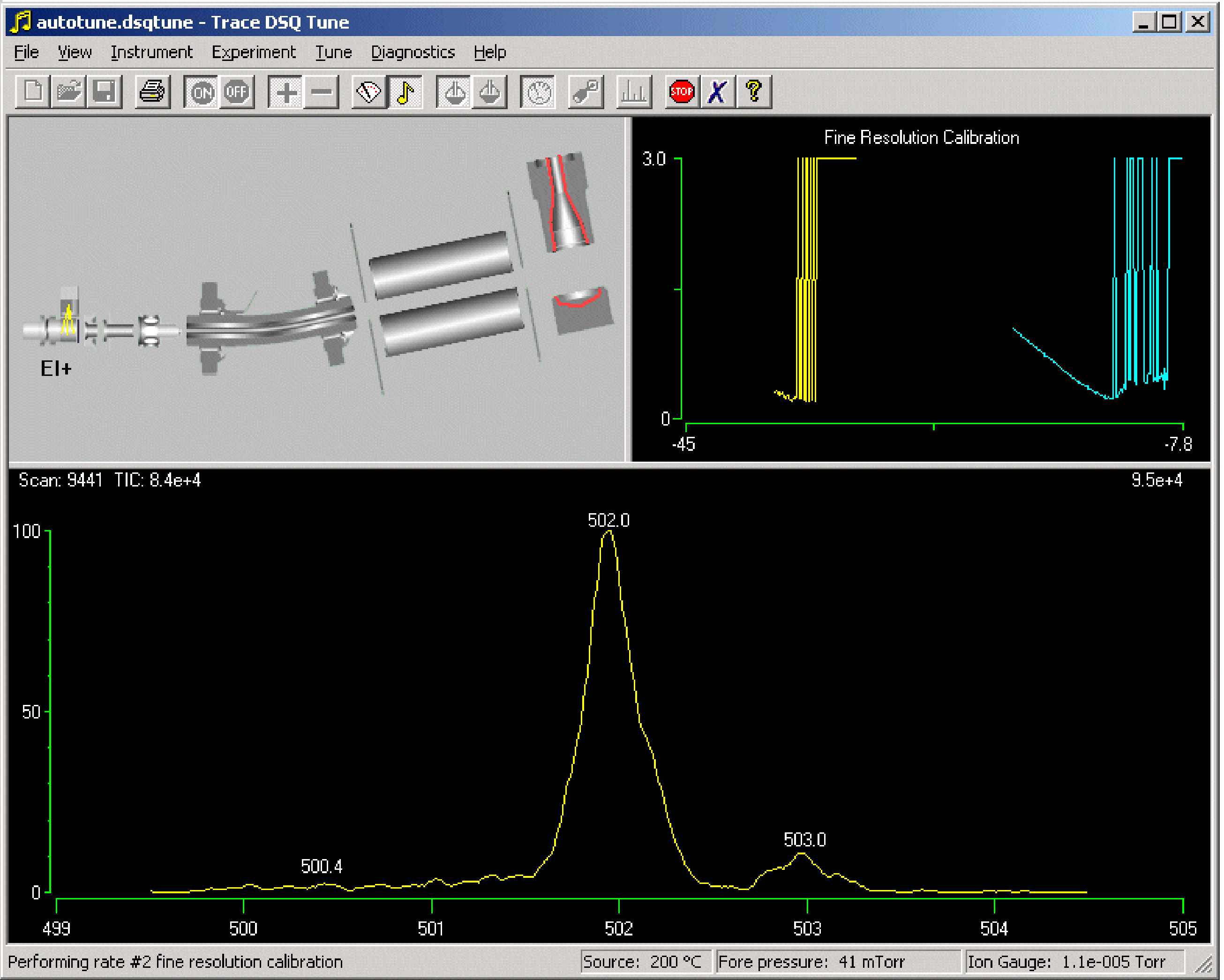
Plot Peak Width (50% height) vs Offset to DC (mV)



- Calibrating the resolution for rate #2
- Width at 50% height is used for the fine calibration because it gives the best linear fit

Resolution Calibration

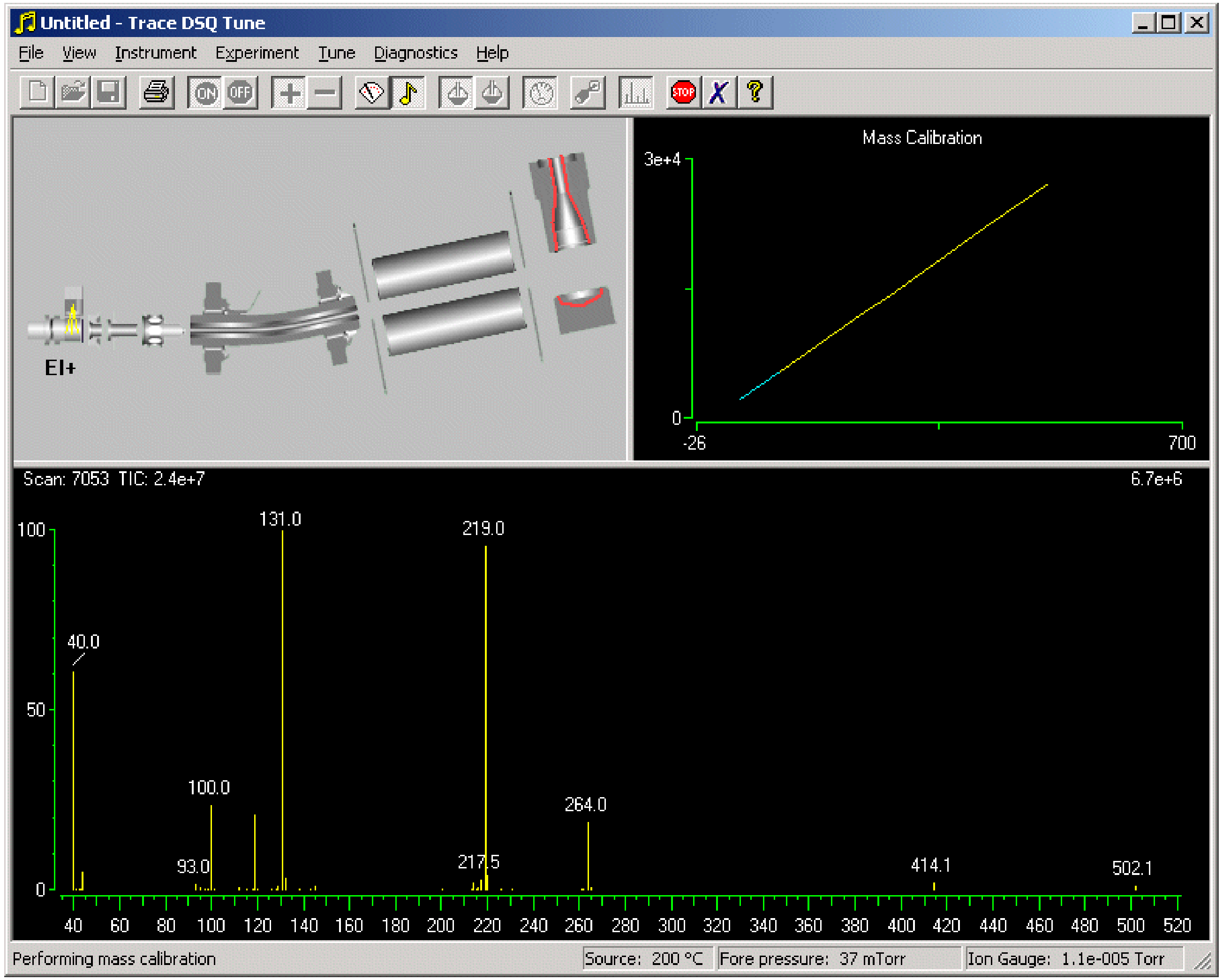
Plot Peak Width (50% height) vs Offset to DC (mV)



- Once rate # 2 calibration is complete, the resolution variables used in tune files are available for all scan rates, masses, and peak widths.

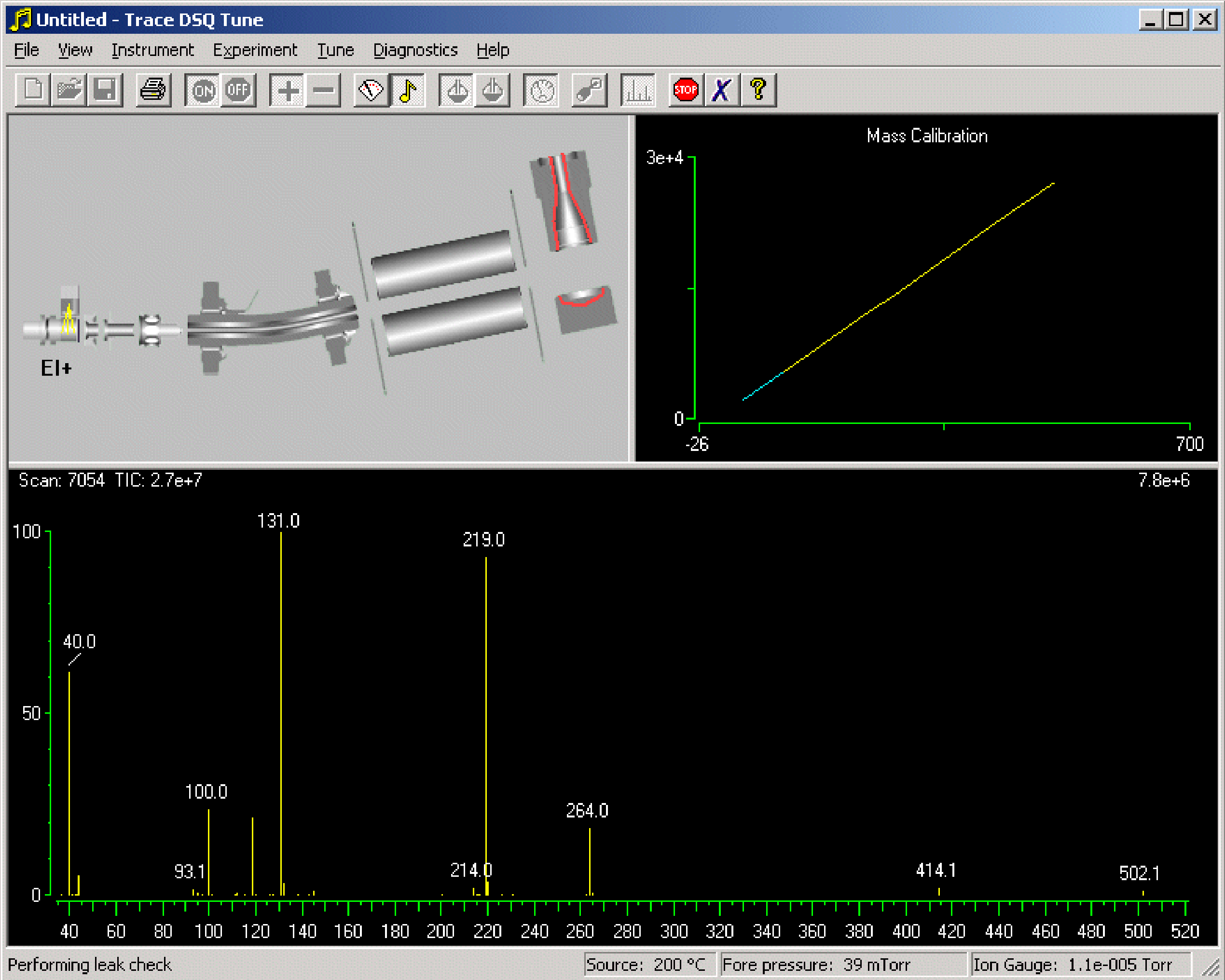
Mass Calibration

Measure Centroid Mass vs RF DAC



- Centroid masses are also recorded at two scan rates for determining the actual RF DAC setting for each mass
- The spectra displayed during the mass calibration are the found masses and do not reflect the changes made from the mass calibration

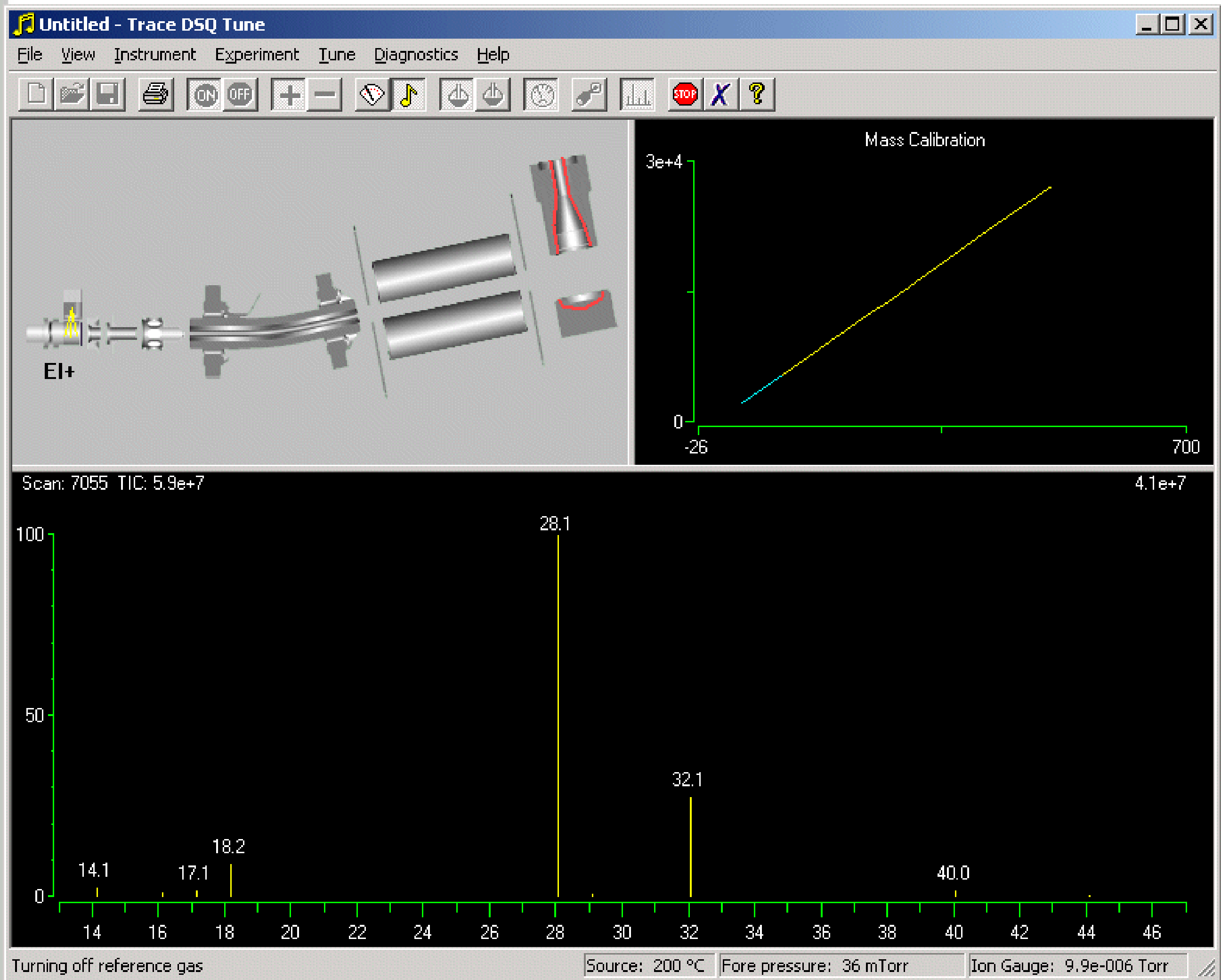
Mass Calibration
Measure Centroid Mass vs RF DAC



- Rate two of mass calibration

Leak Check

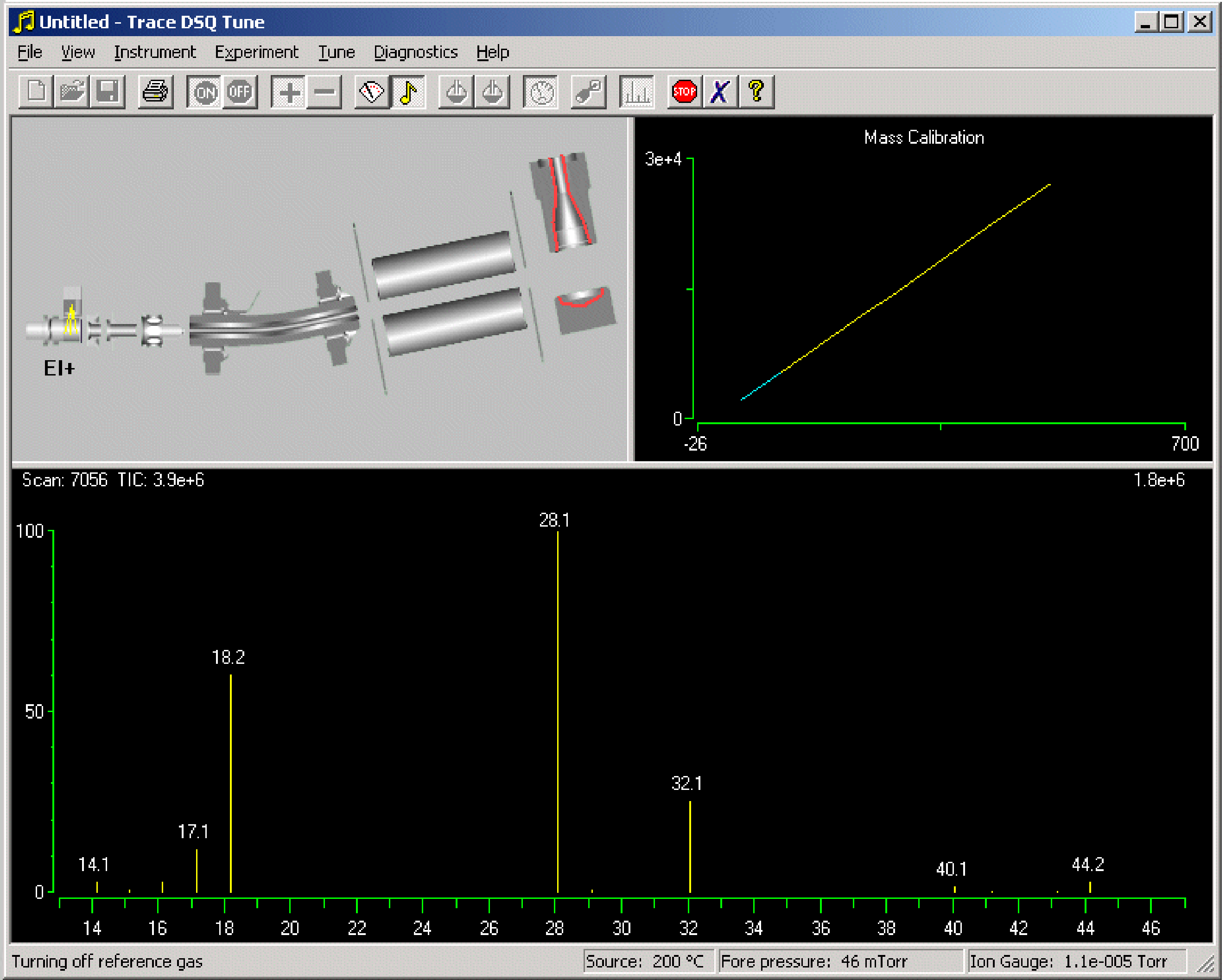
Measure Air from Reference Gas Inlet



- The air from the reference gas (cal gas module) is measured
- This leak check is more accurate than previous comparisons to helium or cal gas ions. The same scan is used for the same ions with reference on and off…

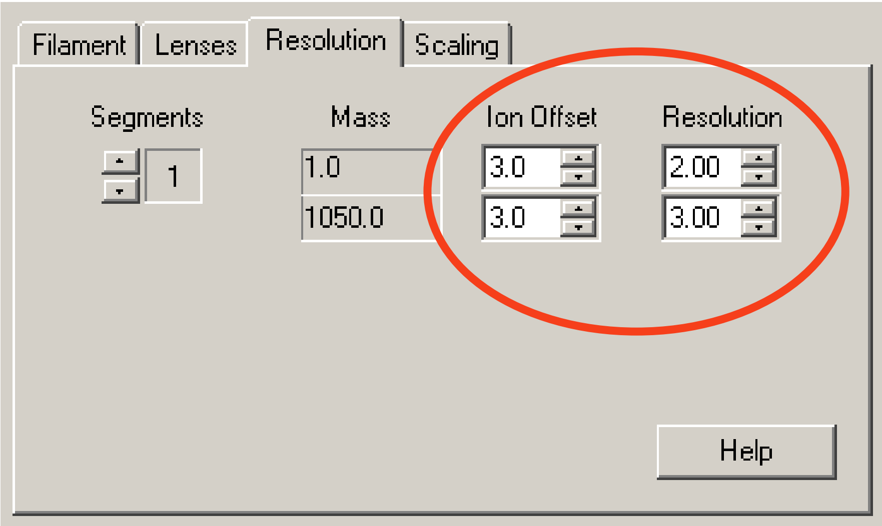
Leak Check

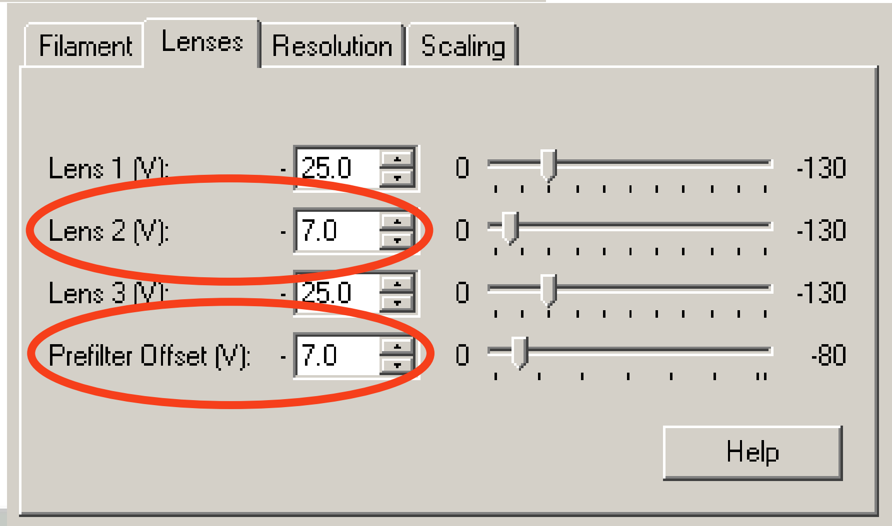
Measure Air from Background



- Measure after 30 seconds of pumping without reference
- M/z 32 is used to determine leaks because it can be filtered from the helium and is harmful to the instrument
- > 10% m/z 32 is a small air leak
- > 50% m/z 32 is a bad air leak and fails the check

Full Auto Tune

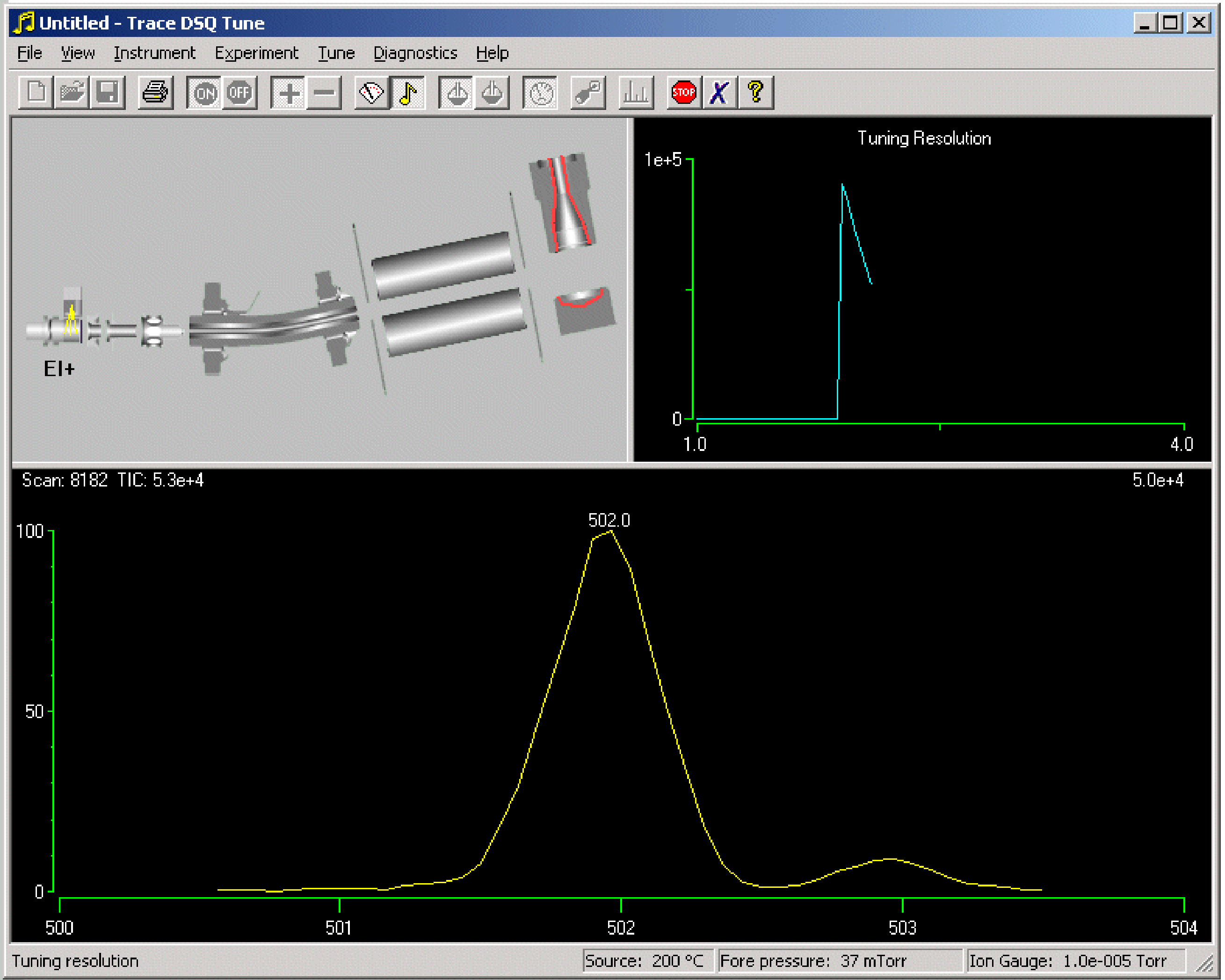




- Tunes Resolution
- Tunes Ion Offset
- Tunes Prefilter Offset
- Tunes Lens 2
- Tunes Resolution

Tune Resolution

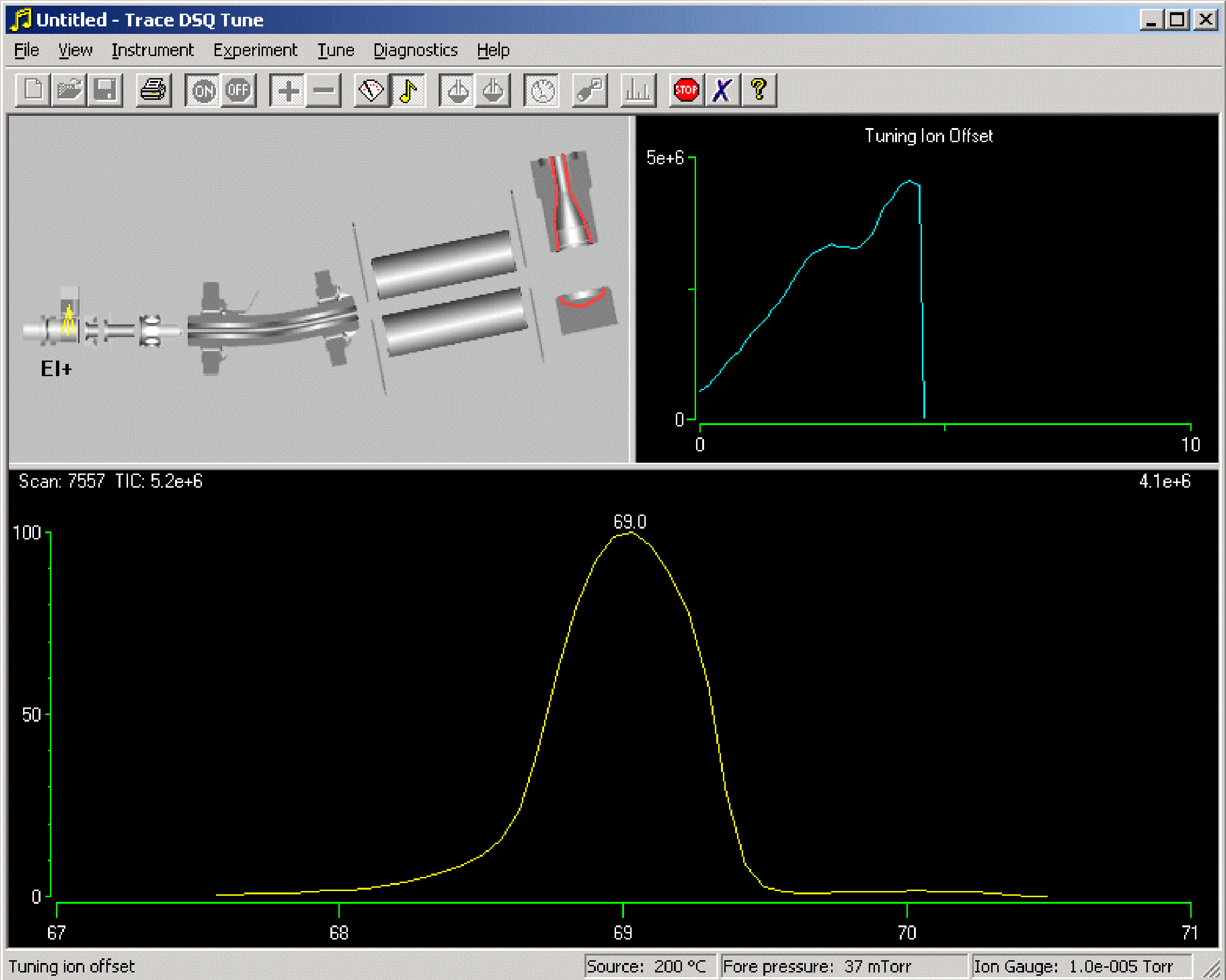
Measure Intensity and Width vs Resolution Variable



- Discard data where peak is > 0.95 amu at 10% height
- Record optimum Resolution variable for mass 69 and 502
- Apply fit to set Resolution variable in tune file

Tune Ion Offset

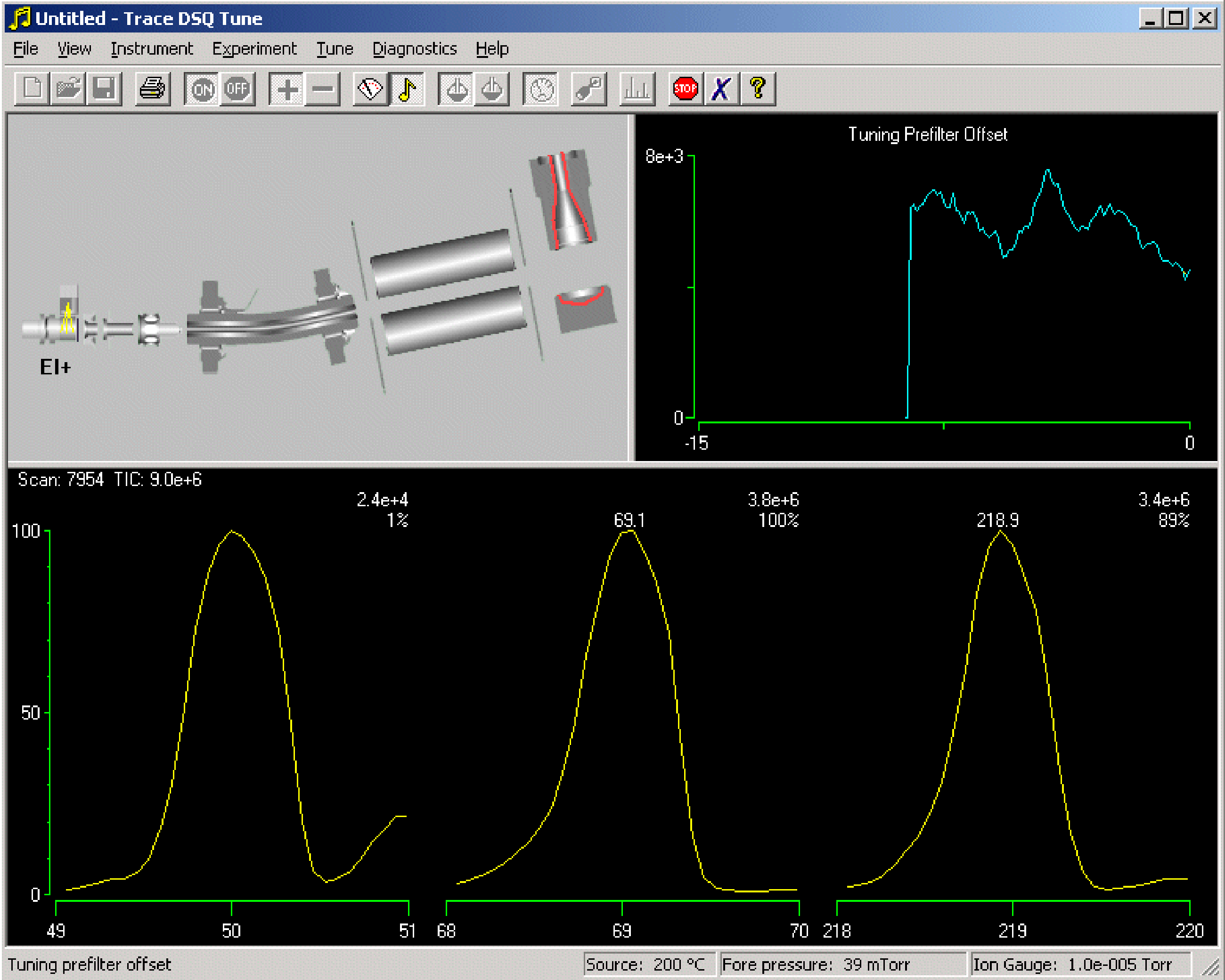
Measure Intensity and Width vs Ion Offset



- Discard data where peak is > 0.95 amu at 10% height
- Record Ion Offset variable for mass 69 and 502
- Apply fit to set Ion Offset variable in tune file

Tune Prefilter Offset

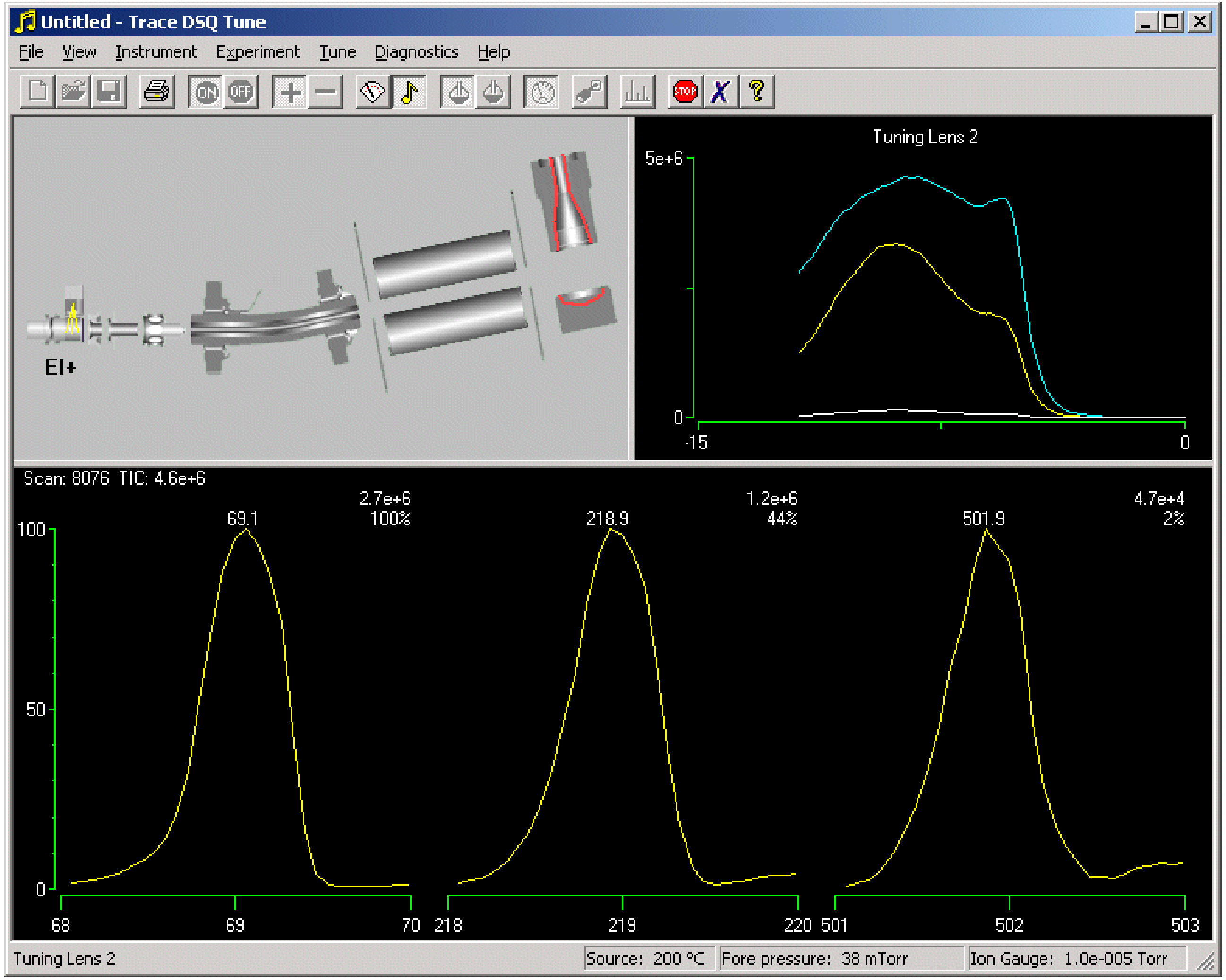
Measure Intensity and Ion Ratios vs Prefilter Offset



- Find optimum intensity of m/z 50, scaling the intensity for the peak appearance
- Discard data where m/z 219 is > 80% of m/z 69

Tune Lens 2

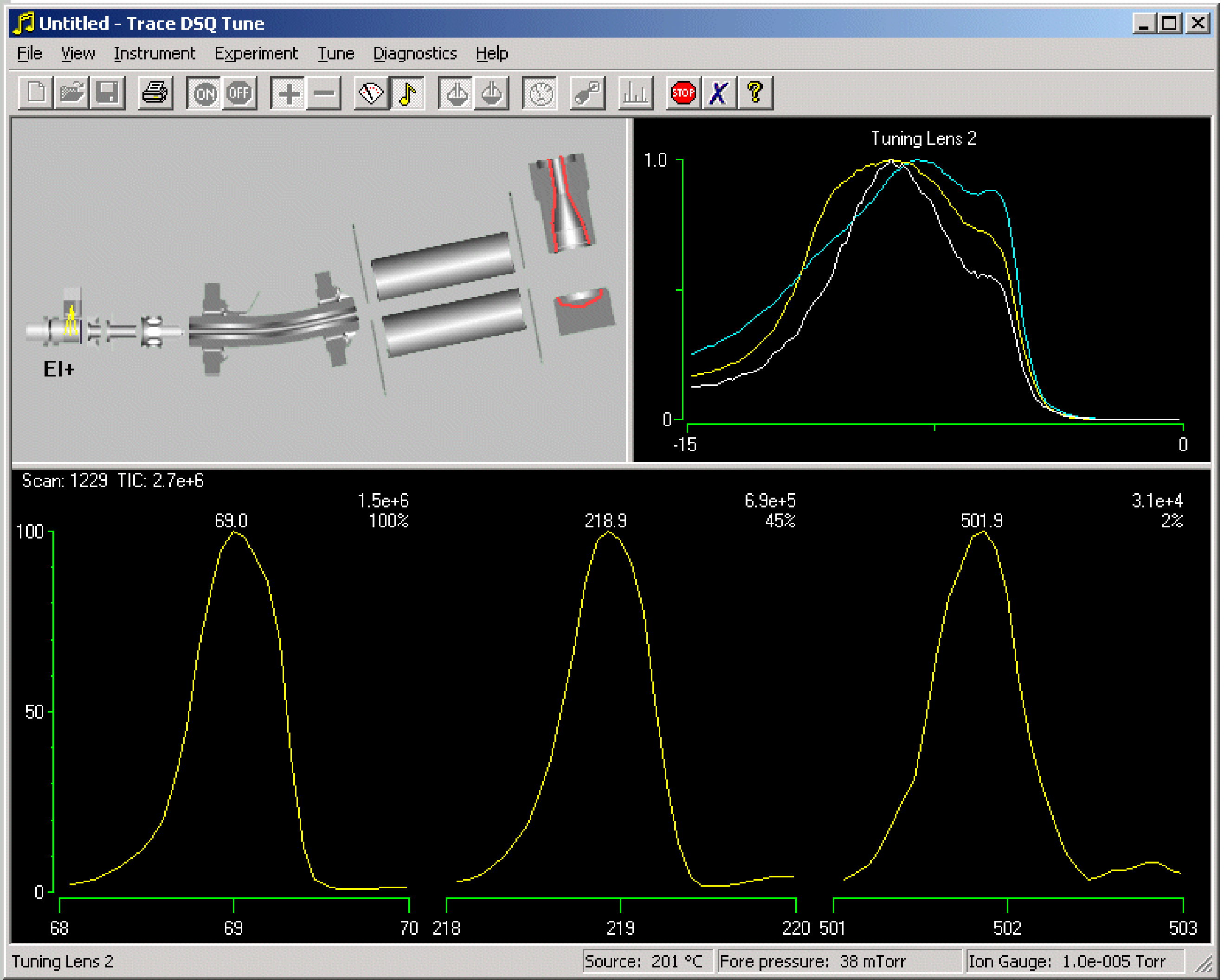
Measure Intensity and Ion Ratios vs Lens 2



- Find optimum intensity of m/z 69 and 502
- Discard data where m/z 219 is > 80% of m/z 69

Tune Lens 2

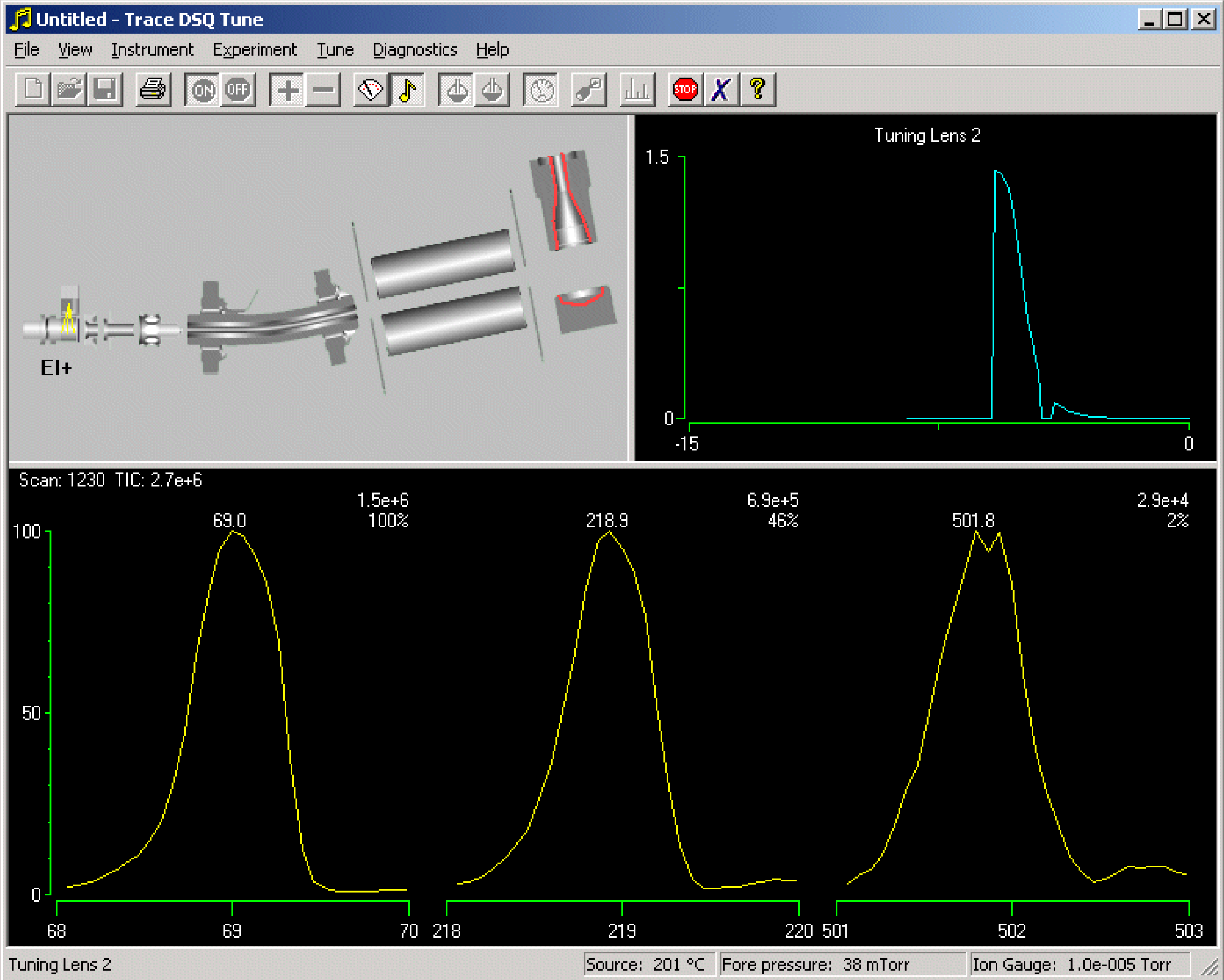
Measure Intensity and Ion Ratios vs Lens 2



- Find optimum intensity of m/z 69 and 502
- Discard data where m/z 219 is > 80% of m/z 69

Tune Lens 2

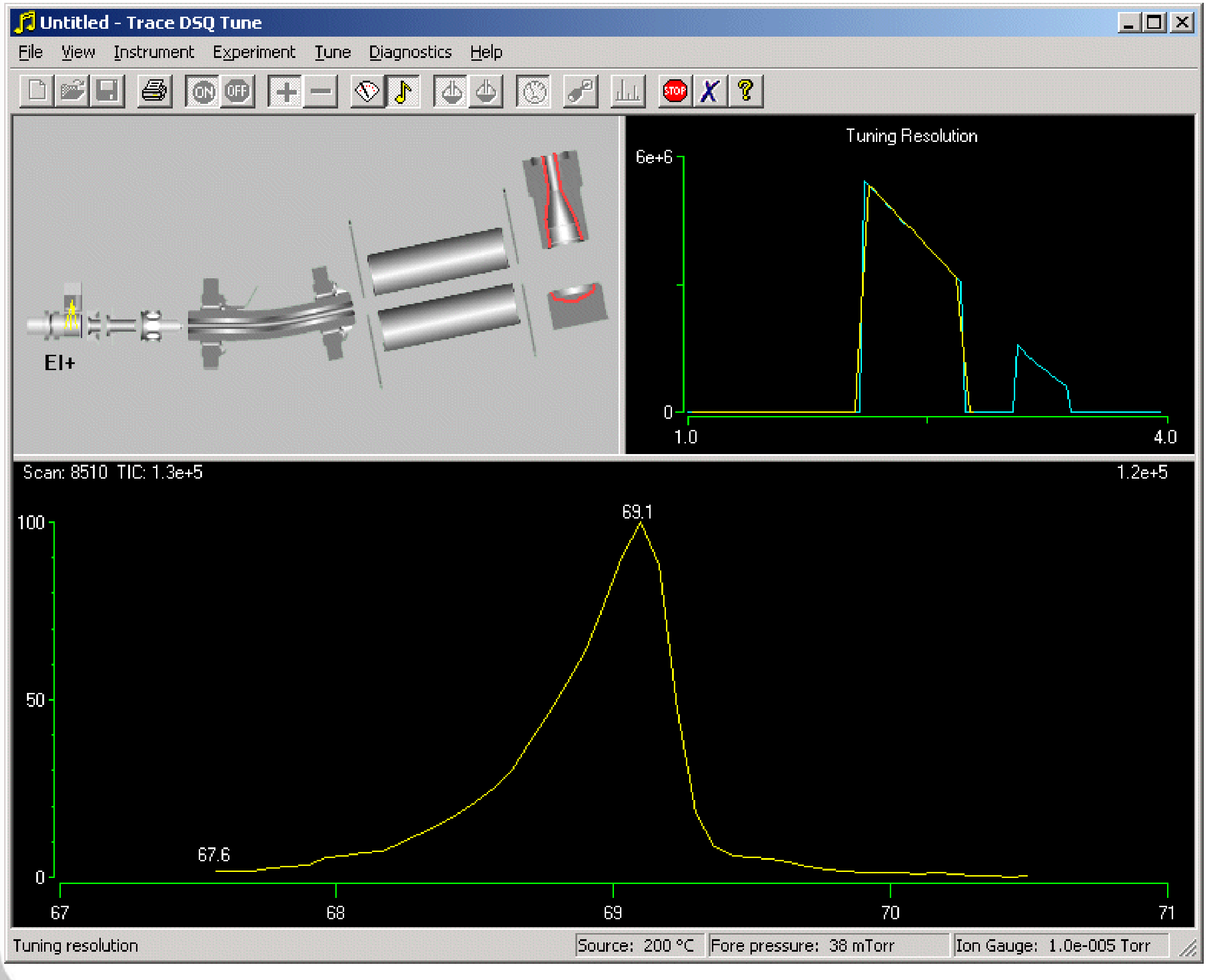
Measure Intensity and Ion Ratios vs Lens 2



- Find optimum intensity of m/z 69 and 502 by adding their normalized intensities
- Discard data where m/z 219 is > 80% of m/z 69

Tune Resolution

Measure Intensity and Width vs Resolution Variable

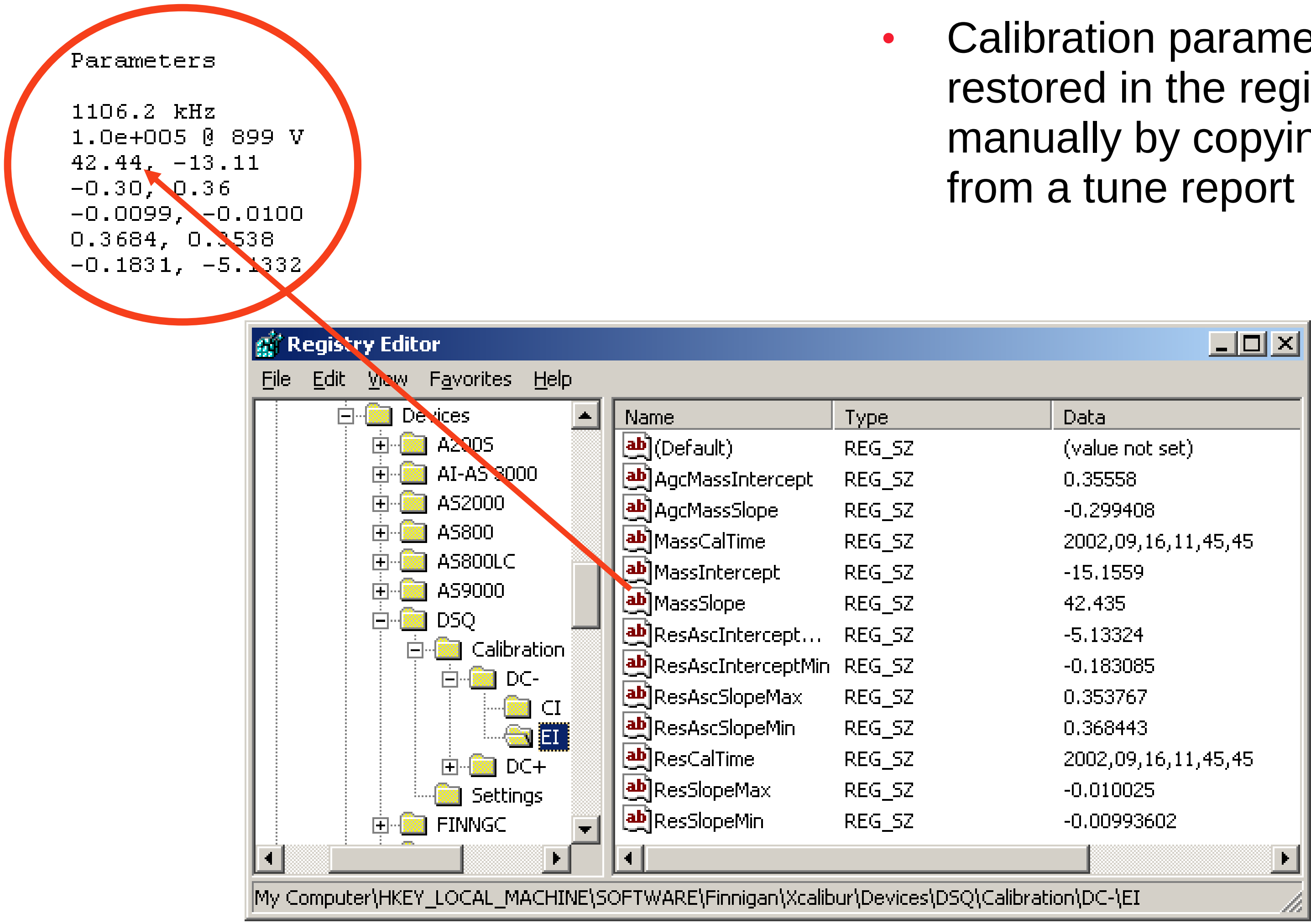


- Tune resolution using new tune values
- Tune report is generated after this tune

Automatic Tune

Tune and Calibration Report

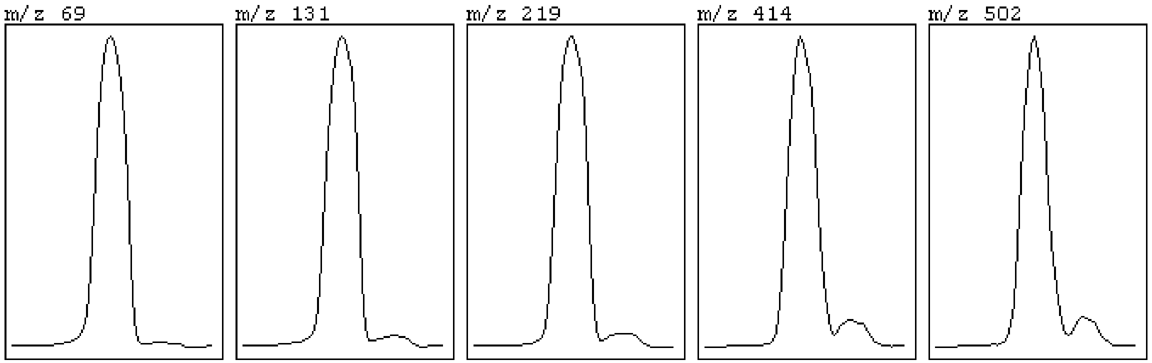
- Calibration parameters can be restored in the registry manually by copying values from a tune report



Automatic Tune

Tune and Calibration Report

Tune Report — Instrument: ULTRALIGHT
Source: EI — Rod DC: B
Mon Dec 16 14:27:43 2002 — Operator: brody



Theory	Actual	Apex	Intensity	% Base Pk	Isotope Ratio
69.0	68.9	68.9	8134299	100.0	1.2
131.0	130.9	130.9	4939338	60.7	3.2
219.0	218.9	218.9	6645213	81.7	4.1
414.0	413.9	413.9	399822	4.9	8.5
502.0	501.9	501.9	351291	4.3	9.0
614.0	614.0	614.0	37673	0.5	13.2

Automatic Tune

Calibration	Result	Parameters	Last Calibrated
RF Frequency	Not Run	1104.9 kHz	Dec 05, 2002 04:25 PM
Detector	Passed	1.0e+005 @ 1143 V	Dec 16, 2002 02:20 PM
Mass	Passed	42.99, -15.98 -0.35, 0.36	Dec 16, 2002 02:22 PM
Resolution (+)	Passed	-0.0523, -0.0543 0.3504, 0.3542 4.9730, 0.4583	Dec 16, 2002 02:22 PM
Leak Check	Passed	4.3% of reference	

Automatic Tune

Tune and Calibration Report

```
Tune file: autotune.dsqtune

Lens 1             =   -25.00 V      Ion Source Temp     =        200 C
Lens 2             =    -9.30 V      Fore Pressure       =         36 mTorr
Lens 3             =   -25.00 V      Ion Gauge Pressure  =   1.1e-005 Torr
Elec Lens          =    15.00 V
Electron Energy    =   -70.00 V
Prefilter Offset   =    -5.10 V
Emission Current   =   100.00 uA

No Scaling Factors

Mass-Resolution Tuning Factors:

   Mass =    1.0  Ion Offset = 4.0  Res. Factor = 1.7
   Mass = 1050.0  Ion Offset = 4.5  Res. Factor = 2.1
```