ASTON IMPACT
MASS AXIS CALIBRATION

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ATONARP ASTON IMPACT Mass Access Calibration Guide, USM-980-0006

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Revision History

Revision	ECO	Change Log	Author
A	ECO-001XX	No Content Changes	Vinay Kulkarni

About Mass Axis Calibration

As an analytical instrument producing signal abundance (Y-axis) as each atomic mass unit (X-axis), Aston requires calibration to obtain the most accurate quantitation (partial pressures) for specific compounds. The calibration is performed using a gas mixture. The selection of the appropriate mixture of components depends on the mass range over which the calibration is desired.

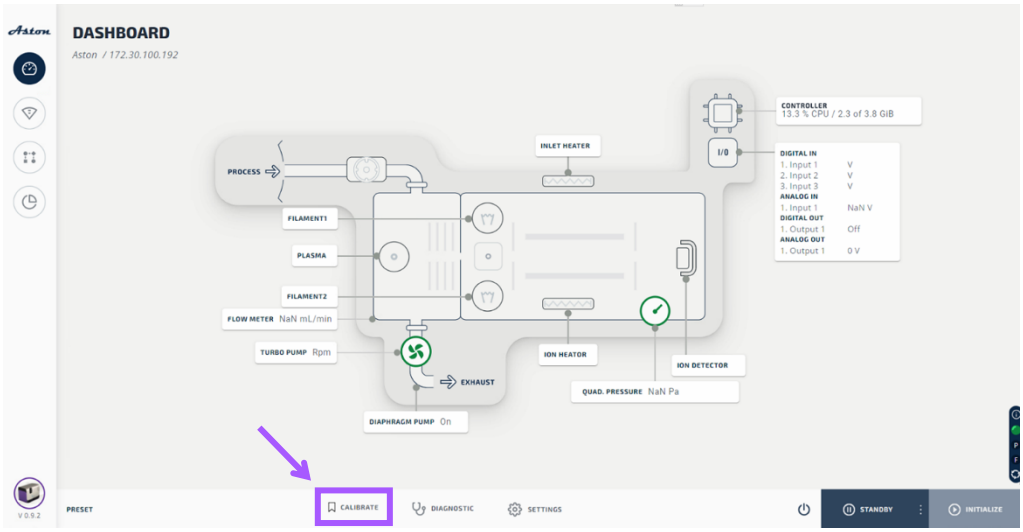
For a limited mass range, a gas cylinder of Argon with a list of known impurities or a mixture of noble gases and Nitrogen is adequate. An example of a mixture is shown in the table below. The gas can be introduced via a metering valve to establish the desired pressure in the mass spectrometer chamber.

FC43 (or PFTBA) is a high molecular weight liquid standard for higher masses with a vapor pressure of 146 Pa (1.1 Torr) at 25°C. The mass is given in the table below. The masses of abundant peaks can be used to perform the mass-axis calibration, particularly above 100 amu.

Component	Calibration Mass
H_2	2
He	4
N_2	28
Ar	36, 38, 40
Kr	78, 80, 82, 83, 84, 86

PFTBA	
Calibration Mass	Normalized Intensity
69	99
131	38
219	66
100	11
119	12
264	11
113	4

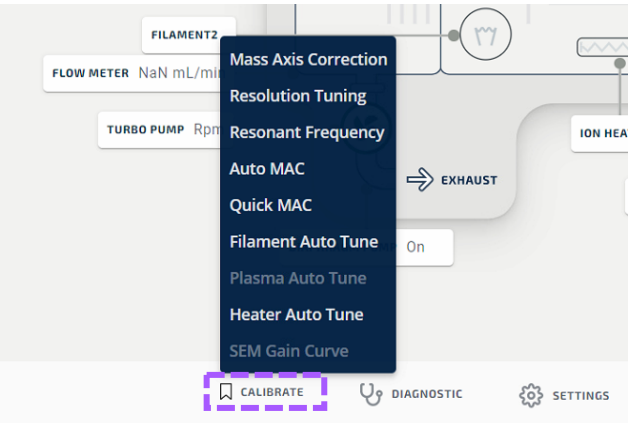
This chapter focuses on the top four (4) spectral calibration procedures under CALIBRATE, which is accessible at the bottom of the DASHBOARD. Aston units are factory-calibrated, and calibration (for Mass-Axis and Resolution Tuning) files (in .csv format) are locally stored and can be readily accessed. The spreadsheets can be modified under Excel and saved as XLSX files. The files can be uploaded and used as new calibration files.



Aston spectral calibration process involves the following three steps:

1. Resonant Frequency (RF) Search
2. Mass Axis Correction

3. Resolution and Sensitivity Tuning

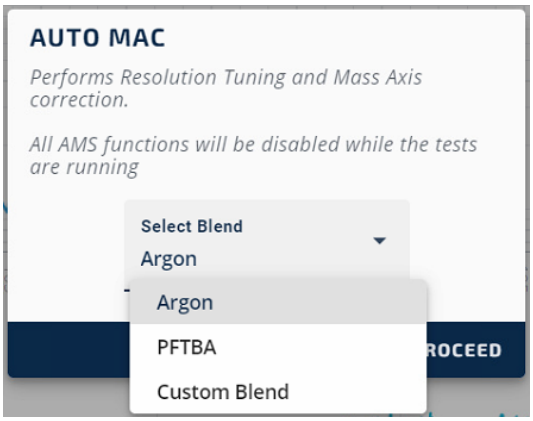


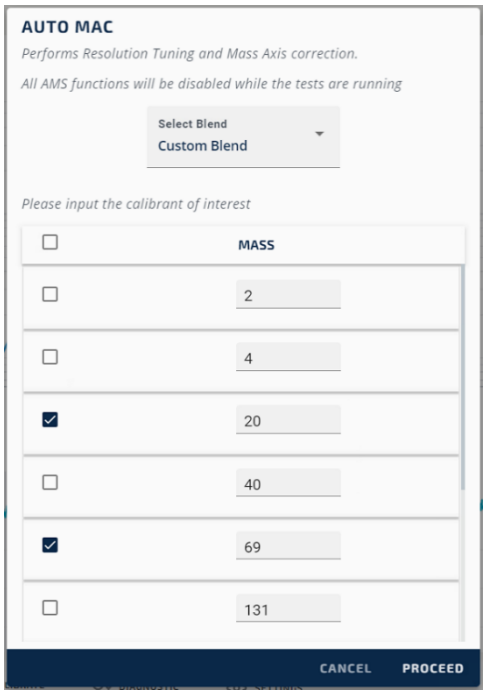
CALIBRATE menu items are accessible at the bottom of the SCAN screen (see the image below).

All three steps involve the x-axis calibration: the resonant frequency and mass axis correction ensure that the mass peaks are accurately located along the amu axis within less than +/- 0.1 amu. The resolution and sensitivity tuning determines the optimal AC-DC ratio schedule for constant peak width across the mass range. The calibration can be performed either manually by executing all three steps or automatically using the Auto Mac function. We will start with the Auto Mac function, which is the most straightforward. The manual calibration procedures, recommended for advanced users, will follow.

AUTO MAC

The Auto MAC function is a computer-automated method for calibrating an Aston system. Auto MAC steps through Resonant Frequency, Resolution Tuning, and Mass Axis Correction procedures internally via an advanced algorithm. Under Auto MAC, the system performs the three steps using a single source of a gas mixture. It allows the user to select from some preloaded gases, e.g., Argon and PFTBA, or a custom gas blend that can be defined. Once the calibration gas is selected, click on **PROCEED** button.



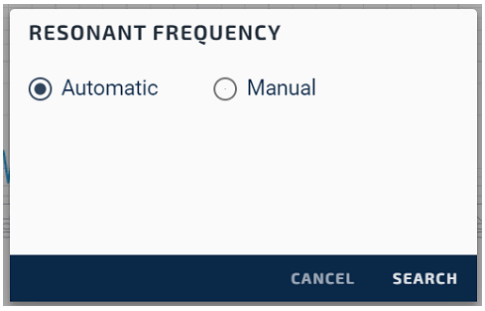


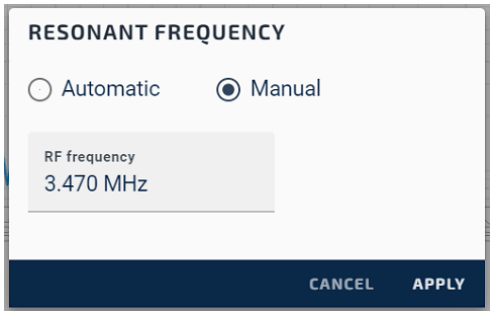
✳ NOTE

While Auto MAC usually calibrates the Aston without issue, always double-check the quality and location of the peaks after the Auto MAC calibration is done. Sometimes the user must run the Auto MAC function again or manually calibrate the system using the steps detailed in the following sections.

RESONANT FREQUENCY (RF)

As mentioned above, the resonant frequency calibration is performed at the factory, and this procedure should be considered a verification-only step. The Resonant Frequency can be set via Automatic or Manual options. Using the Automatic option, the system determines the resonant frequency through a few seconds-long computer controller tuning procedure. The resonant frequency is then displayed and is usually in the 3.4-3.6 MHz range. The user can then apply or discard the value.





Under Manual, the user can input a custom RF frequency value. Advanced users should only use the Manual option of setting the frequency as far-off values can cause overheating of the RF circuitry.

NOTE

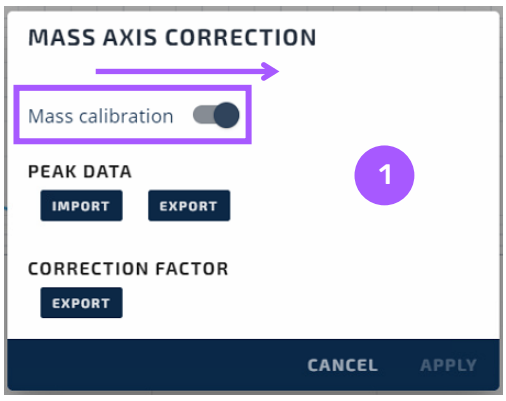
Set the RF frequency before attempting Mass Axis Correction or Resolution Tuning. As Aston quadrupole sensors are identical within a narrow range of capacitance, there will be slight differences in resonant frequencies to ensure a perfectly tuned RF circuit.

MASS AXIS CORRECTION

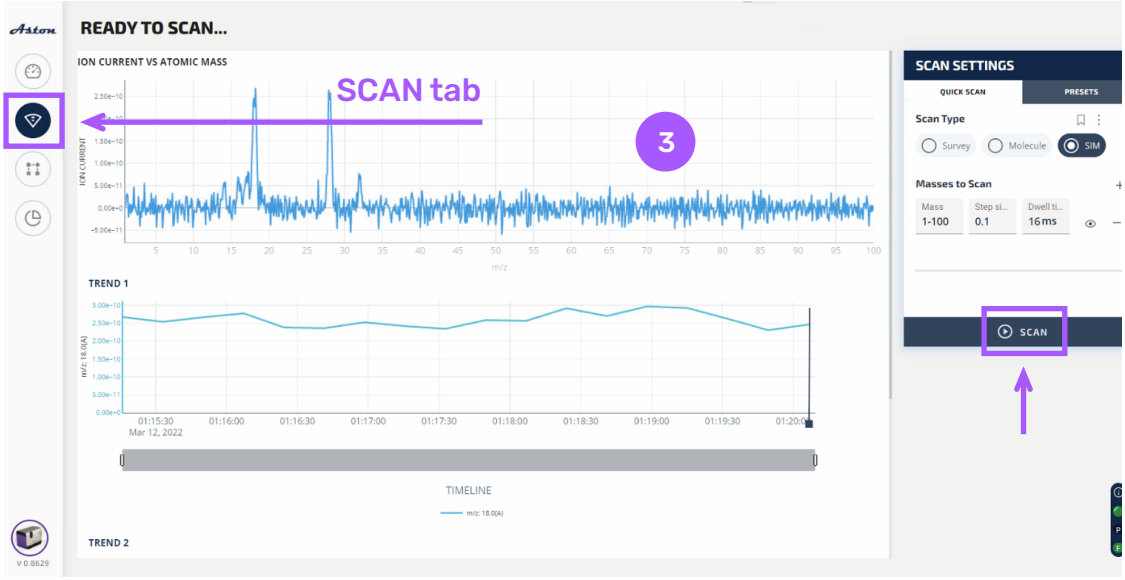
The MASS AXIS CORRECTION function allows the user to correct peak location errors across the mass spectrum. Enabling Mass calibration lets the user import and export calibration files. The user can change the spreadsheet under Excel in the exported .csv file and import it to apply new calibration. The correction is accomplished using the following steps:

1. Ensure Mass calibration is enabled. Enable/disable Mass calibration by toggling this field. The settings from the imported file are applied when enabled.

2. Click on EXPORT and make a note of the path that appears on the bottom left of the screen. The peak_data.xlsx file will be downloaded in this location.



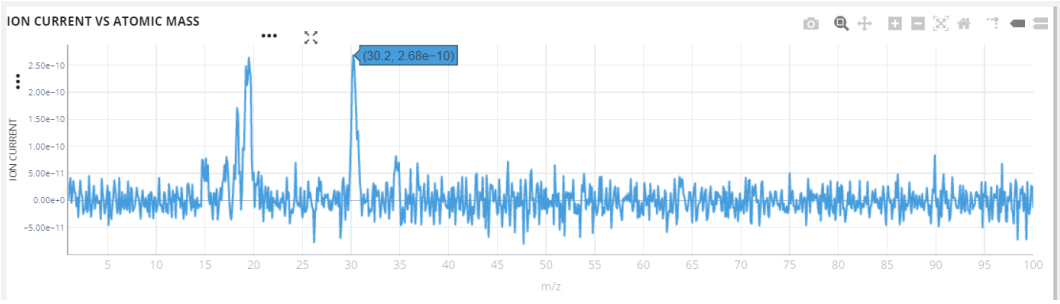
3. Navigate to the **SCAN** tab and click on SCAN.



NOTE

Atonarp suggests a dwell time of at least 16 ms for calibration purposes.

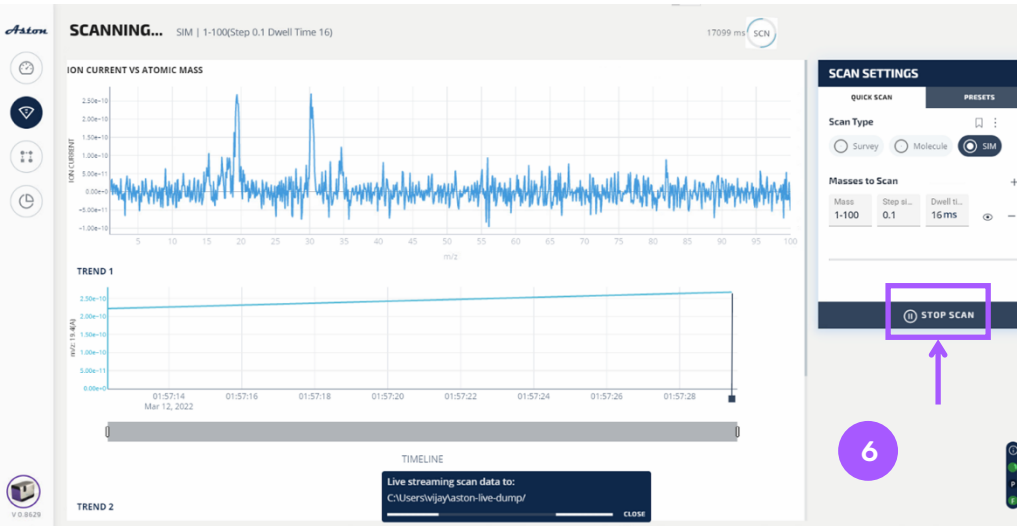
4. Look at scan data and identify a peak of interest. As an example, the figure below shows a peak at 30.2 m/z. This peak corresponds to Nitrogen and thus should be at 28 m/z instead.



5. The spreadsheet in the peak_data.xlsx file shows two columns: **Reference mass** and **User-observed mass**. Continuing with the previous example, the user will input the observed mass (30.2) next to the reference mass (28), as shown below. Save the file. Repeat steps 5 and 6 for all reference masses/peaks being calibrated.

	A	B
1	**Reference mass**	**User-observed mass**
2	4	
3	14	
4	20	
5	28	30.2
6	40	
7	84	
8		

6. Once all the masses are appropriately located and corrected, stop the current scan and open the Mass Axis Correction function again.



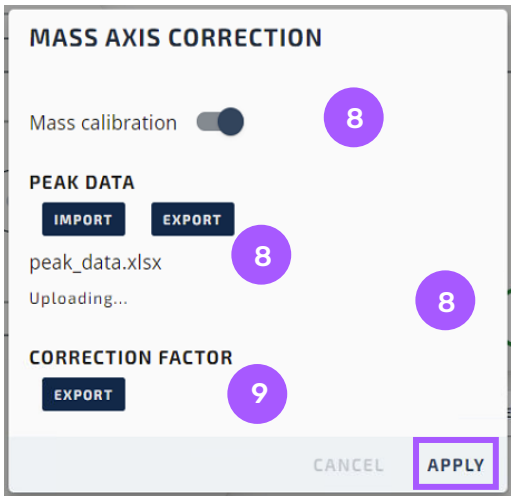
7. Enable **Mass calibration,** click on **IMPORT** to upload the newly saved *peak_data.xlsx*, and then click **APPLY.** The embedded algorithm in Atonlab software will now perform the necessary corrections.

✳ NOTE

Following Resolution and Sensitivity Tuning (next section), verify the location of the spectral peaks on the mass axis is still correct. Resolution and Sensitivity Tuning could alter the locations slightly.

8. Click on the EXPORT button to export the CORRECTION FACTOR. Exported XLSX file will be saved in the local folder.

Exported correction factor

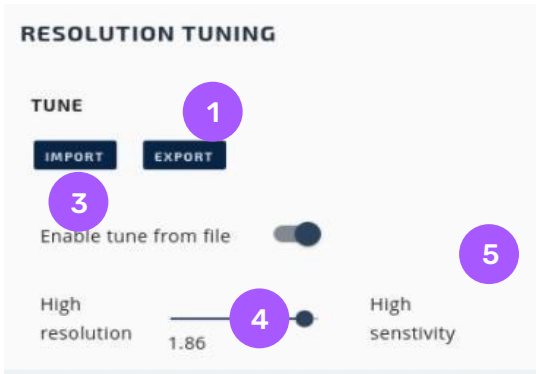


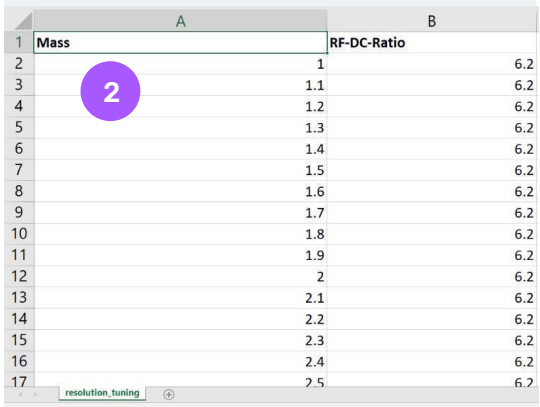
	A	B
	Mass	**RF correction**
	1	1
	1.1	1
	1.2	1
	1.3	1
	1.4	1
	1.5	1
	1.6	1
	1.7	1
	1.8	1
	1.9	1
	2	1
	2.1	1
	2.2	1
	2.3	1
	2.4	1
	2.5	1

RESOLUTION TUNING

The RESOLUTION TUNING function enables the user to set an RF-DC ratio schedule to achieve a target peak width for the entire mass range. Set the RF/DC values using Enable tune from the file. The tuning is accomplished using the following steps:

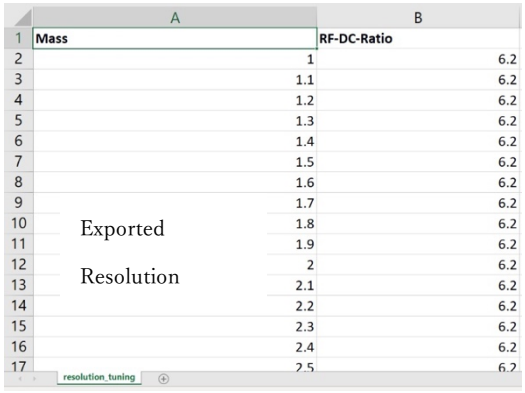
1. Click on **EXPORT** and note the path on the bottom left of the screen in the below TUNE. The exported template will be saved in the local Aston folder.
2. The exported resolution tuning template (shown below) has Mass and RF-DC ratio fields wherein the user can modify the RF-DC ratio values
3. After setting RF-DC ratio values for the entire mass range, click on **IMPORT** to upload the XLSX file
4. Use the High Resolution/High Sensitivity slider to set the target peak width
5. Click on **PROCEED**



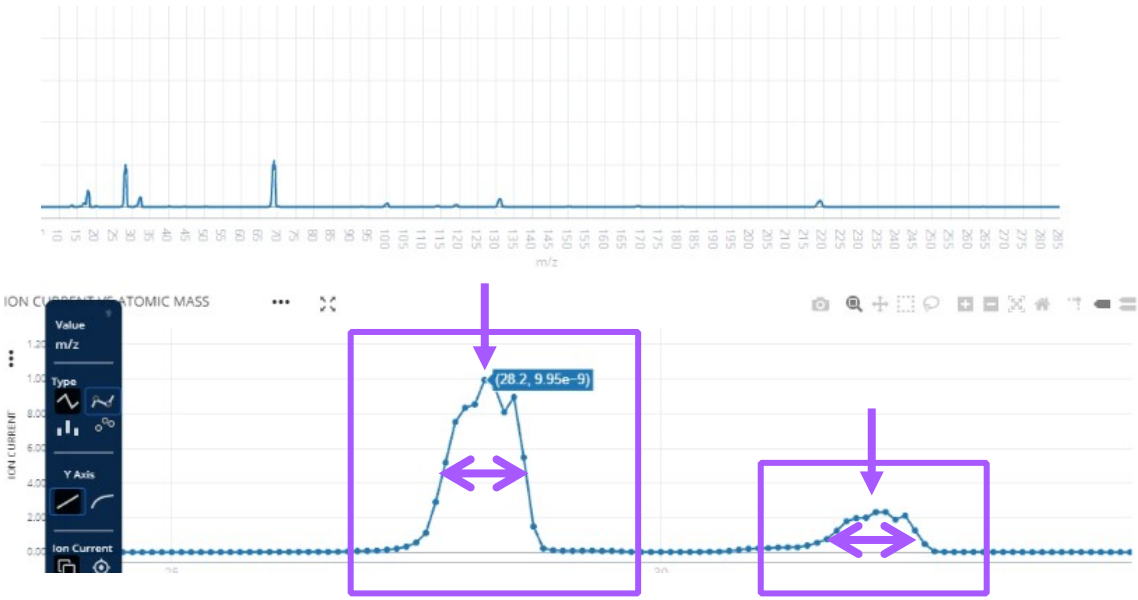


	A	B
1	Mass	RF-DC-Ratio
2	1	6.2
3	1.1	6.2
4	1.2	6.2
5	1.3	6.2
6	1.4	6.2
7	1.5	6.2
8	1.6	6.2
9	1.7	6.2
10	1.8	6.2
11	1.9	6.2
12	2	6.2
13	2.1	6.2
14	2.2	6.2
15	2.3	6.2
16	2.4	6.2
17	2.5	6.2

1. Examine the spectrum by confirming each target compound's mass axis location and resolution (FWHM).



	A	B
1	Mass	RF-DC-Ratio
2	1	6.2
3	1.1	6.2
4	1.2	6.2
5	1.3	6.2
6	1.4	6.2
7	1.5	6.2
8	1.6	6.2
9	1.7	6.2
10	1.8	6.2
11	1.9	6.2
12	2	6.2
13	2.1	6.2
14	2.2	6.2
15	2.3	6.2
16	2.4	6.2
17	2.5	6.2



NOTE

Change RF-DC ratio values and acquire spectrum again if the target peak width is not achieved.

2. Modify the exported file accordingly by setting the AC-DC ratio values for each target compound. The end result is a file with an AC-DC ratio schedule for individual segments of the mass spectrum.
3. After setting RF-DC ratio values for the entire mass range, click on IMPORT to upload the XLSX file and Enable tune from the file
4. Click on PROCEED

CALIBRATION VERIFICATION

Aston_Impact is factory-calibrated using standard gas mixtures and PFTBA to cover the complete mass range using the same procedures detailed in Chapter 8. As the mass spectrometer sensor and vacuum chamber get exposed to various gases, some more reactive than others with surfaces, slight changes in electrode voltages may lead to shifts in peak locations. This effect is significantly mitigated by keeping the vacuum chamber and the sensor heated up to 350 °C to minimize surface adsorption. It is recommended to verify the calibration according to a user-settable preventive maintenance schedule, which depends on the usage of the instrument. This verification is crucial ahead of long process runs to ensure that data is collected using optimum instrument performance.

The calibration verification can be achieved using any available gas, e.g., Argon or gas mixture, by performing a survey scan and verifying the location of the relevant peaks to be within +/- 0.1 amu. If the peaks are shifted slightly to the left or right, running the Auto MAC (according to the procedure in Chapter 8) function may be the fastest method to re-align the peaks. Manual calibration, stepping through RESONANT FREQUENCY, MASS-AXIS CAL, and RESONANT TUNING may also be considered.

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