

General Single Ion Calibration

Pete

14-May-09

Purpose of SI calibration

- Measure the instrument response of a single ion.
- Necessary for understanding of error in instrument (counting statistics)
- Calculation of AB and IE
- The value is important, but cancels out in the calculation of mass.

How to Do SI calibration

- m/z calibration
- baseline / threshold calibration
- check for saturation
- look at SI values
- thresholding (advanced diagnostics)
- m/z ratio - in particular 40/28
- if on a HR-ToF-AMS do it for BOTH modes

m/z calibration

ToF-AMS DAQ

M1 : V-TOF : EI

5/14/2009 8:27:20 AM

ToF-AMS DAQ

Data Acquisition and Instrument Control Software
3.0.3 (06-March-2009)

Researcher(s) Pete

Experiment Training

Menus < >

Acquire PTOF MS BFSP

Calibrate Servo BitWise

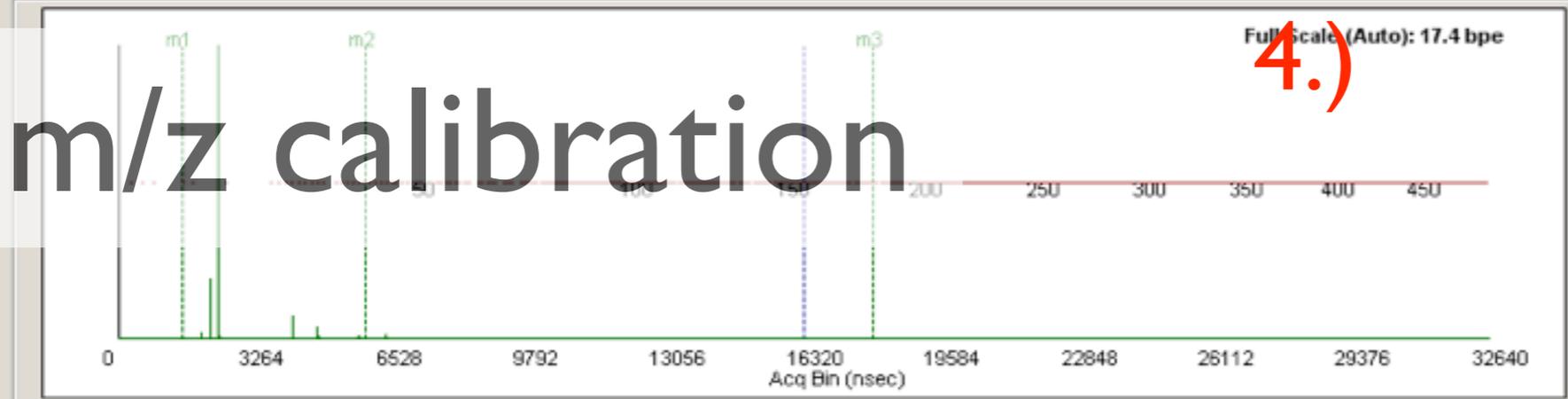
Flow Rate (cm3/s): -0.38 Fil. Emission (mA): 0.95 Heater T (C): 606 Chopper (Hz): 0.0

TPS

Nbr	Name	Ion	TOF	Modes	Save	Switch
1	V_EI_pos	EI	V	M.P.	150.0	Y
2	W_EI_po	EI	W	M.B.	375.0	Y
3	V_Drive	EI	V	M.P.	5.0	
4	V_pos_F	EI	V	M.P.	120.0	
5	W_pos_F	EI	W	M.	120.0	
6	V_MS_6s	EI	V	M.P.	60.0	
7	V_MST_	EI	V	M.P.	60.0	
8	W_1m	EI	W	M.	12.0	
9	IECal_V	EI	V	M.P.B.	100.0	
10	IE_W	EI	W	M.P.	60.0	

5/13/2009 5:58:06 PM: Menu 1 to TPS-RC
5/13/2009 5:12:16 PM: Backed-up Menu(s): 1; 2; 3; 4; 5; 6; 7; 8; 9; 10;

- the m/z calibration window is also where you can tune the instrument



m/z calibration

4.)

26548 ns || m/z 349.13 || 3.3 bpe

Intensity: Lin Log Auto

Time: x2 x10 FS 10000

Chopper:

Calibrate Tune 1.)

14
5.0e-2 bpe
A: 0.15
R: 1422
S: 0.18

m/z 14.0031
A. Bin 1529

40
4.4e-2 bpe
A: 0.16
R: 2008
S: 0.41

m/z 39.9624
A. Bin 5848

184
1.0e-2 bpe
A: 0.05
R: 3520
S: 0.29

m/z 183.9509
A. Bin 17972

2.)

m/z 14.003 || -16 ppm
m/z 39.962 || 12 ppm
m/z 183.951 || -2 ppm

3.)

Revert to Saved Calibration
Save Current Calibration

$m = 1674.30$
 $b = -4736.32$
m/z Range: 9 to 497

Acq. Bin = $m \cdot \sqrt{m/z} + b$

4.0026	He
7.016003	Li
9.012182	Be
12	C
13.00336	j13C
13.00782	CH
13.00746	COnline?

Auto Calibrate During Acquisition

1. Line up the masses in the 3 boxes using the arrows
2. Check that the points fall in or close to the blue dotted lines
3. Check that the points fall on a line
4. If all is okay, press "save all and exit"
5. If the points are not on a line, then one of the masses is probably off.

Bitwise window

ToF-AMS DAQ
Data Acquisition and Instrument Control Software
3.0.3 (06-March-2009)

Researcher(s)
Experiment

Menus < >

Acquire PTOF MS BFSP

Calibrate Servo **BitWise**

Flow Rate (cm³/s): -0.38 Fil. Emission (mA): 0.95 Heater T (C): 606 Chopper (Hz): 0.0

TPS

Nmbr	Name	Ion	TOF	Modes	Save	Switch
1	V_EI_pos	EI	V	M.P.	150.0	Y
2	W_EI_po	EI	W	M.B.	375.0	Y
3	V_Drive	EI	V	M.P.	5.0	
4	V_pos_F	EI	V	M.P.	120.0	
5	W_pos_F	EI	W	M.	120.0	
6	V_MS_6s	EI	V	M.P.	60.0	
7	V_MST_	EI	V	M.P.	60.0	
8	W_1m	EI	W	M.	12.0	
9	IECal_V	EI	V	M.P.B.	100.0	
10	IE_W	EI	W	M.P.	60.0	

5/13/2009 5:58:06 PM: Menu 1 to TPS-RC
5/13/2009 5:12:16 PM: Backed-up Menu(s): 1; 2; 3; 4; 5; 6; 7; 8; 9; 10;

- The bitwise window is where one can check the baseline, thresholding, Saturation, and single ion

Baseline / Threshold

The screenshot shows the BitWise 3.0.3 software interface. The top bar displays 'M1 : V-TOF : EI', 'Group 1', and 'Advanced Diagnostics'. The main control panel includes 'Start', 'Stop', and 'Set Baseline' buttons, which are highlighted with red boxes and labeled '2.)' and '3.)'. The MCP control panel shows 'Low' and 'Menu Val' buttons, also highlighted with red boxes and labeled '1.)' and '4.)'. The MCP panel displays 'Set (V): 2360' and 'Read (V): 2364'. The 'PASS' and 'TOTAL' sections show 'bits' and 'mV' values. The 'Single Extraction Peaks' section shows a graph with a yellow bar and 'Peaks: 0'. The 'AP240 CH1' section shows 'Full Scale (V): 0.20', 'Baseline (mV): -21.4302', 'Baseline (Bits): 13', and 'Threshold: 4 -24.56 mV'. The 'Acquisition Range' is '-0.011 to -0.211 V'. The 'Single Ion' section is partially visible at the bottom.

1. Set the MCP value to "Low" which = 1000 V (wait until value read value reaches 1000)
2. Press Start and wait for at least 30 passes to complete then press stop.
3. Inspect the baseline, if all appears okay, then press "Set Baseline"
4. Reset the MCP to the "Menu Val" wait for the Read value to reach the set value, and then wait for another minute or so.

M1 : V-TOF : EI

Group 1
1 2 3 4 5
6 7 8 9 10

Note Capture

Baseline & Peaks MS: Ratios

Electronic Baseline

Baseline Check Output

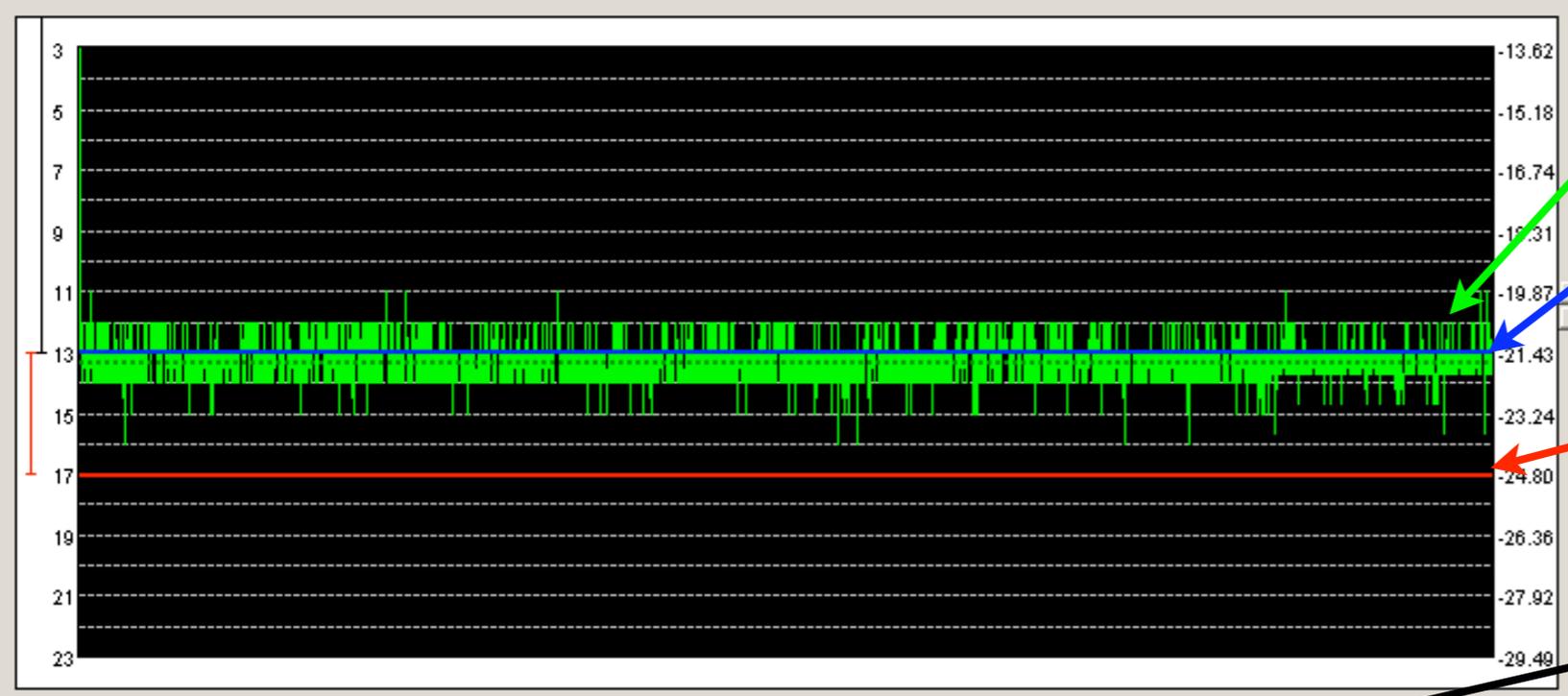
Start
Stop
Set Baseline

	PASS	TOTAL
Passes Completed: 54	13.31 bits (0.72)	13.31 bits (0.03)
	-21.67 mV (0.57)	-21.67 mV (0.03)

MCP

Low Menu Val -10V
+10V

Set (V): 1000
Read (V): 1007



Menu Baseline: -21.7 mV Menu Thresh: 4 Bits / 3.1 mV 0 V

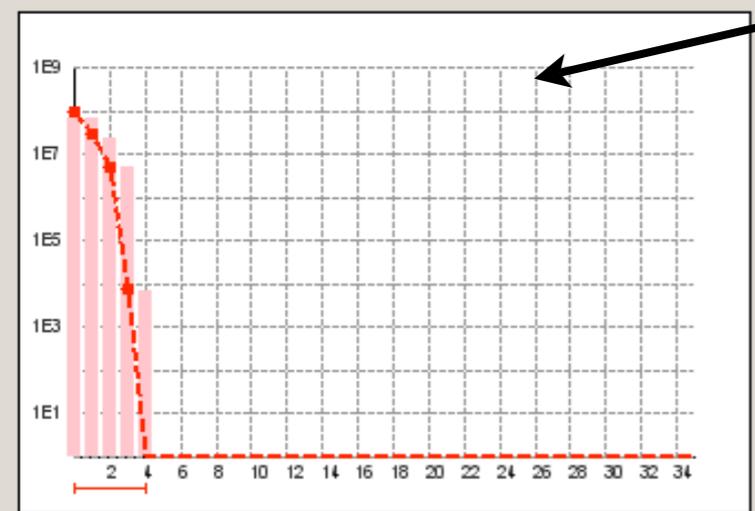
raw electronic baseline

Set electronic baseline in ADC
Blue line should be in center of green trace

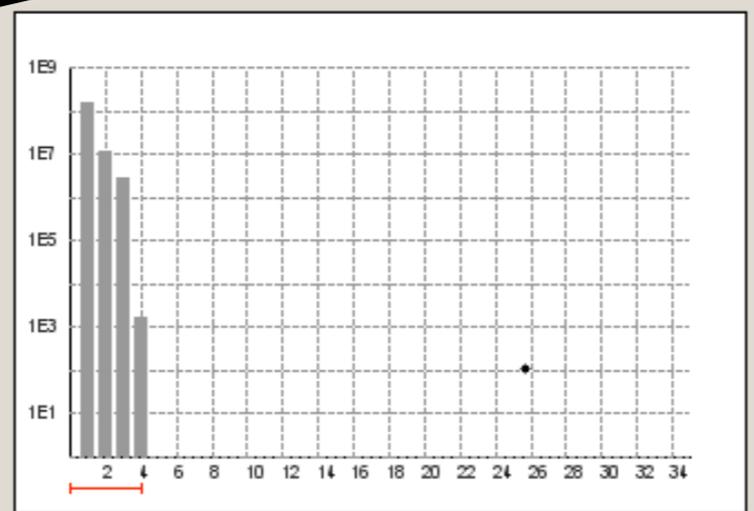
Set Threshold value in ADC

This graph shows the electronic noise around the baseline. In general one does not want vertical bars exceeding your threshold value red line at bottom. One should *not* adjust the baseline to prevent this, as that can reduce the linearity of the ion measurement and compromise data quality. The next tests guide ones decision on the threshold value

Threshold Breaker Frequency (Hz)



Scattered Ion Height Distribution (Hz)



Check for Saturation and SI setup

1. Check the "Advanced Diagnostics" box
2. I generally like to set the Pk. Discrim. above the Threshold value by 1-2 bits (Play with this and see how it affects the SI value)
3. Click "D" in the SI masses. I often remove inorganic ions such as 46, 64, and some lower masses such as 14
4. Make sure the Chopper is open and the valve is too, since we first check for Saturation of the detector
5. Press Start - then the graphs will appear, and you will start to see the SI value calculated.

Baseline & Peaks | MS: Ratios | Peaks: Threshold

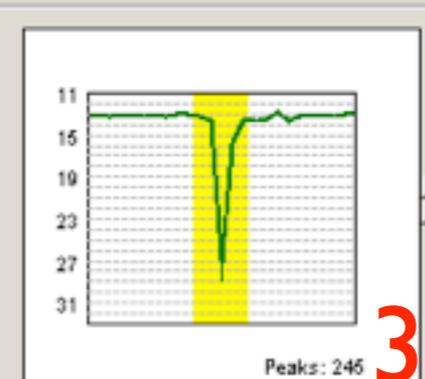
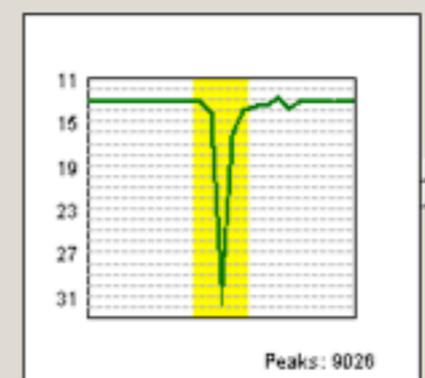
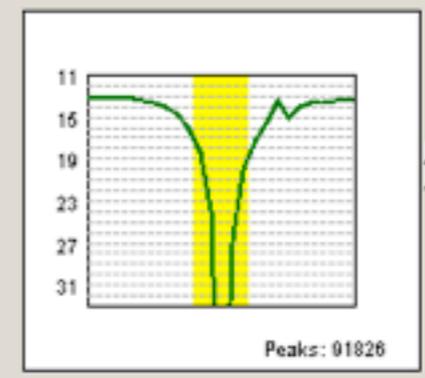
Electronic Baseline | Spectral Baseline

Start
Stop
Set Baseline

PASSES TOTAL
Passes Completed: 51 23.0 bits (0.72) 23.0 bits (0.03)
-21.67 mV (0.57) -21.67 mV (0.03)

MCP
Low Menu Val -10V +10V
Set (V): 2360
Read (V): 2364

Single Extraction Peaks



AP240 CH1
Full Scale (V) 0.20
Baseline (mV) -21.6733
Baseline (Bits) 13
Threshold 4 -24.80 mV
Acquisition Range: -0.012 to -0.212 V

Single Ion 24.9 bit-n

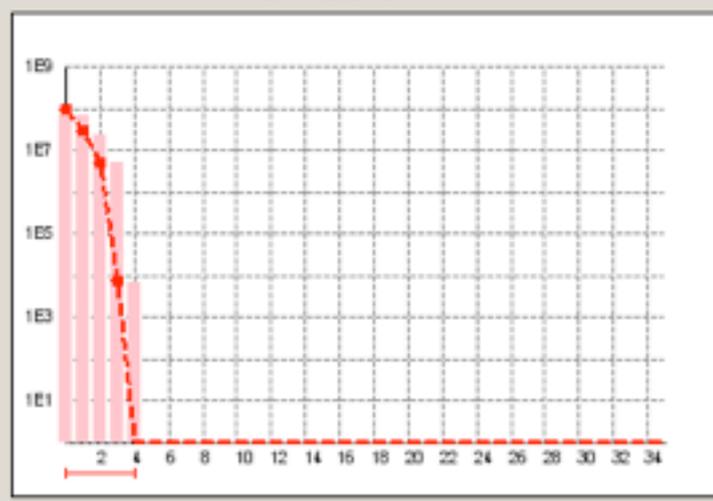
Peak Analysis
m/z Pk. Discrim.
28 6
40 6
184 6

m/z 28
Avg Pk Area: 79.3
Pk Prob: 0.9575
Pk/Noise: --
Max Bit Rec: 266
Sat Prob: 0.002

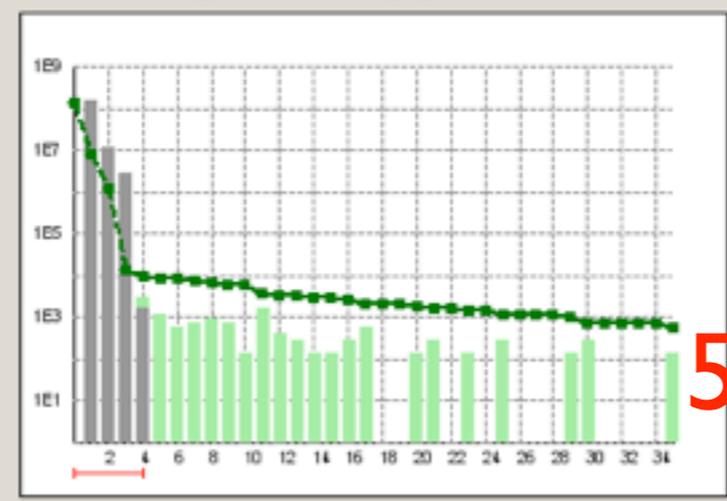
m/z 40
Avg Pk Area: 22.4
Pk Prob: 0.0941
Pk/Noise: --
Max Bit Rec: 106

m/z 184
Avg Pk Area: 18.3
Pk Prob: 0.0026
Pk/Noise: --
Max Bit Rec: 70

Threshold Breaker Frequency (Hz)



Scattered Ion Height Distribution (Hz)



SI Masses [D]
12,19,20,25,26,27,31,34,35,36,37,38,40,
42,43,45,50,51,52,53,54,55,56,57,58,63

Start
Stop
Save HDF

Integration Area (nsec) 5

AP240 Thresh.
Applied 0

Chopper
Open Blocked

1.)

2.)

3.)

5.)

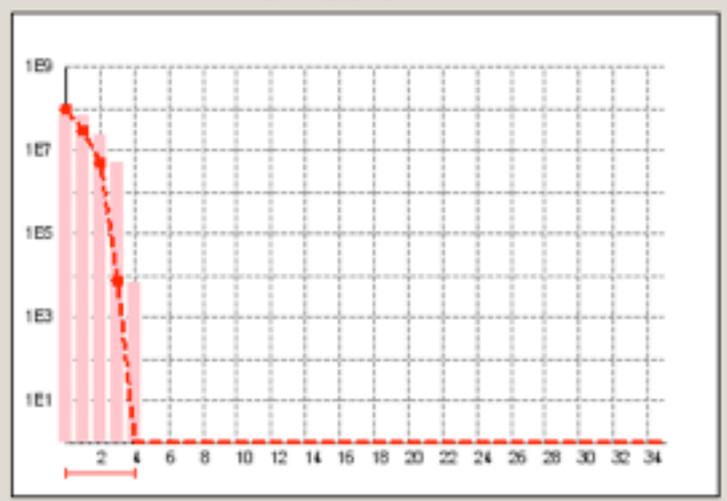
4.)

Recalculate SI with blocked if in V mode

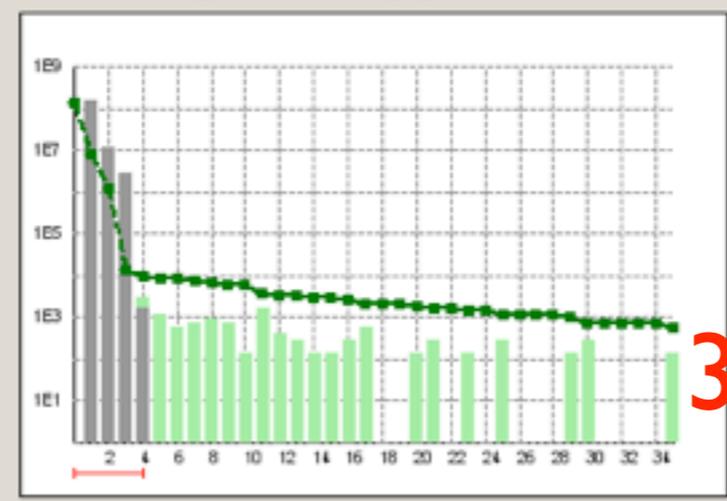
1. See how frequently the m/z 28 signal saturated the ADC card. If the probability is $\ll 1\%$ this should be okay.
2. If in V-Mode changing the chopper value to blocked is recommended for the SI determination. If in W-mode, one does not need to do this, and can skip the the next slide.
3. Press Start - then the graphs will appear, and you will start to see the SI value calculated.

Menu Baseline: -21.7 mV Menu Thresh: 4 Bits / 3.1 mV 0 V

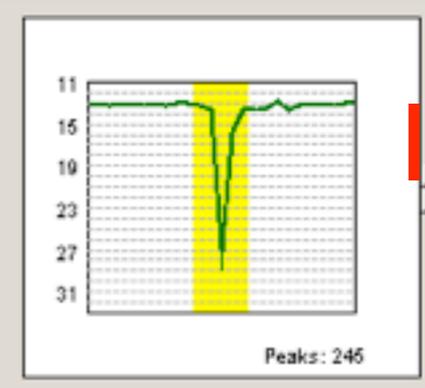
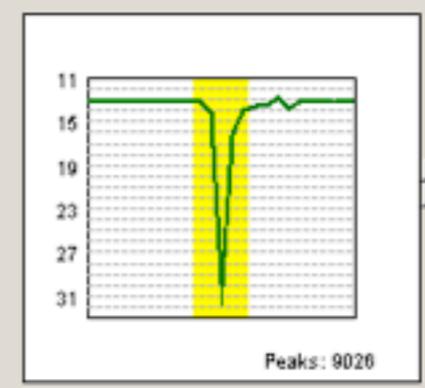
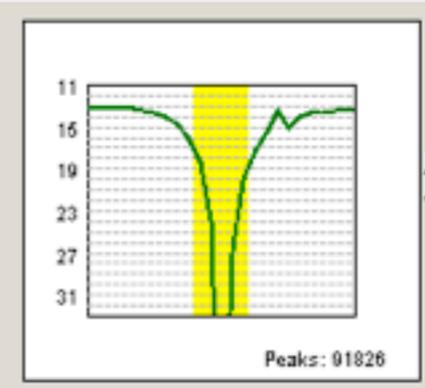
Threshold Breaker Frequency (Hz)



Scattered Ion Height Distribution (Hz)



Single Extraction Peaks



SI Masses [D]
 12,19,20,25,26,27,31,34,35,36,37,38,40,
 42,43,45,50,51,52,53,54,55,56,57,58,63

36492 Peaks / 23.0 bit-ns

Start (dashed box)
 Stop
 Save HDF

Integration Area (nsec) [5]

AP240 Thresh.
 Applied [0]

Playback, Pause, Stop

AP240 CH1

Full Scale (V) [0.20]

Baseline (mV) [-21.6733]

Baseline (Bits) [13]

Threshold [4] -24.80 mV

Acquisition Range: -0.012 to -0.212 V

Single Ion [24.9] bit-ns

Peak Analysis

m/z	Pk. Discrim.
[28]	[6]
[40]	[6]
[184]	[6]

m/z 28

Avg Pk Area: 79.3
 Pk Prob: 0.9575

Pk/Noise: --
 Max Bit Rec: 266
 Sat Prob: 0.002

m/z 40

Avg Pk Area: 22.4
 Pk Prob: 0.0941

Pk/Noise: --
 Max Bit Rec: 106

m/z 184

Avg Pk Area: 18.3
 Pk Prob: 0.0026

Pk/Noise: --
 Max Bit Rec: 70

Chopper [Open] [Blocked] (dashed box)

3.)

2.)

Reading the Values

Start

Stop

Set Baseline

Passes Completed: 54

PASS

13.31 bits (0.72)

-21.67 mV (0.57)

TOTAL

13.31 bits (0.03)

-21.67 mV (0.03)

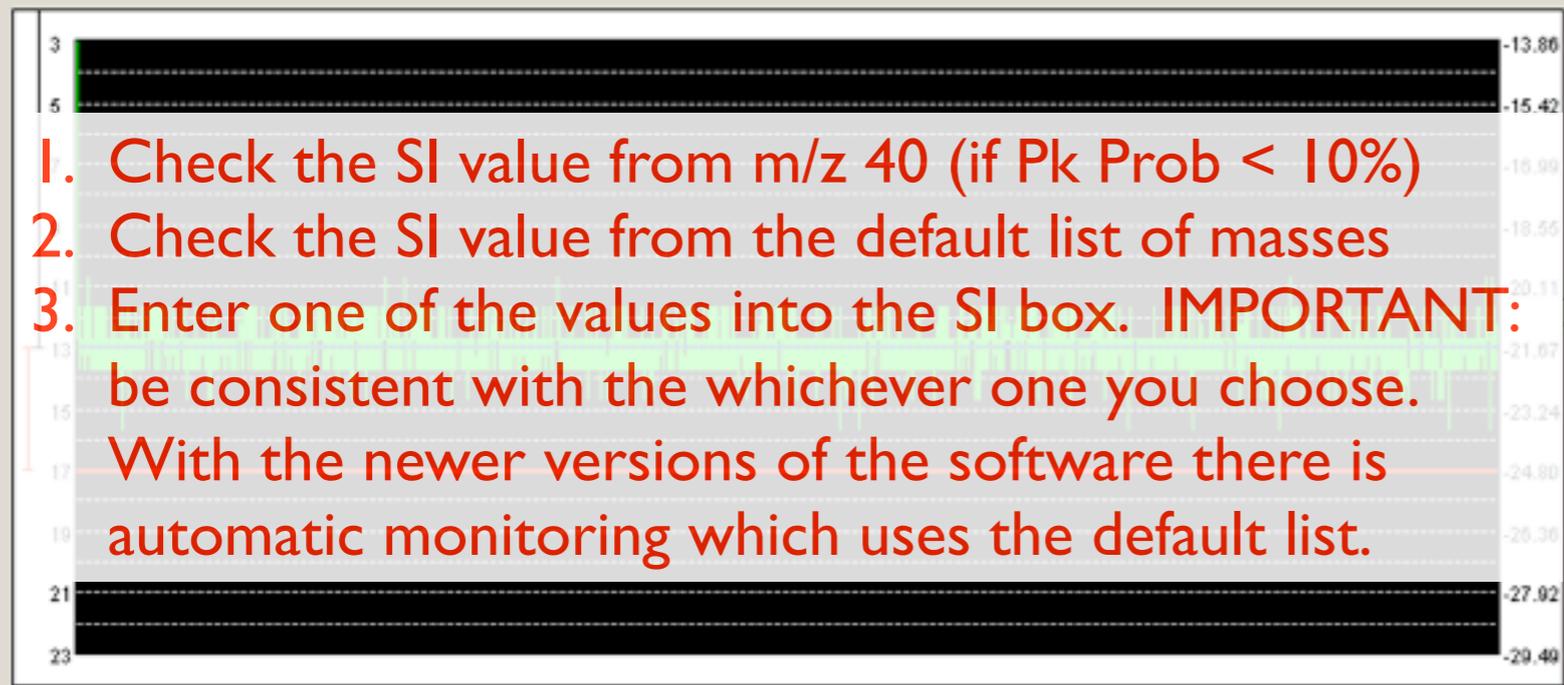
MCP

Low

Menu Val -10V +10V

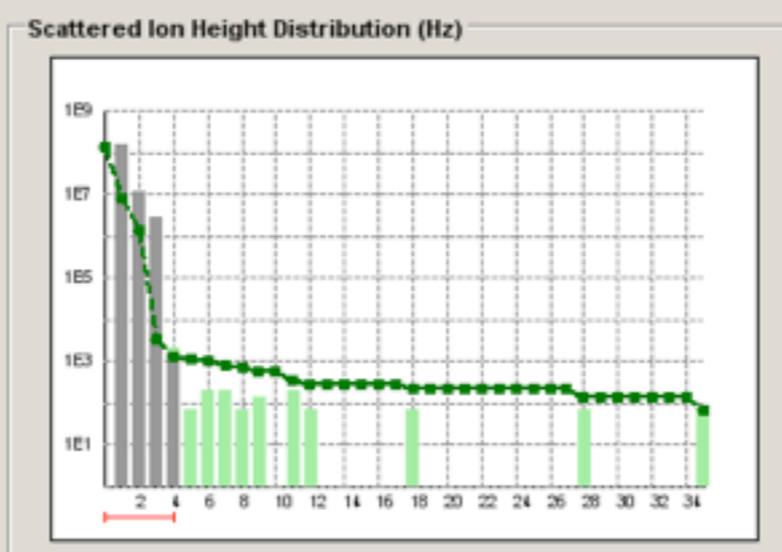
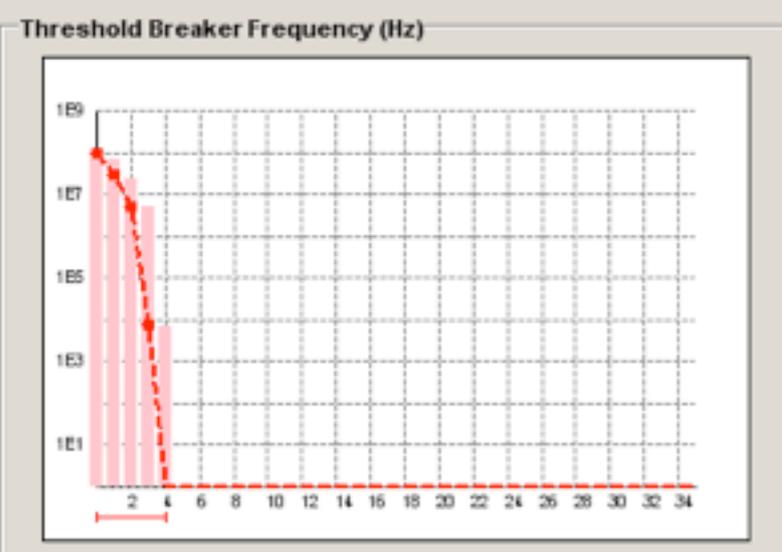
Set (V): 2360

Read (V): 2364

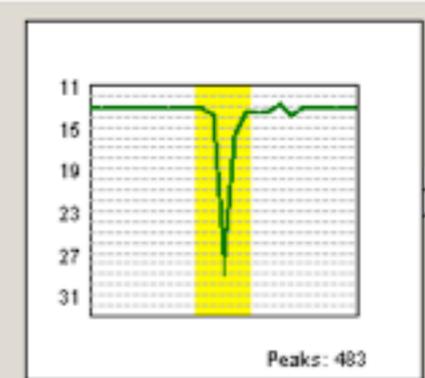
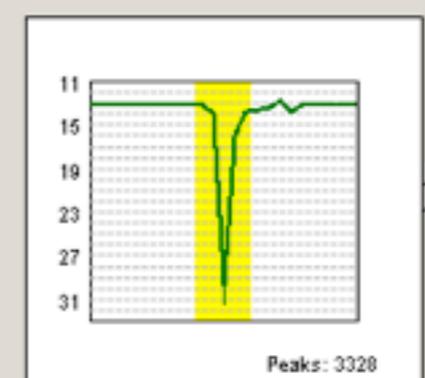
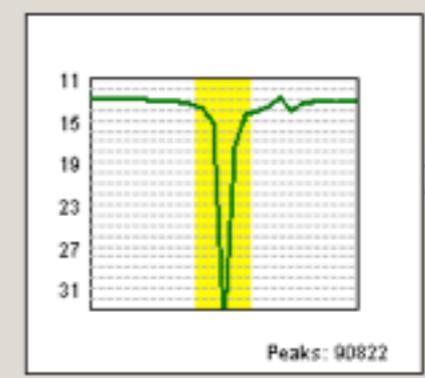


1. Check the SI value from m/z 40 (if Pk Prob < 10%)
2. Check the SI value from the default list of masses
3. Enter one of the values into the SI box. **IMPORTANT:** be consistent with the whichever one you choose. With the newer versions of the software there is automatic monitoring which uses the default list.

Menu Baseline: -21.7 mV Menu Thresh: 4 Bits / 3.1 mV 0 V



Single Extraction Peaks



SI Masses

12,19,20,25,26,27,31,34,35,36,37,38,40,42,43,45,50,51,52,53,54,55,56,57,58,63

20616 Peaks / 22.4 bit-ns

Start

Stop

Save HDF

Integration Area (nsec) 5

AP240 Thresh.

Applied 0

AP240 CH1

Full Scale (V) 0.20

Baseline (mV) -21.6733

Baseline (Bits) 13

Threshold 4 -24.80 mV

Acquisition Range: -0.012 to -0.212 V

Single Ion 22.4 bit-ns

Peak Analysis

m/z	Pk. Discrim.
28	6
40	6
184	6

m/z 28

Avg Pk Area: 30.9

Pk Prob: 0.4578

Pk/Noise: --

Max Bit Rec: 144

m/z 40

Avg Pk Area: 21.4

Pk Prob: 0.0168

Pk/Noise: --

Max Bit Rec: 81

m/z 184

Avg Pk Area: 18.8

Pk Prob: 0.0024

Pk/Noise: --

Max Bit Rec: 71

Chopper

Open Blocked

3.)

1.)

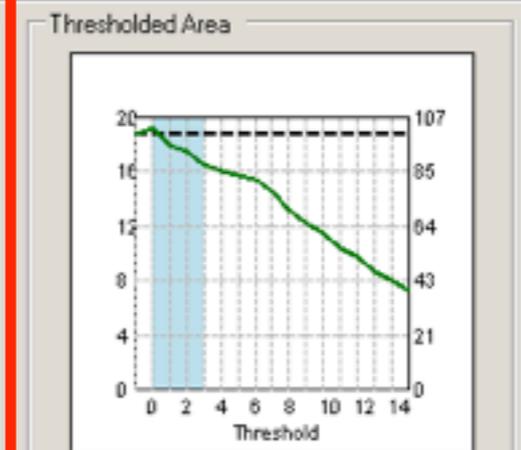
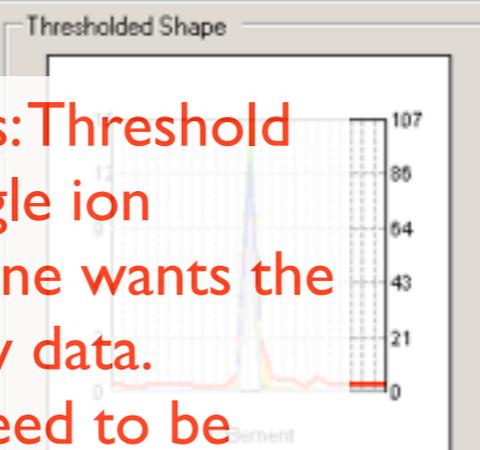
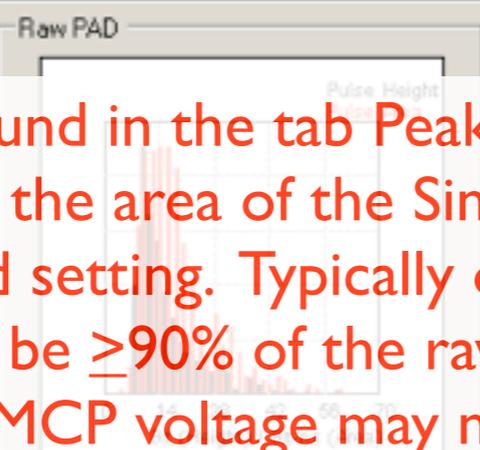
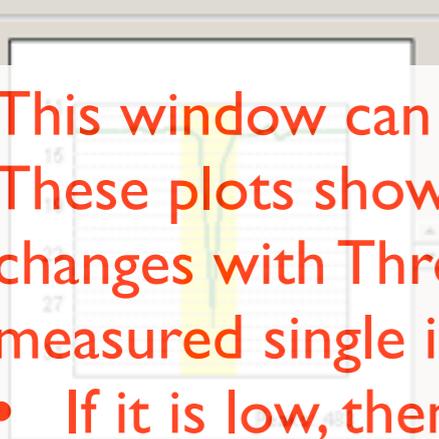
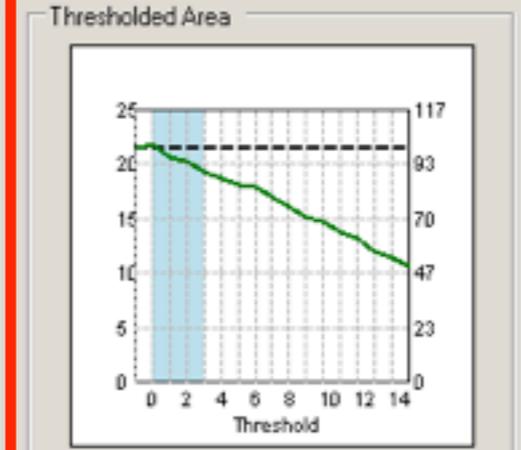
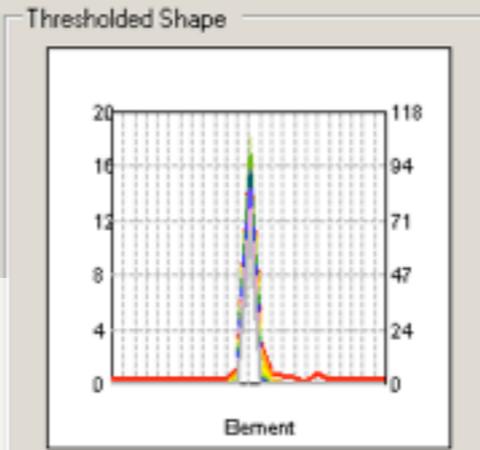
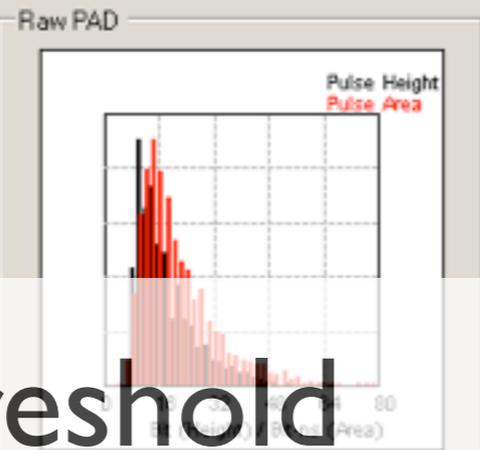
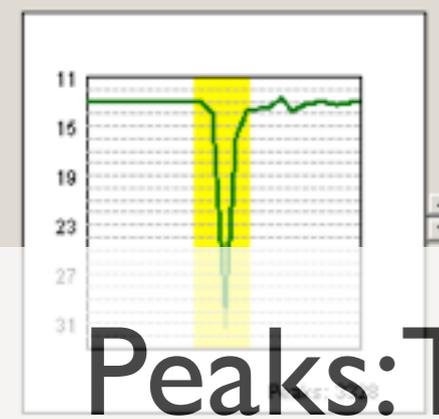
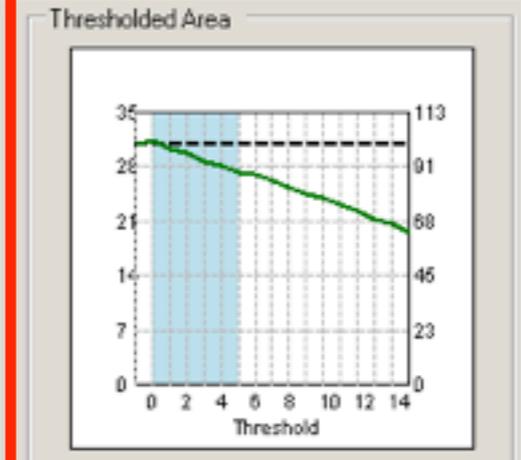
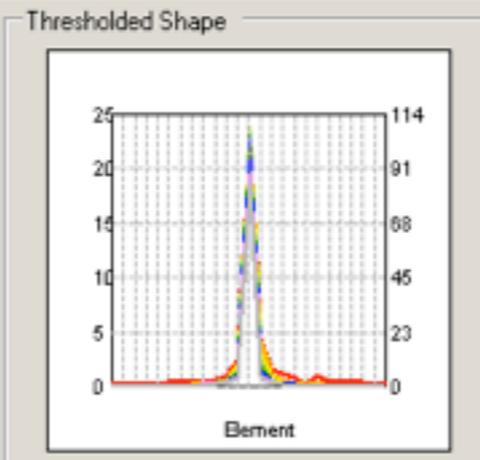
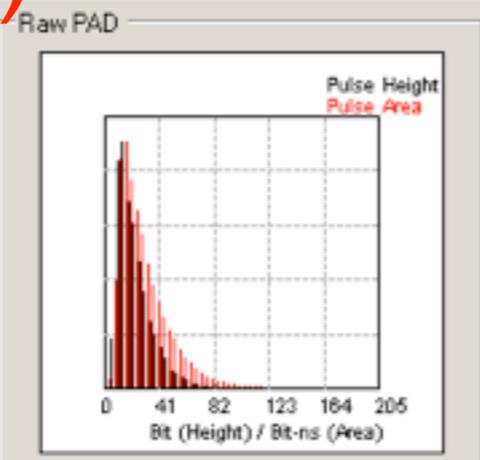
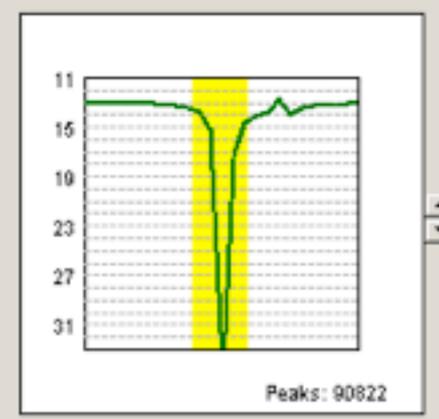
2.)

2.)

Peaks: Threshold

1.)

Single Extraction Peaks



Peaks: Threshold

AP240 CH1

Full Scale (V) 0.20

Baseline (mV) -21.6733

Baseline (Bits) 13

Threshold 4 -24.80 mV

Acquisition Range: -0.012 to -0.212 V

Single Ion 22.4 bit-ns

Peak Analysis

m/z	Pk. Discrim.
28	6
40	6
184	6

m/z 28

Avg Pk Area: 30.9
Pk Prob: 0.4578

Pk/Noise: --
Max Bit Rec: 144

m/z 40

Avg Pk Area: 21.4
Pk Prob: 0.0168

Pk/Noise: --
Max Bit Rec: 81

m/z 184

Avg Pk Area: 18.8
Pk Prob: 0.0024

Pk/Noise: --
Max Bit Rec: 71

Chopper

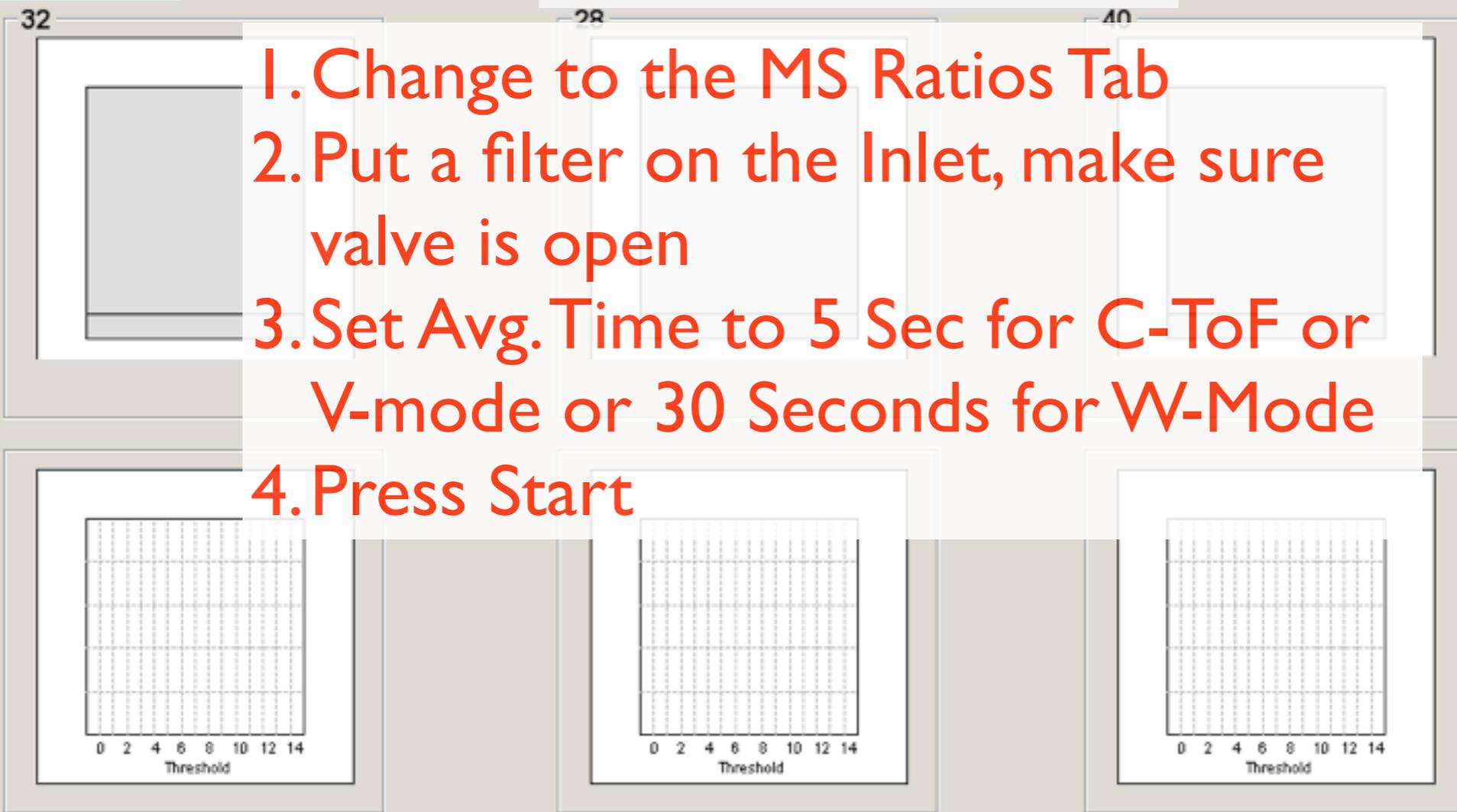
Open Blocked

- This window can be found in the tab Peaks: Threshold
- These plots show how the area of the Single ion changes with Threshold setting. Typically one wants the measured single ion to be $\geq 90\%$ of the raw data.
 - If it is low, then the MCP voltage may need to be increased.

m/z ratios

1.)

1. Change to the MS Ratios Tab
2. Put a filter on the Inlet, make sure valve is open
3. Set Avg. Time to 5 Sec for C-ToF or V-mode or 30 Seconds for W-Mode
4. Press Start

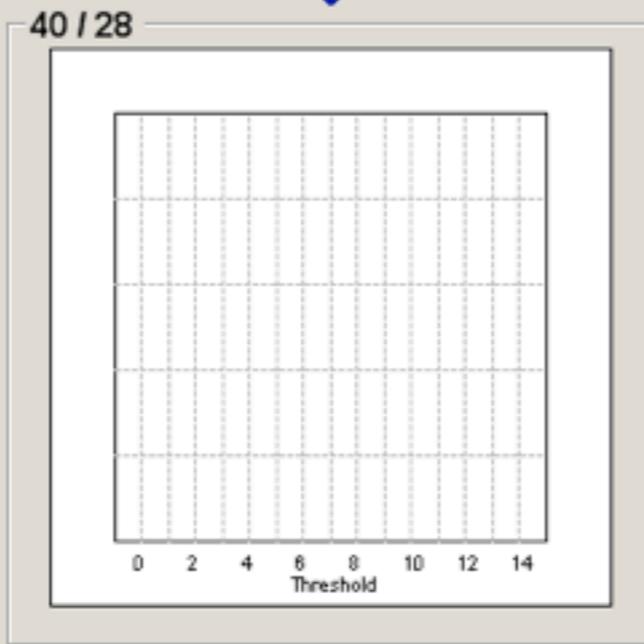
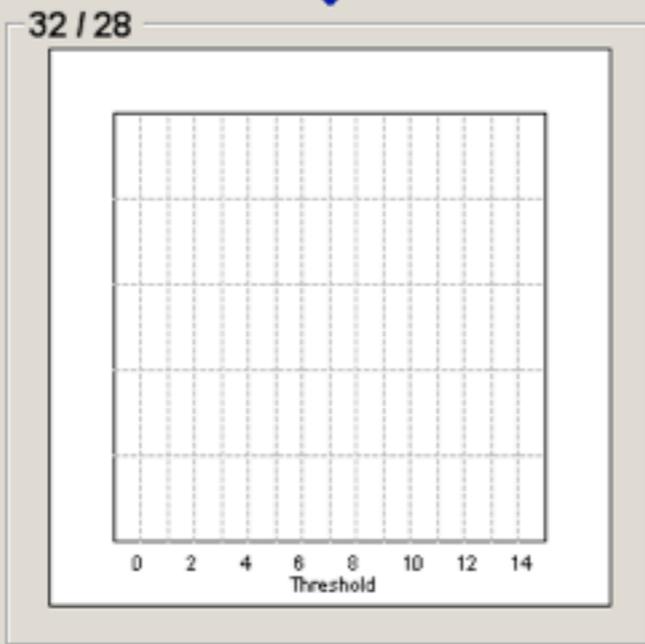


4.) 3.)

Start

Avg. Time: 5 sec

IDLE



AP240 CH1

Full Scale (V): 0.20

Baseline (mV): -21.6733

Baseline (Bits): 13

Threshold: 4 -24.80 mV

Acquisition Range: -0.012 to -0.212 V

Single Ion: 22.4 bit-ns

Peak Analysis

m/z	Pk. Discrim.
28	6
40	6
184	6

m/z 28

Avg Pk Area: 30.9

Pk Prob: 0.4578

Pk/Noise: --

Max Bit Rec: 144

m/z 40

Avg Pk Area: 21.4

Pk Prob: 0.0168

Pk/Noise: --

Max Bit Rec: 81

m/z 184

Avg Pk Area: 18.8

Pk Prob: 0.0024

Pk/Noise: --

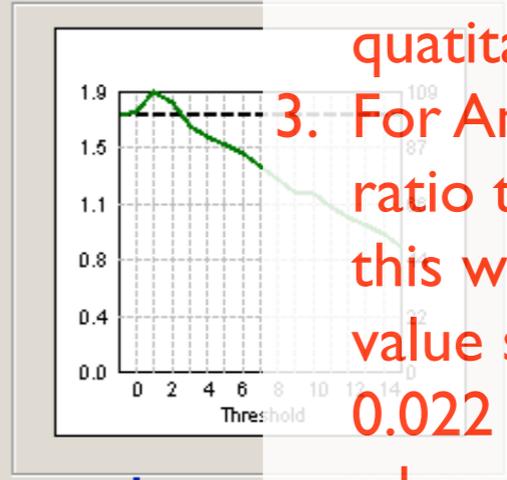
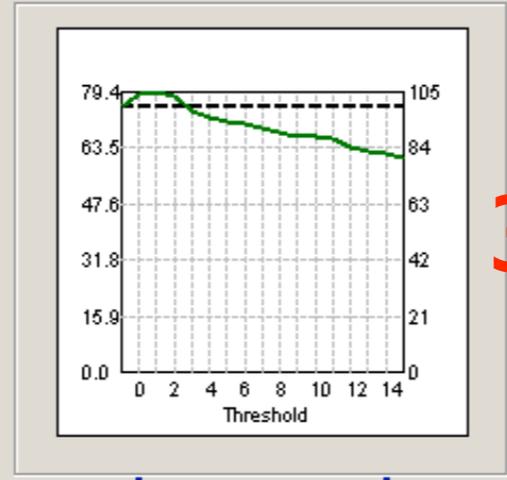
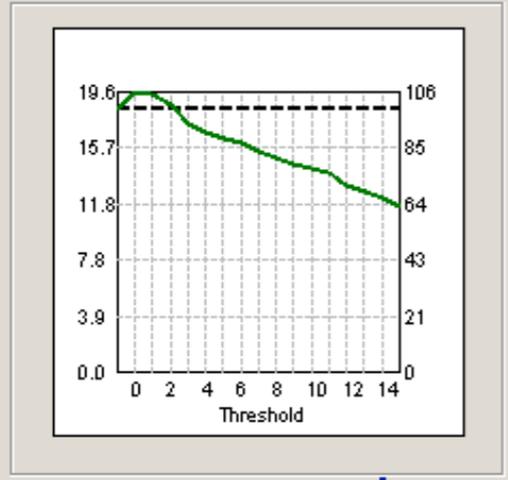
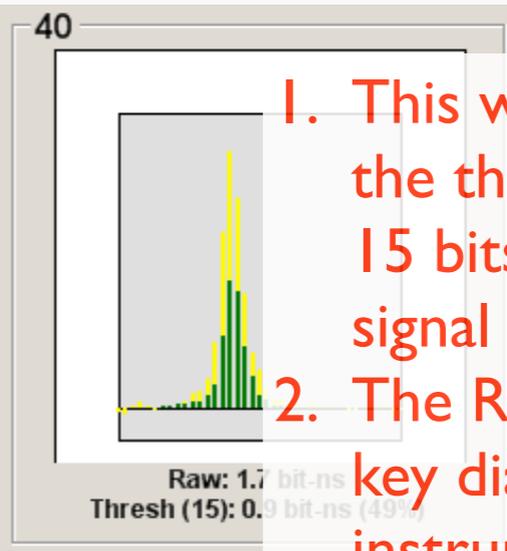
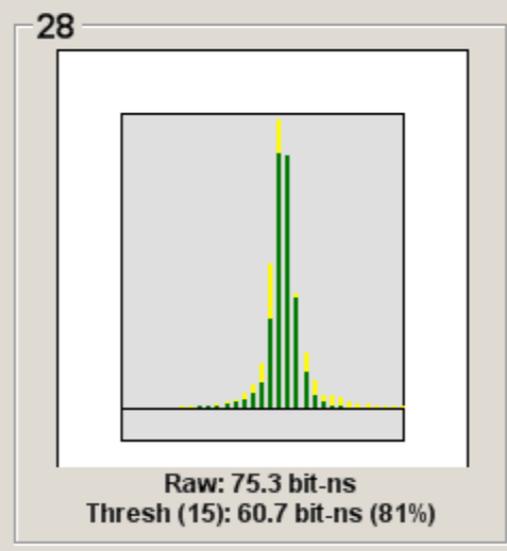
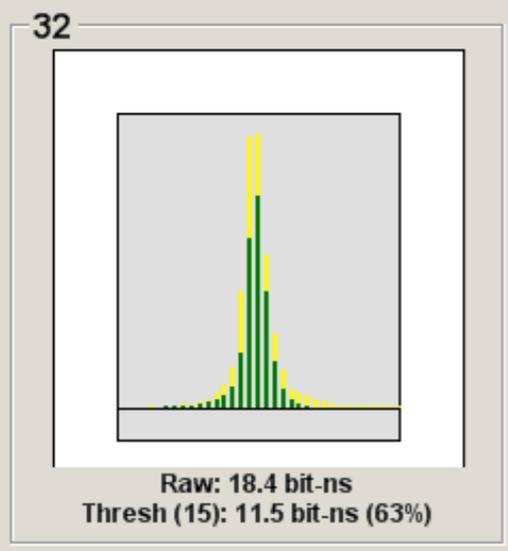
Max Bit Rec: 71

Chopper

Open Blocked

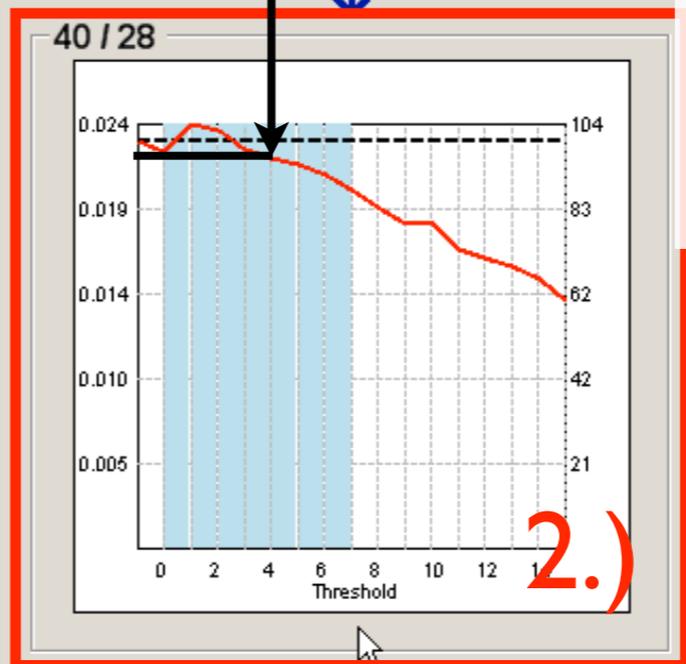
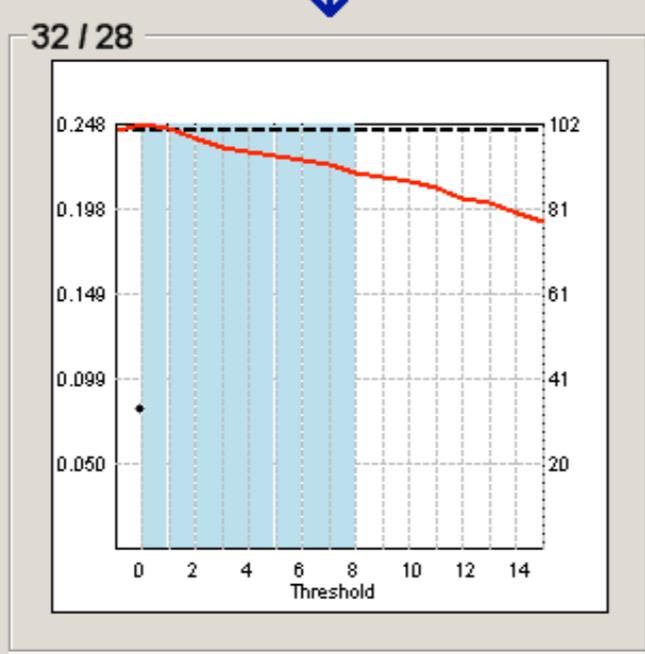
m/z ratios output

Baseline & Peaks MS: Ratios Peaks: Threshold



1. This window scan through the thresholds from raw to 15 bits, and calculates the signal at m/z 32, 28, and 40.
2. The Ratio of m/z 40/28 is a key diagnostic for the instrument performance and quatitation.
3. For Ambient air this is the ratio to Argon to N2 and in this window the absolute value should be around 0.022 for the threshold used when measuring (when duty cycle corrected in the DAQ window and in Sqr1 the number is 0.018)

3.)



Start Stop Avg. Time 10 sec

IDLE

Baseline, SI, and Threshold

- Baseline set in instrument based on electronic noise with low V on MCP
- Saturation of m/z 28 checked to see if MCP too high (if too high reduce emission current or MCP)
- Single Ion value determined from m/z 40 or default ion list, and updated in text box
- Threshold parameters checked
- Check 40/28 ratio to make sure threshold value is appropriate for measurement