

Maintaining lab operations in extraordinary times

Dealing with operating impacts from the COVID-19 Coronavirus

GC/MS	Thursday, April 16 th
HPLC and LC/MSD	Friday, April 17 th
ICP-OES & ICP-MS	Wednesday, April 22 nd
LC/QQQ & LC/QToF	Thursday, April 23 rd
GC	Thursday, April 30 th



Important general information for managing lab operations

Follow your SOP's – but here are some additional things to consider

- How has operational changes affected your SOP's? Are there impacts to instrument maintenance, qualifications, etc? Has instrument usage changed as this may affect service/consumable replacement intervals? Document these and prepare a plan to bring them online once you resume normal operations.
- Develop a new schedule/routine for working in the lab. This is a challenging time for everyone and routine helps everyone acclimate to these impacts.
- Prioritize time in the lab - Can any tasks be shifted remote or online (training, remote monitoring, data analysis)? Is your IT department aware of these and is bandwidth/VPN/remote access capable of handling this.
- Are service providers allowed on-site? Or is there remote work they can do? Discuss this before any scheduled visit. Our service teams are currently offering free live video conferences to support labs around the world – more info at the end of the presentation.
- Proactively replace lab consumables before you see performance issues. If ordering supplies, check if shipping/receiving/logistics for your company has been affected.
- When you return to normal operations, have a detailed restart plan that outlines priorities and timelines. Agilent will provide additional information on returning/restarting your lab in a few weeks.

Simple Strategies for GC/MS Success

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Time robbers in the GC/MS lab

- **Looking for leaks**
 - Leaks affect sensitivity, damage column, shift peak retention times
 - Locating leaks can be tedious
- **Unplanned maintenance**
 - Over time, deposits on the MS source impair performance
 - Closer inspection of routine tune reports can proactively identify need for source maintenance
- **Planned maintenance**
 - Performing source maintenance properly



GC/MS Time Robber #1: Leaks

Hunting for leaks: weapon of choice

Tip: Arm yourself with the best...or in a pinch, what's nearby

- A good leak detector is an MS chemist's best friend
 - High sensitivity
 - Recommended for leak detection in gas plumbing and fittings

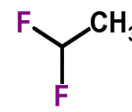
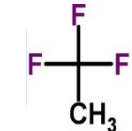
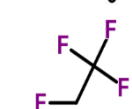


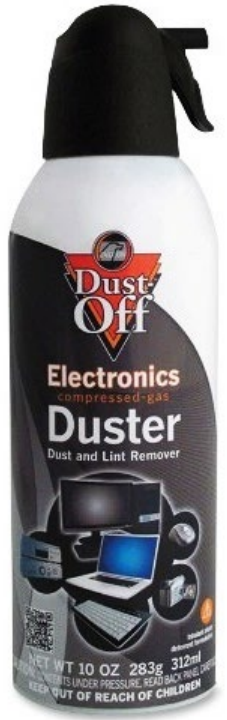
Agilent G338B Leak Detector

[link](#)

- If you don't have a leak detector, use electronics duster instead
 - Hold can upright (don't spray liquid!)
 - Spray short bursts around likely leak suspects
 - Spray just enough
 - "Live" tune scanning for ions to pinpoint leak

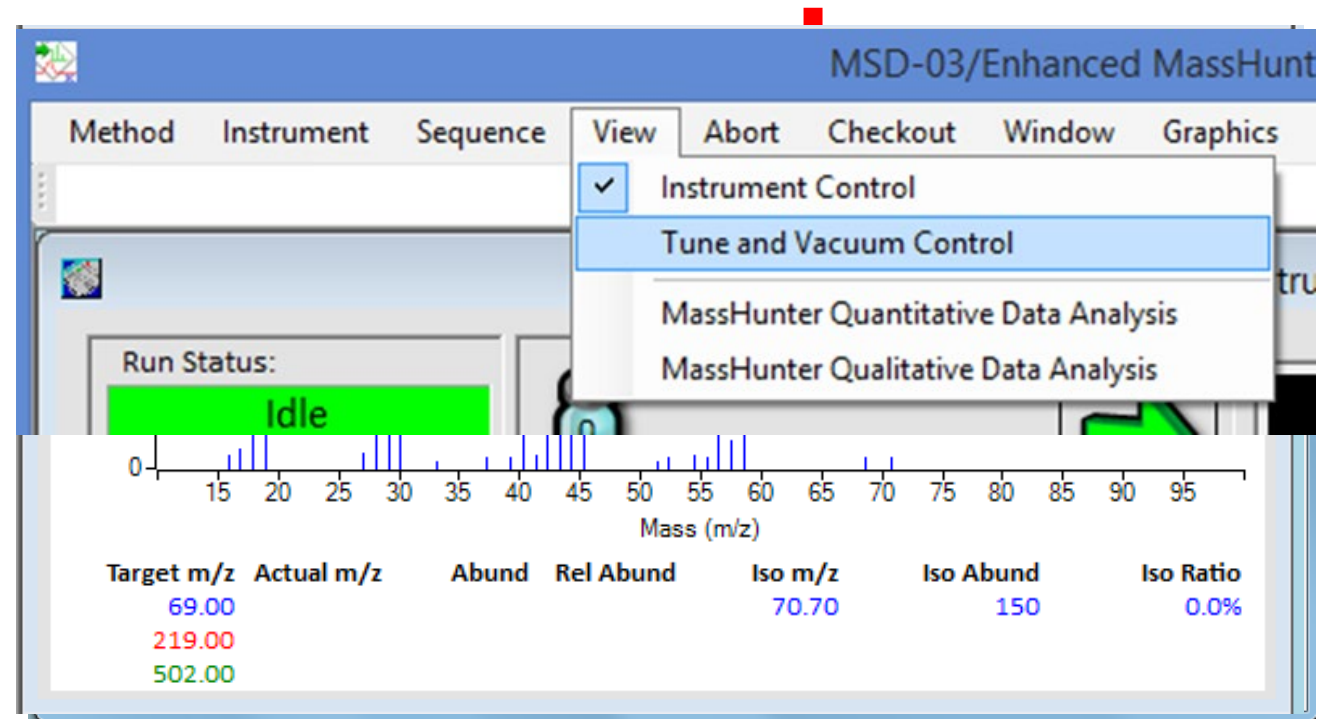
Typical Electronic Duster Components and Ions

	1,1-difluoroethane	m/z 51,65
	1,1,1-trifluoroethane	m/z 83
	1,1,1,2-tetrafluoroethane	m/z 101

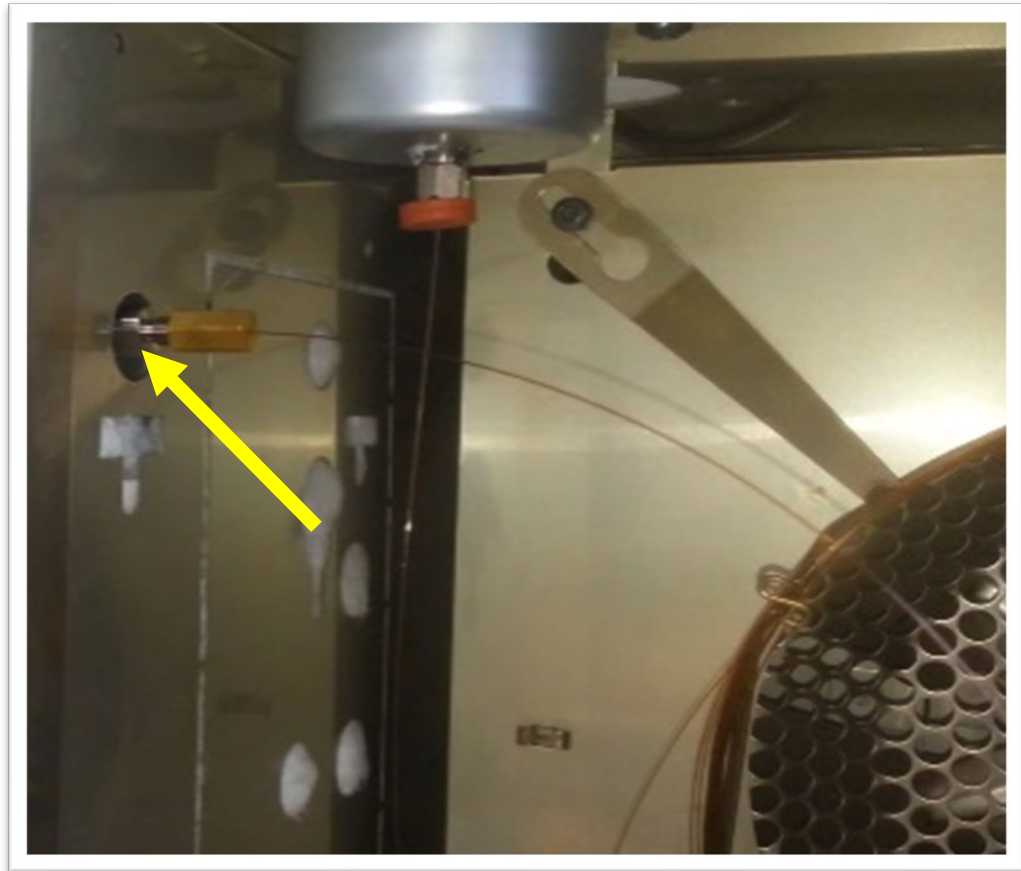


Hunt for leaks in manual tune mode

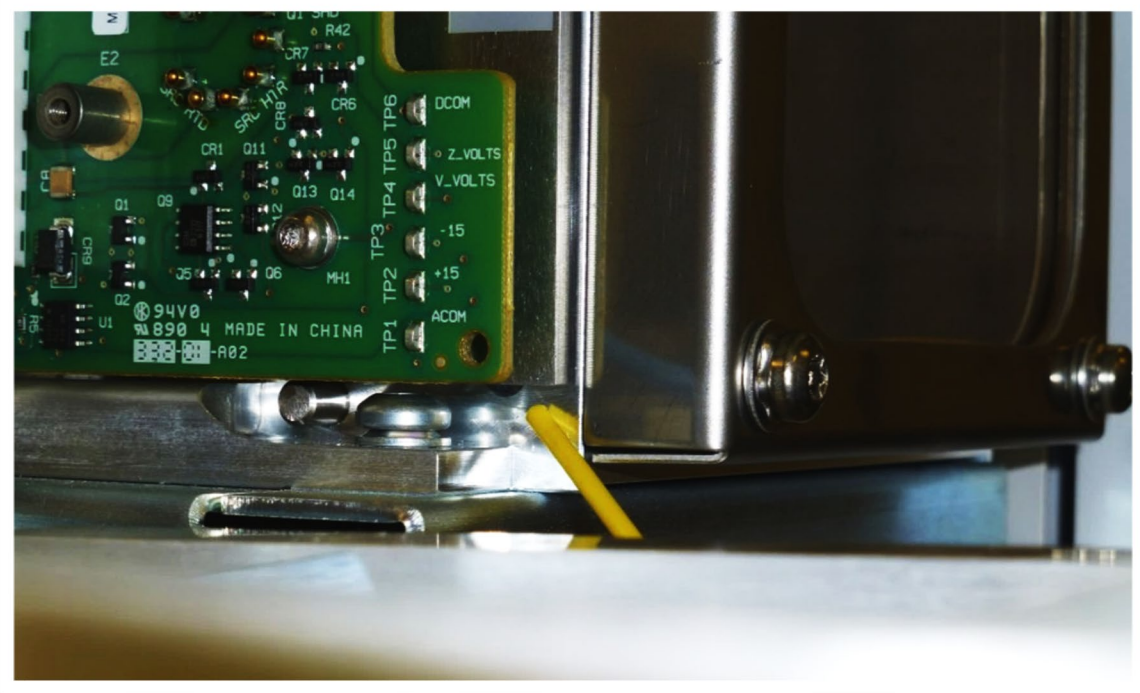
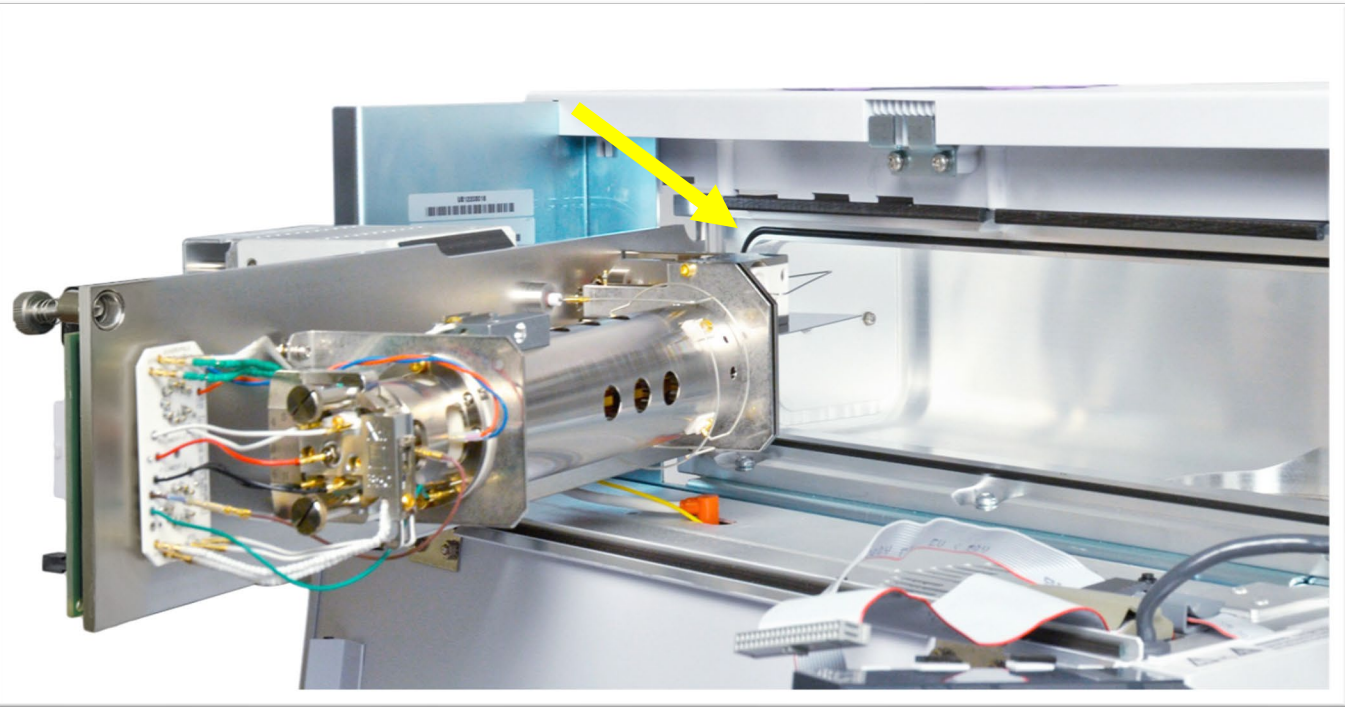
- From MS tuning interface
- In Agilent's MassHunter Acquisition, Vacuum/Tune Control window
 1. Select Parameters > Manual Tune
 2. Click Scan tab on right-hand side
 3. Set low m/z = 10, high m/z = 100 amu
 4. Ensure PFTBA valve closed
 5. Click Scan
 6. Spray common leak sites, watch scan window for large duster ion spikes
 7. Exit manual tune screen, do not save parameters
 8. Return to acquisition



🔍 Where to look: common leak sites



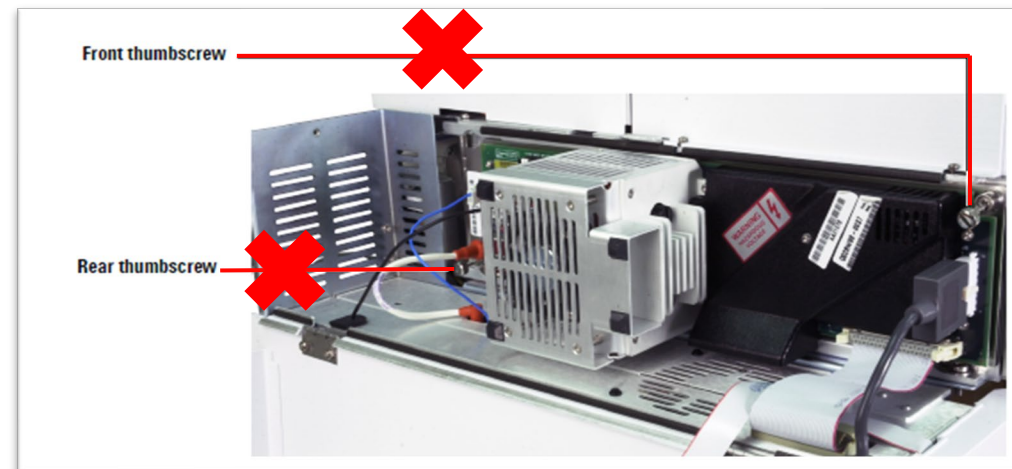
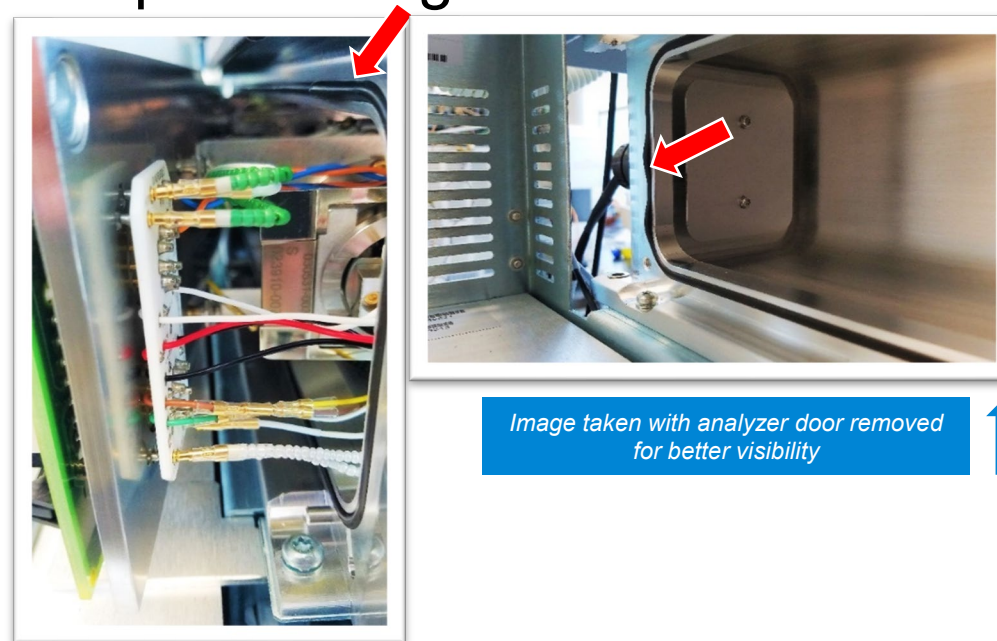
Where to look: common leak sites



Hunting for leaks: the analyzer side plate

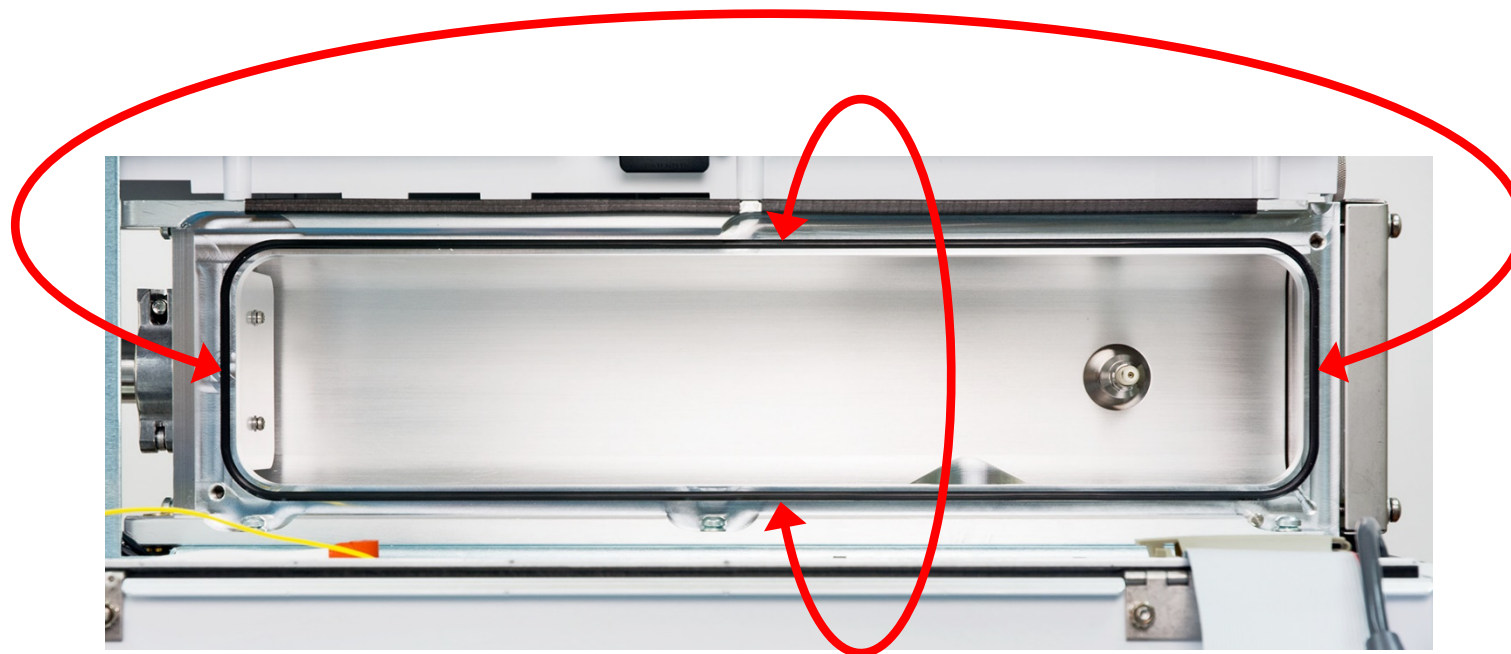
Tip 1: Ensure seal integrity at the side plate o-ring

- Factors that disturb seal integrity
 - Sticking/dislodging upon opening
 - Aging and wear
 - Physical obstructions: dust, lint, metal fines
- How is it fixed and prevented?
 - Using gloves, gently manipulate o-ring within groove to ensure uniform surface
 - Visually inspect seal integrity prior to pump down
 - Replace worn or damaged O-rings
 - Wipe O-ring perimeter with lint-free tissue
 - Stop using thumbscrews
 - Can create metal fines with excessive use
 - Only use during shipment/relocation
 - Ensure MS is well-ventilated
 - Periodically vacuum beneath outer covers



Hunting for leaks: installing the analyzer side plate

Here and here....



Then here and here.

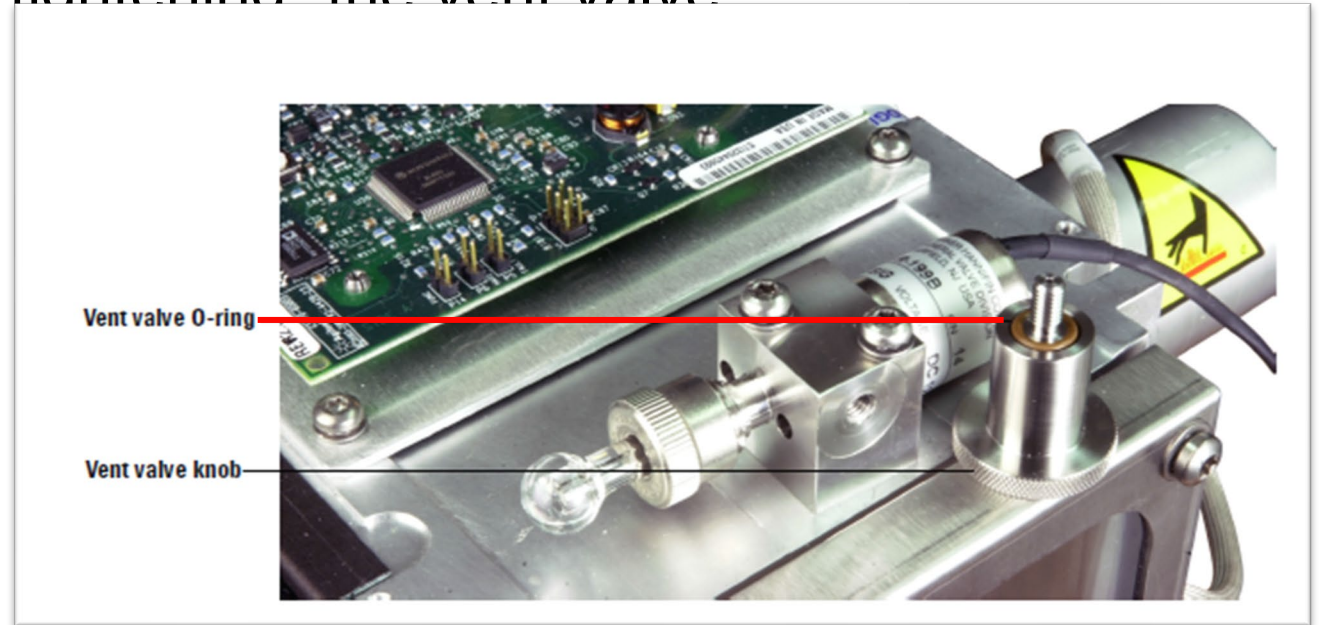


Agilent GCMS Side plate O-ring P/N 0905-1442

Hunting for leaks: the vent valve

Tip 2: Forgetting to retighten- or overtightening- the vent valve

- How does this happen?
 - Easily overlooked
 - Urge to overtighten fittings
- Why is it bad?
 - Overtightening can create metal shavings
 - Prevents leak-free seal formation
- How is it fixed?
 - Tighten gently to avoid cross-threading
 - If leaks are persistent, replace vent valve o-ring

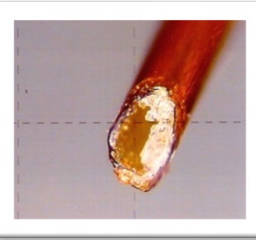


Agilent MSD Vent
Valve o-ring
P/N 0905-1014
[link](#)

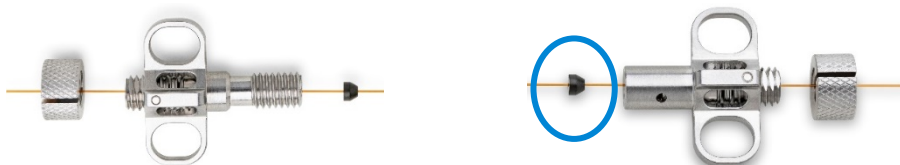
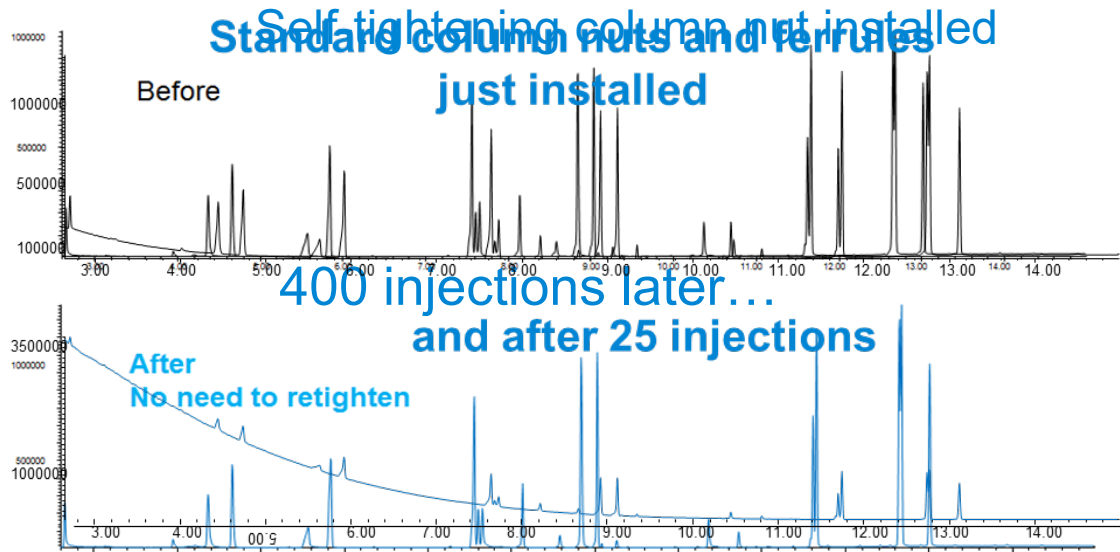


Hunting for leaks: transfer line interface

Tip 3: Tight, but not too tight...Better yet, self-tightening



- Why is it bad?
 - Over tightening damages transfer line threads, column
 - Audible squealing → overtightening
- How is it fixed?
 - Stop overtightening
 - Stripped transfer line threads require replacement by service engineer
 - Just tight enough- use MS in manual tune mode to check
 - Switch to self-tightening MS column nuts
 - Spring-driven piston **continuously** presses against ferrule
 - Easily installed, no tools
 - Collar helps with column installation depth
 - Benefits- **longer column lifetime, lower column bleed**



For Agilent GC Inlet/Detector P/N G3440-81011, for MS transfer line P/N G3440-81011

A critical connection

    A proper connection between the column and MS is critical to achieving a leak free seal and getting good chromatography.



Focus on your ferrules



Even experts get **distracted**. Try to take an extra moment to ensure you grab the right ferrule from others you might have on hand.

Double check the diameter



Make sure the ferrule diameter accommodates your column's ID. Using a 320um ferrule on a 250um column will result in a leak.

HP - 5MS UI		
Length (m)	Diam. (mm)	(µm)
30	0.250	0.25
	Narrowbore	





Just tight enough! Overtightening damages fittings and your column. Periodically retighten brass column nuts, or switch to self-tightening.

The Facts on Ferrules



Recycling is great for the environment, but not so good for ferrules- their ability to make a leak free seal declines with age and wear. **Never reuse ferrules.**

Right side up

To MS


Installing the ferrule the wrong way is a common cause of leaks. The widest part of the ferrule should be oriented toward the MS.



Short selfies and brass barrels



Self-tightening column nuts need short ferrules. Brass column nuts require barrel-shaped ferrules. Use this mnemonic device to help you remember.

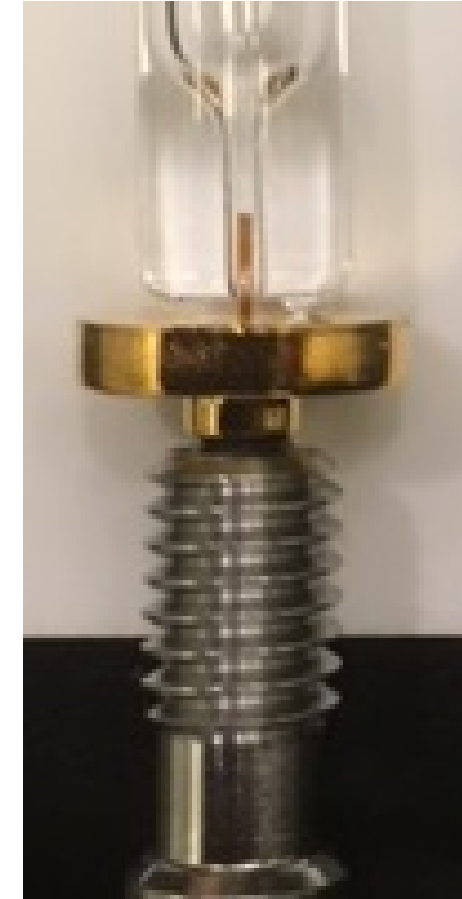
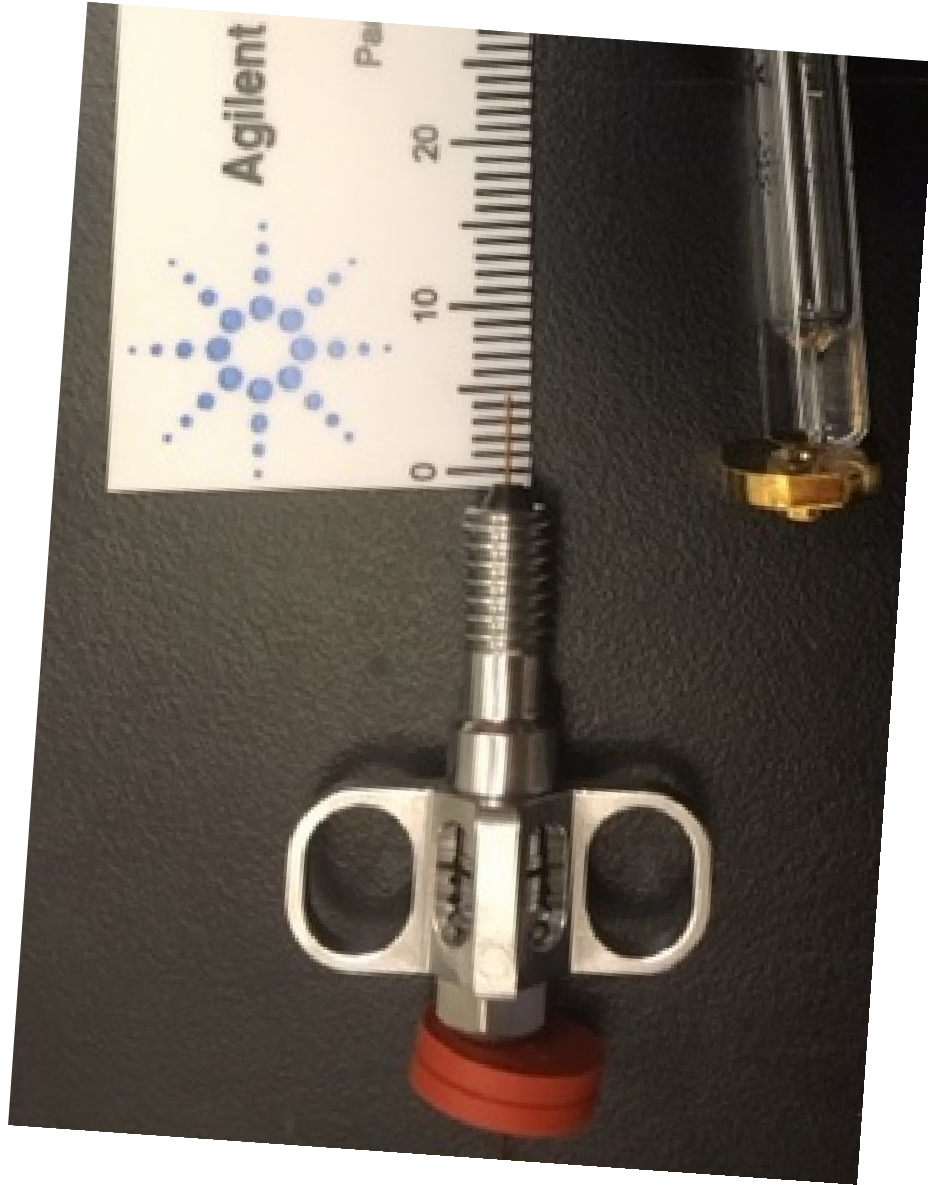


Graphite doesn't go



Pure graphite ferrules are a no-go with GC/MS- it's too soft, permeable, and has a tendency to flake or crumble. **Always use Vespel®/graphite ferrules for GC/MS applications.**

Why Does Distance Matter?



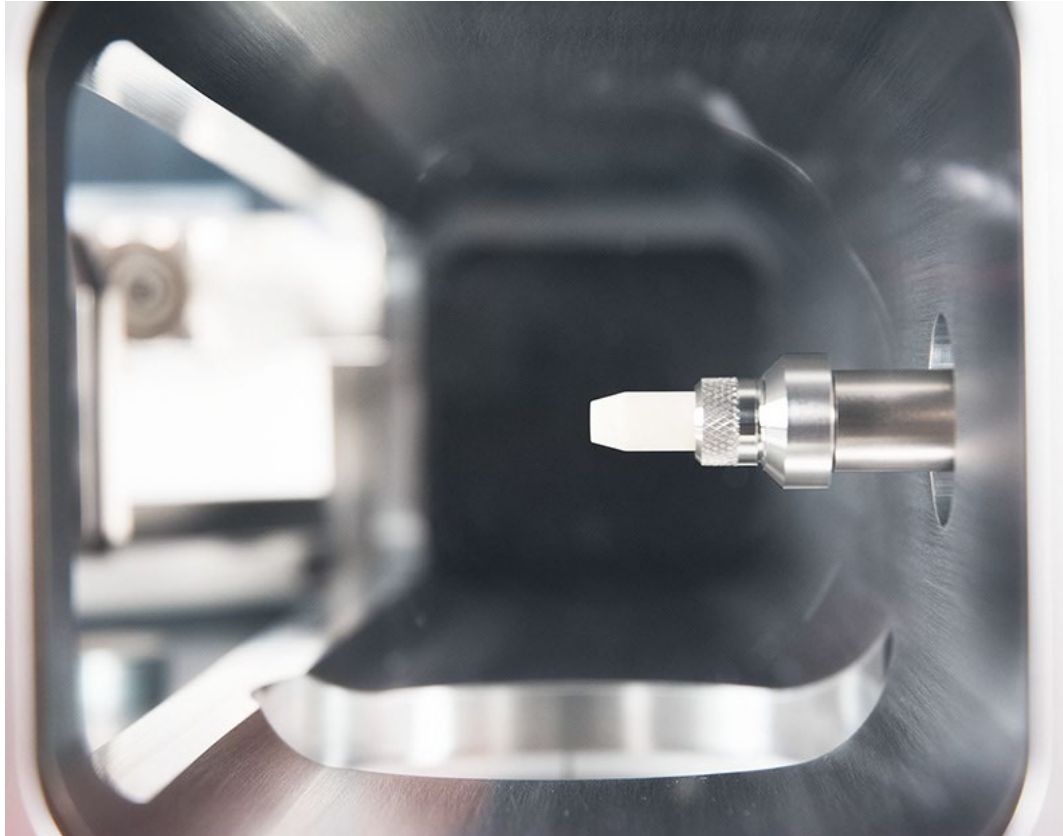
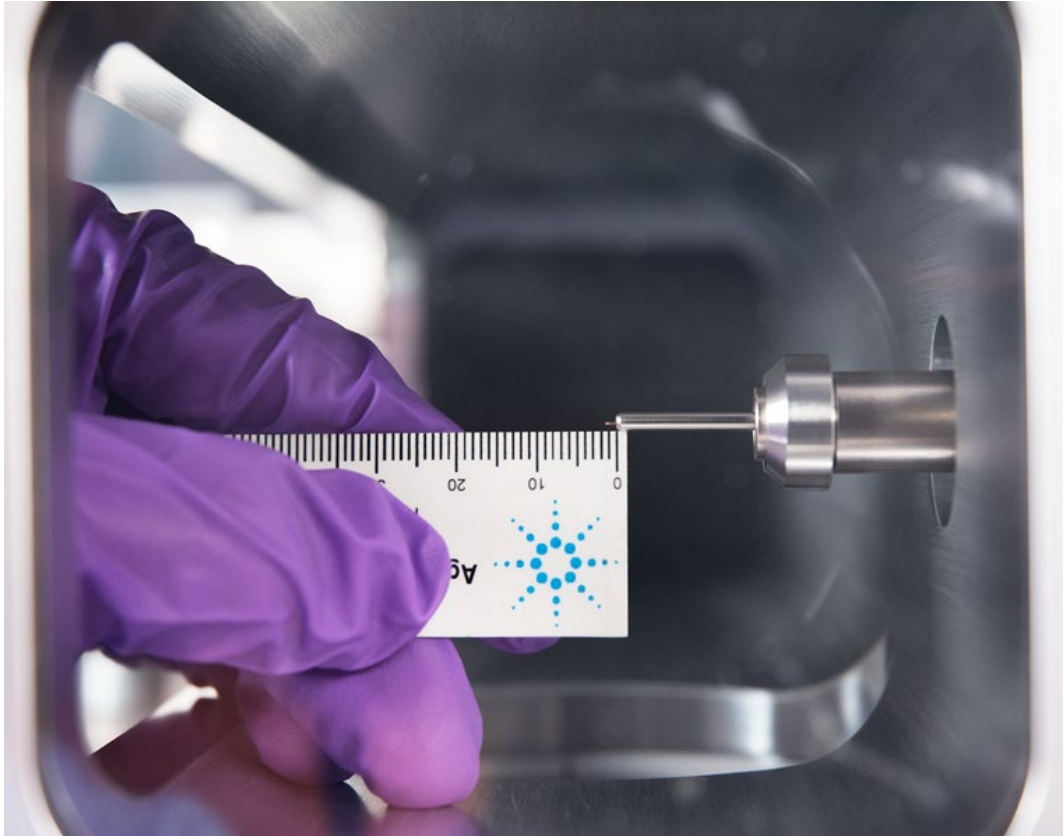
MS Column installation

Inert/Stainless/Extractor/CI

- Tip seal removed 1-2 mm exposed
- w/tip seal installed 0 mm exposed

HES

- Tip seal removed 4-5 mm exposed
- w/tip seal installed 2.5 mm exposed - maximum



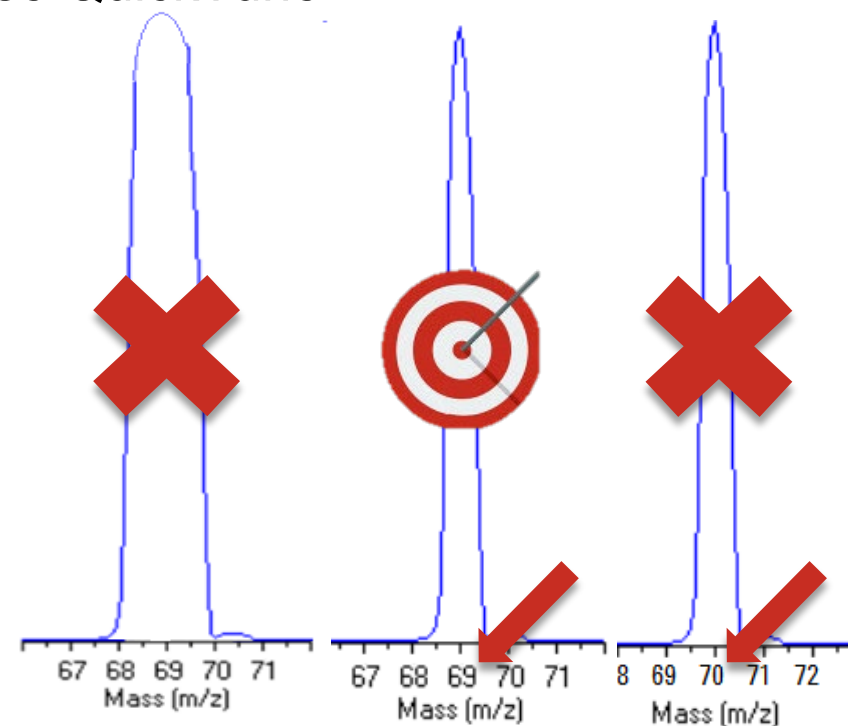
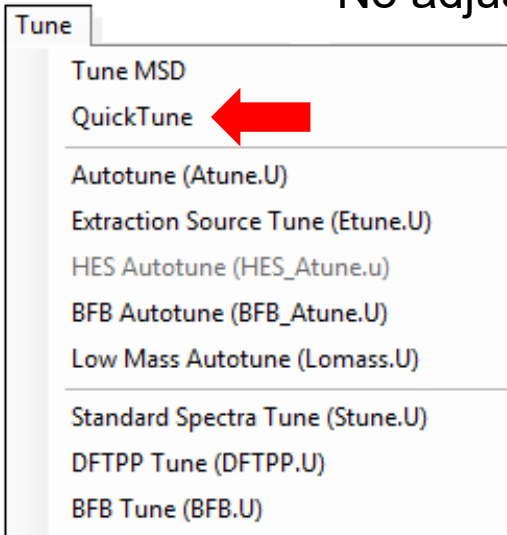
GC/MS Time Robber #2:

Unplanned maintenance- taking tune reports for granted

«U» Tuning tip #1: Tune routinely to monitor MS performance



- Frequency depends on how often MS used
- Recommend at least weekly
- Provides comparison points over time
- If method calibration/response factors are a concern, use QuickTune
 - Adjusts mass axis, peak width, and EM voltage only
 - No adjustment of mass abundances





Tuning tip #2:

Tune before AND after changes to GCMS

- Provides reference point in case something goes wrong
- Compare tune pdf files over time and look for differences
- Use these to troubleshoot, **what changed** from one tune to the next?
- Examples:



- Repair or preventative maintenance
 - After cleaning MS source (changed abundance)
 - Inlet maintenance (ensure no leaks)
 - New column installation/column trim
- Change gas filter or cylinders
 - Quickly catch contaminated gas tank
- Filament switch
- Vacation or prolonged period of downtime
- On Agilent 5977 or instruments after **MassHunter Acquisition vB.07.00**, pdf copies are automatically created
 - Default pathway: D:\MassHunter\GCMS\1\5977\PDF

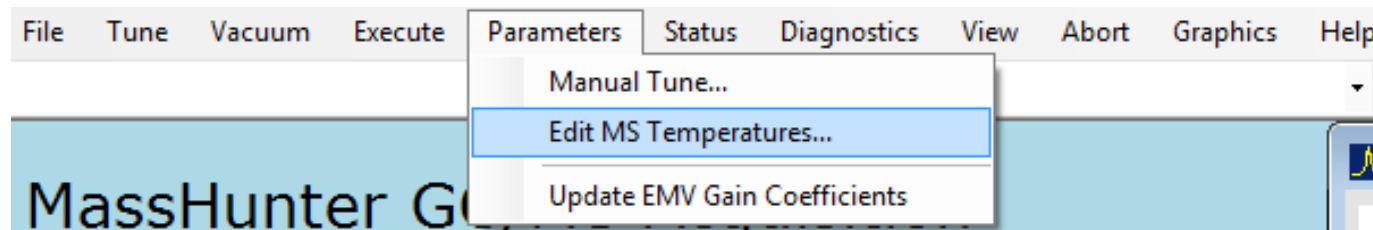


Tuning tip #3:

Create a maintenance GC method and cool MS tune file

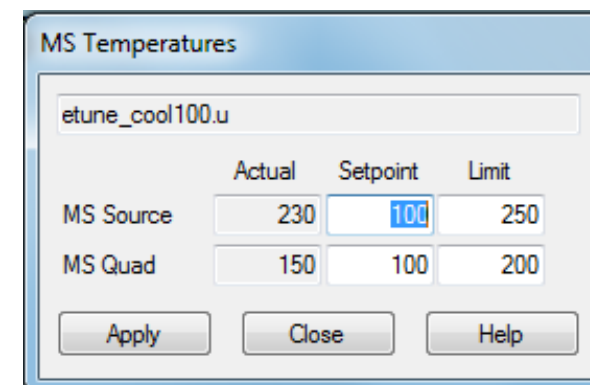
- Create cool.u or Prepare to Vent.U tune file

- Load your normal tune file
- Adjust quad and source temps $\leq 100^{\circ}\text{C}$
- Save as cool.u



- Create a Vent.m or Maintenance.m GC method

- Lower heated zones (set points $\sim 50^{\circ}\text{C}$, ideally cool enough to enable handling)
 - Oven, inlets, mass spec transfer line, etc.
- **Do NOT turn off carrier gas flows**
- Specify cool.u tune file in MS section of method



- Load Maintenance.m GC method before venting MS, or even program into end of sequence for convenient cool down

Tuning tip #4: Monitor tunes for signs of a dirty source

Tune ion peaks sharp and symmetric?

- 502 ion mass usually declines first
- Peak widths (Pw50) are usually ~ 0.6 amu

Consistent primary tune ion abundances?

- Atune, HES Atune: m/z 69 ~450,000 – 600,000
- Etune: m/z 219 ~450,000 – 600,000
- **Consistency** is key

Relative abundances within accepted values?

- 502 ion relative to m/z 69 > 3%
- **Consistency** is key

EM voltage consistent from last tune?

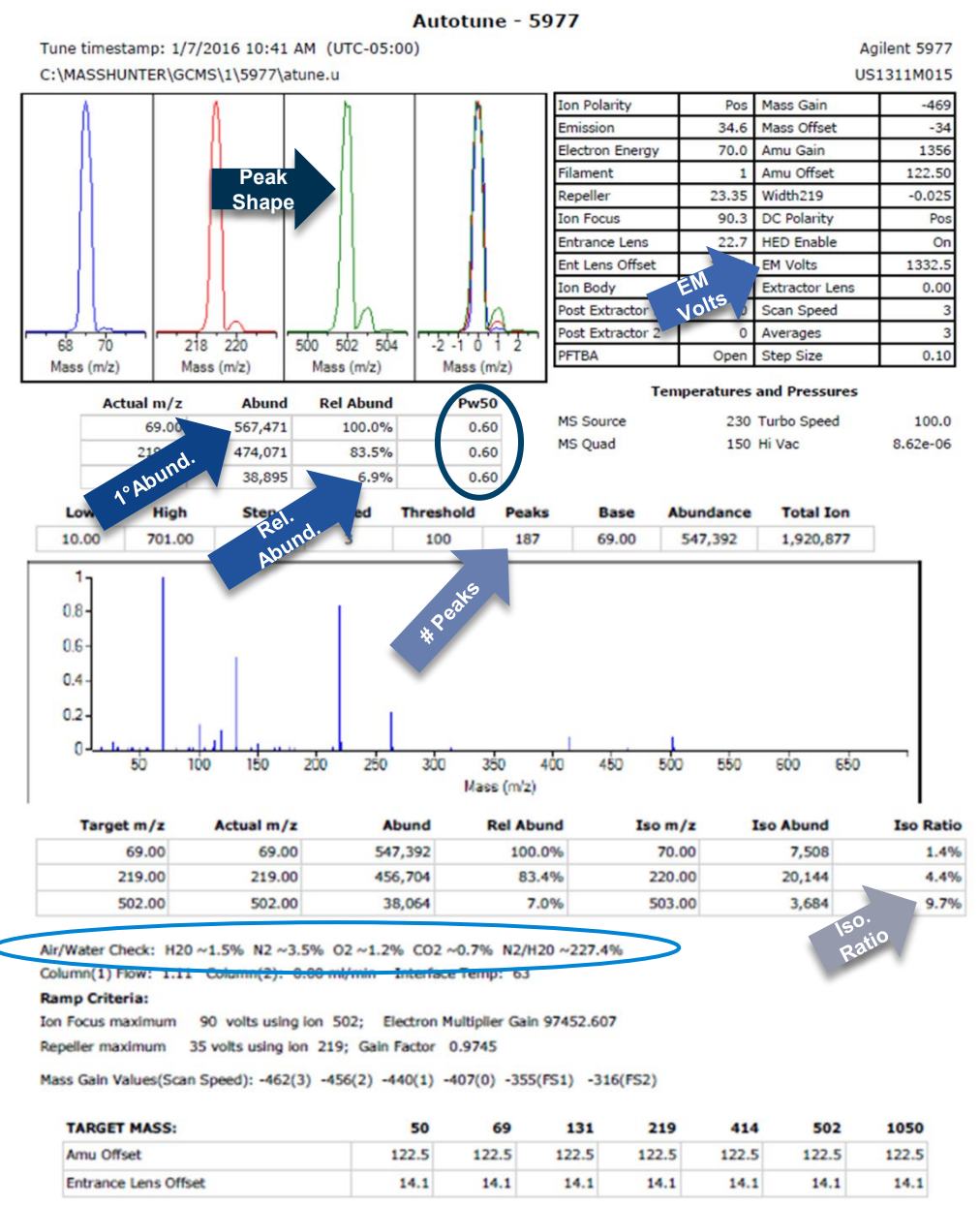
- As source grows dirtier, EM voltage increases
- Max voltage is 3000V
- **Consistency** is key

Is background free from excessive peaks?

- Excessive peaks can indicate source contamination

Isotope ratios within expected values?

- 502:503 isotope ratio roughly 10.1% (±20%)



Planned Maintenance

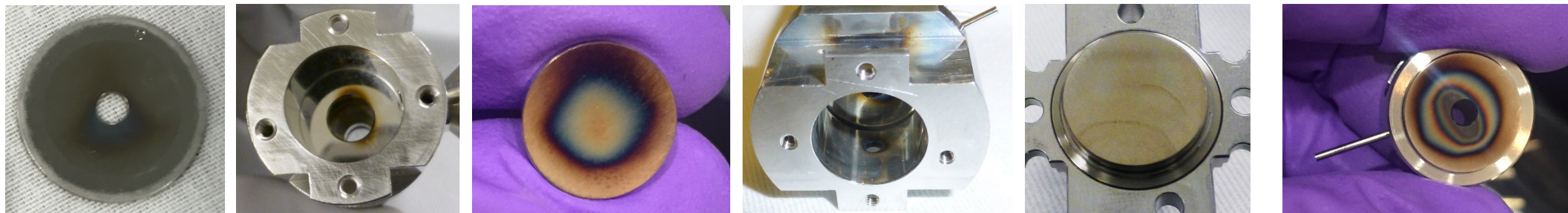
How to clean the source

How to clean an Agilent GCMS Ion Source

During the normal course of operation, GCMS sources require cleaning due to sample throughput. This can be indicated by:

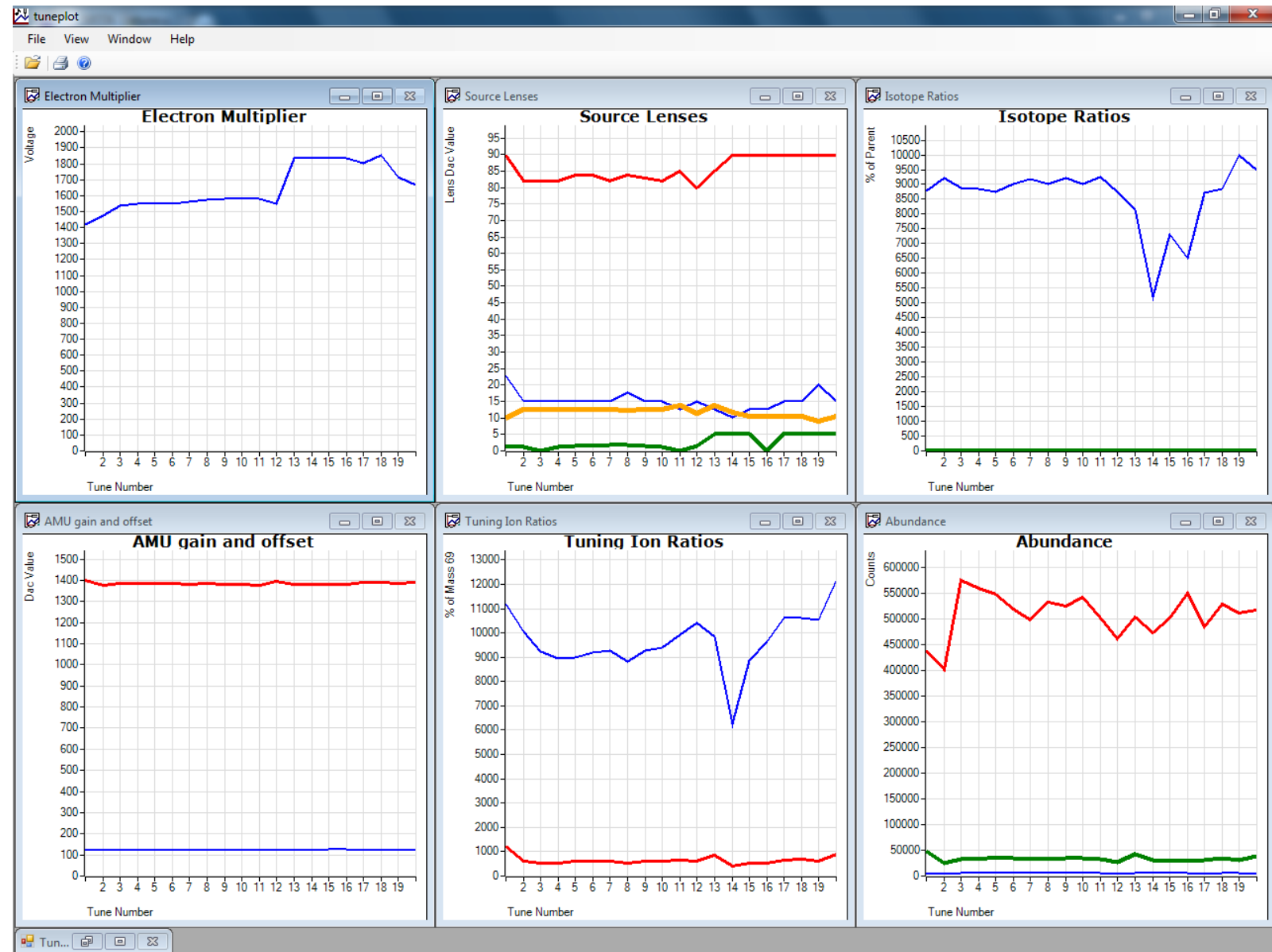
- Loss in analyte response not improved by normal inlet and column maintenance.
- Poor calibrant ion peak shapes during tuning, especially for the 502 ion.
- Escalating tune repeller voltage.
- Escalating tune Electron Multiplier voltage.

Proper cleaning, assembly, and installation are essential to robust and reliable operation. Source cleaning is part of normal user maintenance. These tasks are part of owning an instrument and are the operator's responsibility.



Sample residue, gas contaminants, and pump oil all form an adsorbed organic film that must be removed. This film is often semi-insulating and causes a loss of the required electric fields necessary for optimum performance. Thoroughly cleaning all metal surfaces of the source removes this coating.

How to clean an Agilent GCMS Ion Source



Tune and Vacuum Control -
File -
View Tunes

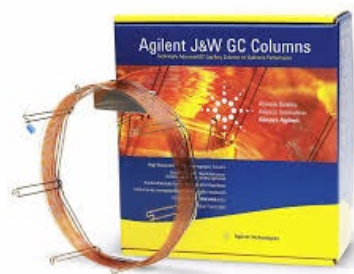
will show a page like this for the currently loaded tune file. It is the charted history of the times that this tune has been performed. You can use the information to help you decide if the source needs cleaning.

- Is the Electron Multiplier Voltage increasing?
- Are the Tuning Ion Ratios decreasing?
- Is the Repeller Voltage increasing?
- The dynamic lens ramping masks the changes as the source ages.

How to clean an Agilent GCMS Ion Source

Have these on hand before you begin:

- Consumables such as filaments, repeller ceramics, ferrules, etc.
- Four clean beakers large enough to lay the ion body down inside.
- DI water
- Pesticide Residue Analysis Grade – Methanol, Acetone, and Hexane.
- Aluminum Oxide powder
- Lint free cloths
- Cotton tipped swabs
- Necessary tools



Create a **Prepare to Vent.M** method with the inlet, oven and transferline at 100 degrees or lower and with a tune called something like **Prepare to Vent.U** that has the source and the quad(s) temperatures set to 100 degrees or lower. Load this method before venting the system for maintenance. When you start it back up after cleaning, the source and quad(s) will only go to 100 anyway, but this keeps them there after you initiate the software, rather than starting to heat up to typical temperatures.

How to clean an Agilent GCMS Ion Source

Cleaning steps:

- Make a thick paste with DI water and Aluminum oxide. [393706201 ALUMINUM OXIDE POWDER 100g]
- Use the cotton-tipped swabs. [8520-0023 Swab 6.0 in LG; cotton tipped, 100/bag]
- Apply a small amount of this paste on the lint-free cloth laying flat on the bench.
[05980-60051 Cloth, lint free 15/PK]
- You are NOT trying to remove metal, only surface contamination. Mechanically, this happens quickly.
- Clean everywhere. Fronts, edges, backs, in the holes, everywhere.

Use DI water to make the abrasive paste so that you do not have to work in a hood. Do not use the green sandpaper on the critical surfaces and holes, it leaves a scratched surface. The only place to use it? See later slide.



How to clean an Agilent GCMS Ion Source

Rinse steps:

- Soapy water – hot tap water with a residue free laboratory detergent. Alconox, for example.
- Clean water – rinse until all the bubbles are gone from the soap.
- DI water – Water removes salts
- Methanol – this step removes the water
- Acetone
- Hexane – this step removes any hydrocarbon residue

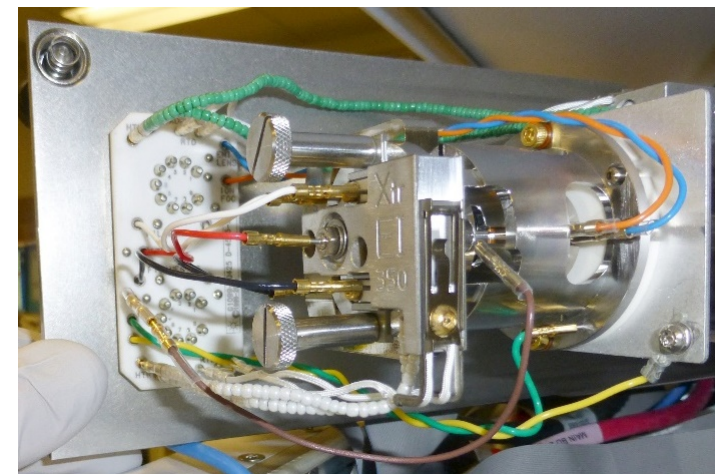
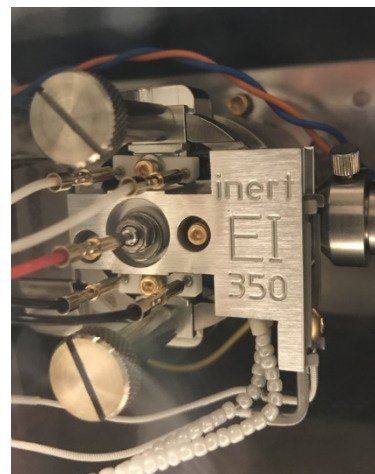
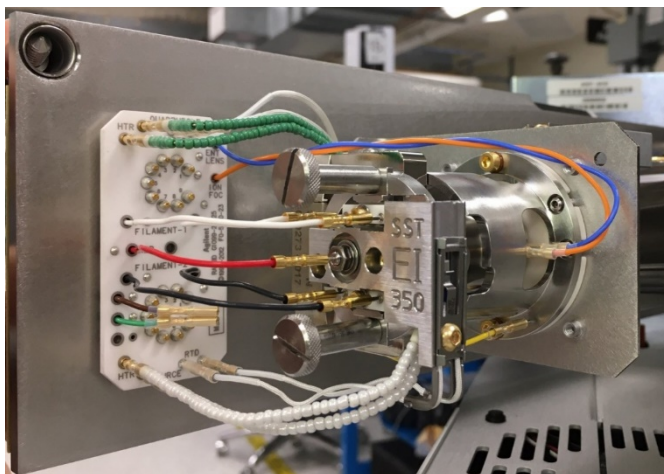
Use a separate beaker for each different type of solvent – four beakers. A top tip is to keep a set of beakers just for source cleaning. Use tweezers to transfer the parts to leave as much residue in the previous solvent as possible. Do not allow the parts to dry in between solvents! This list goes from most polar to least polar on purpose. If you do not really need ultimate cleanliness/sensitivity, you can skip the Acetone and Hexane, but those steps definitely lead to a cleaner source.

Do not sonicate for a long time in each solvent! Long sonication just bangs the pieces up against each other. Three to five minutes in each solvent is sufficient.

How to clean an Agilent GCMS Ion Source

Next:

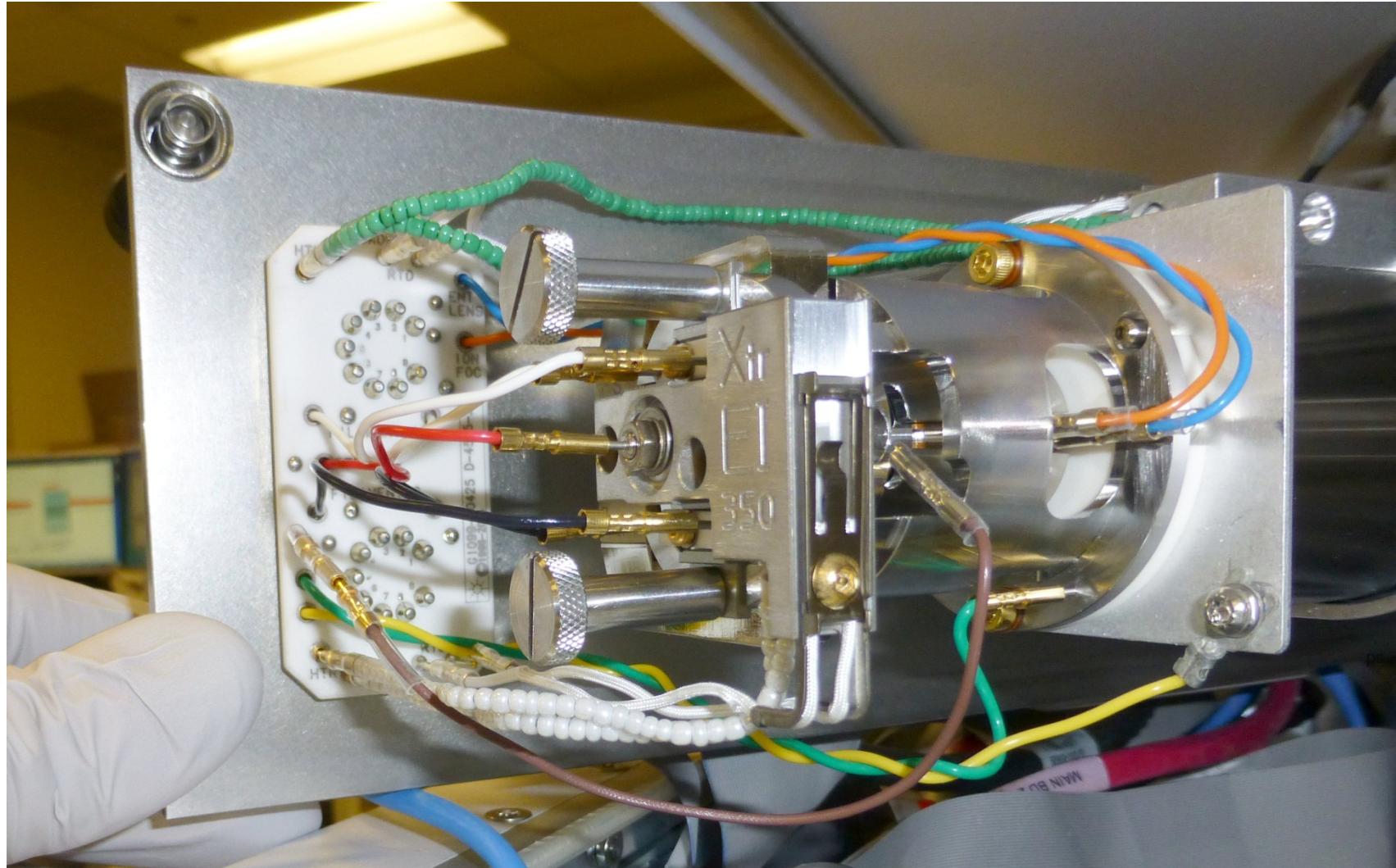
- DO NOT heat up the parts to try to dry them! Absolutely do not heat the parts up above 70 degrees C in an oxygen containing environment.
- Do not bother baking the source parts as they will be very dry after the acetone step and free of organics after the hexane step, which rapidly evaporates.
- Assemble the source and install it in the instrument. Moisture in the air will condense and can attract contaminants. It is better to assemble the source, install it quickly and start it pumping than to worry about carefully drying off the parts.
- In operation the source will be heated in a vacuum! As long as you do not have an air leak, this is the best environment for the source – get it there quickly.



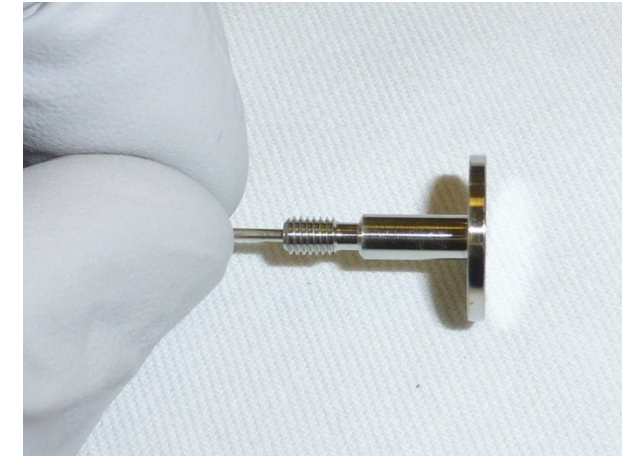
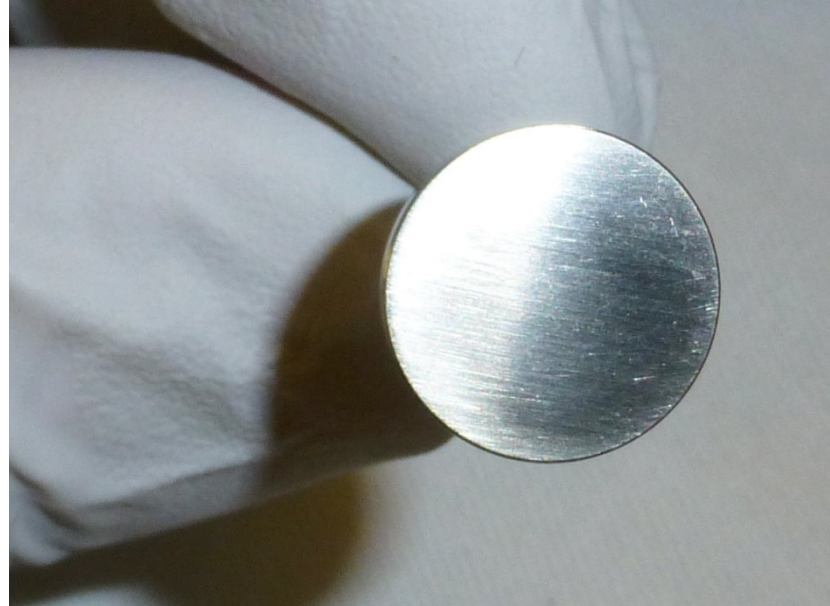
Agilent Single and Tandem Quad GCMS Ion Sources



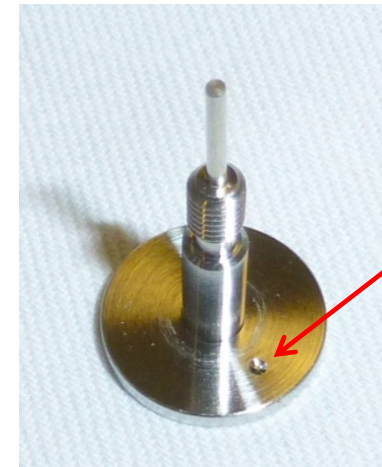
Here's the source.



Repeller

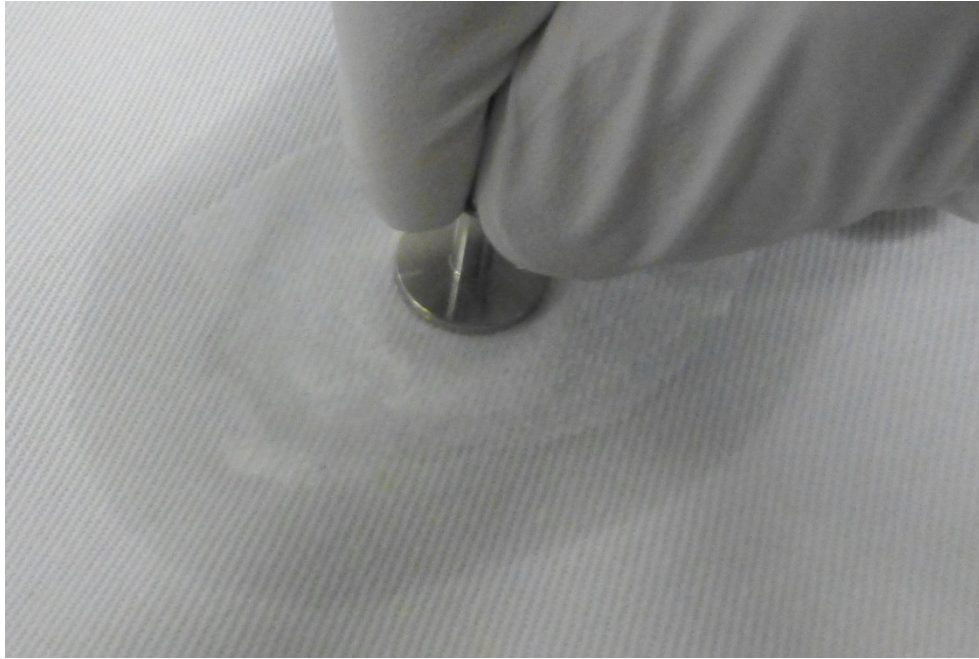


The repeller face must remain flat!



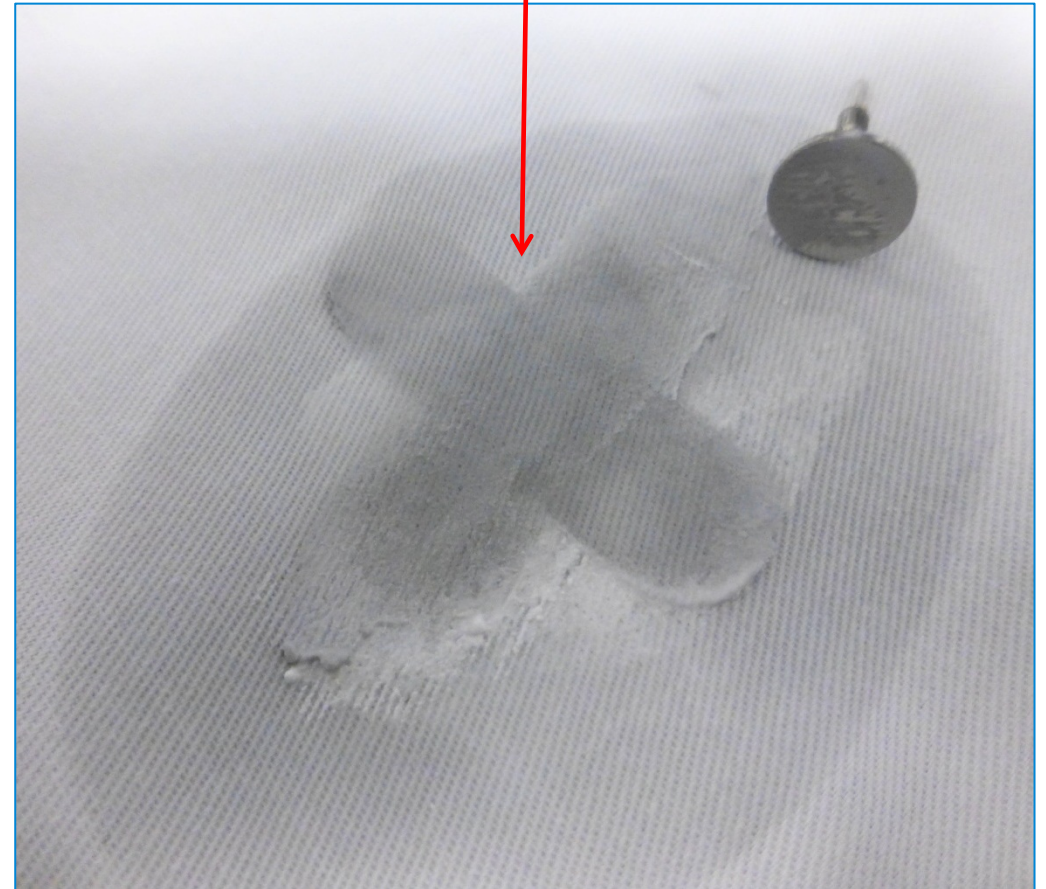
That little dimple denotes that this part is made from the inert metal. Agilent inert and extractor source parts do not have a coating, they are made from an inert alloy.

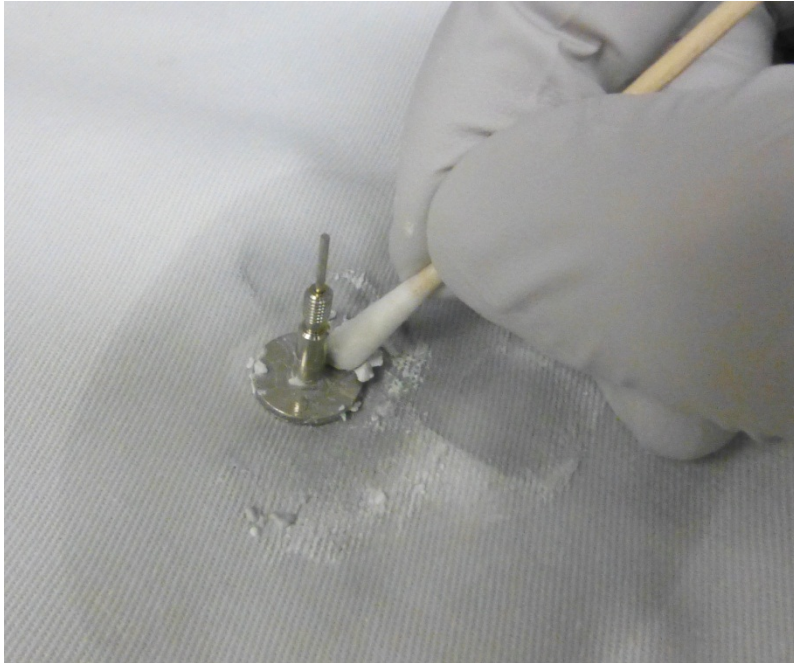
Repeller



The repeller face must remain flat! Do **not** go around in circles - that will wear down the edges of the face. Press it down flat to the bench and then go back and forth on the lint-free, turn it 90 degrees and do some more. You do not need to do this a lot of times, ten times back and forth each direction should be sufficient.

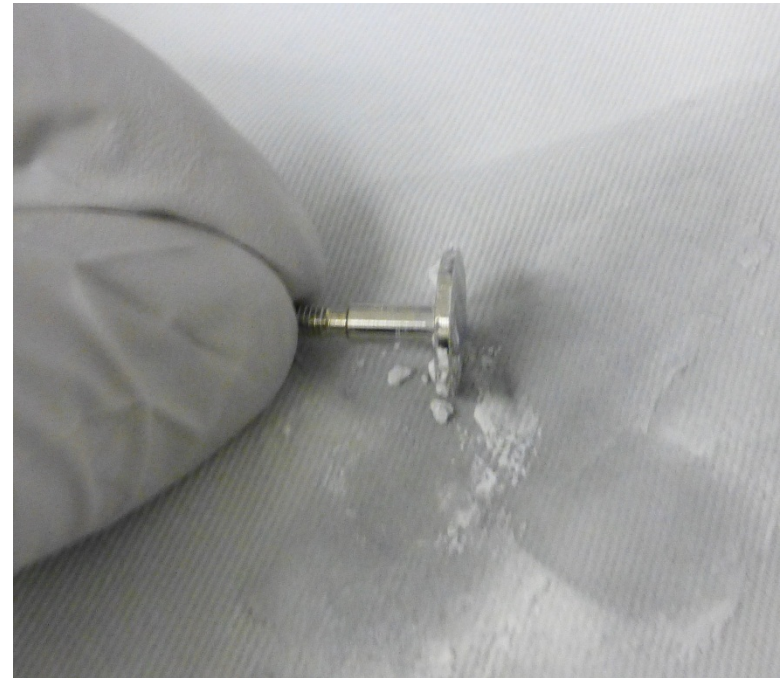
This dark grey deposit is **not** the sign of a dirty source. It is metal that has been removed. Deposits on the face are gone before you see this.





Clean the back. Use the swabs and clean all around. Do not be frugal – use lots of swabs. You can use a swab to clean the face, too...

Clean the edge. Spin it on the lint-free cloth.



Repeller insulators and washers.



These insulators are reasonably clean. They do not have to be perfect but should not be broken. The insulators must do their job and not outgas or be conductive. Make sure to purchase spares.



The large flat washer goes on the bottom next to the ceramic and the curved Belleville spring washer goes on top of that, cup down. The Belleville washer in this picture is cupped side up. It is cupped side down in the left picture.

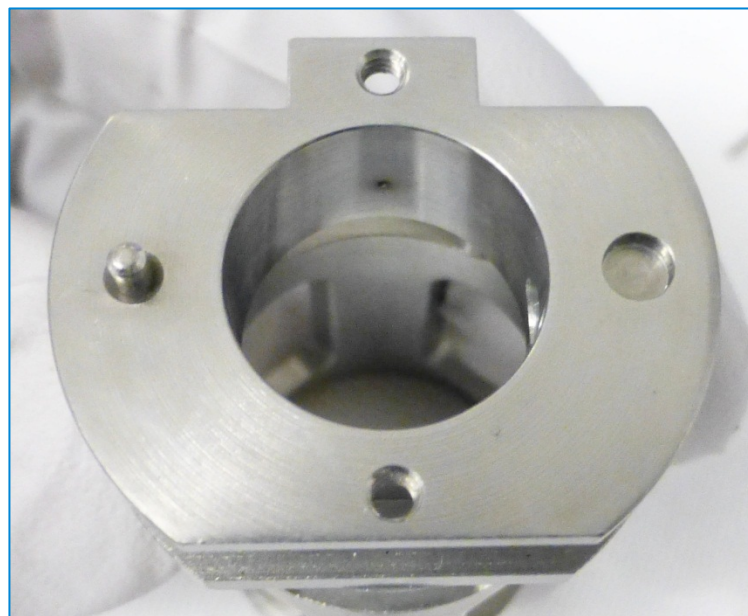


A Belleville washer, or conical washer, is a spring. It is used to hold a very small amount of tension on the repeller to keep it from loosening up and wiggling out of alignment.

Ion Volume.



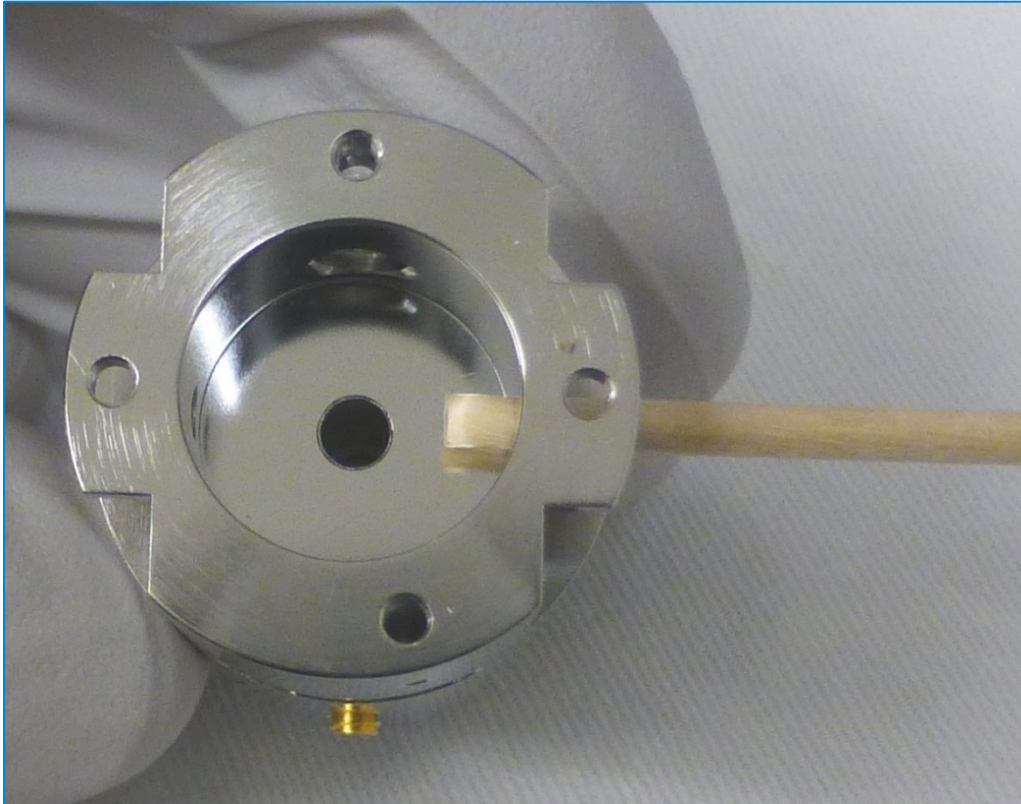
The ion volume, along with the repeller face and the draw out/extractor face, is the most important area to clean. Dip a swab into the DI water and then into the aluminum oxide. Use that to polish the entire inside surface.



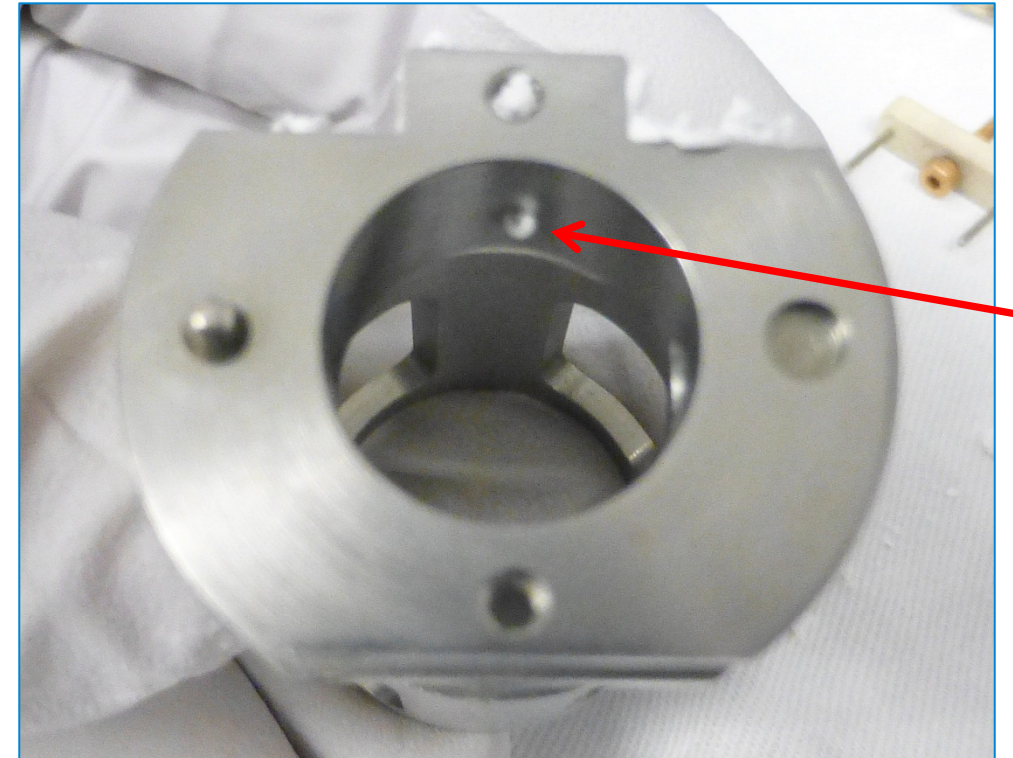
A top tip – use three swabs at a time to get this done a bit quicker.

Ion Volume.

Clean the electron beam hole out **last**. The stick of the swabs that come with the systems can be used along with the aluminum oxide slurry to scrub the electron beam hole in the EI source.

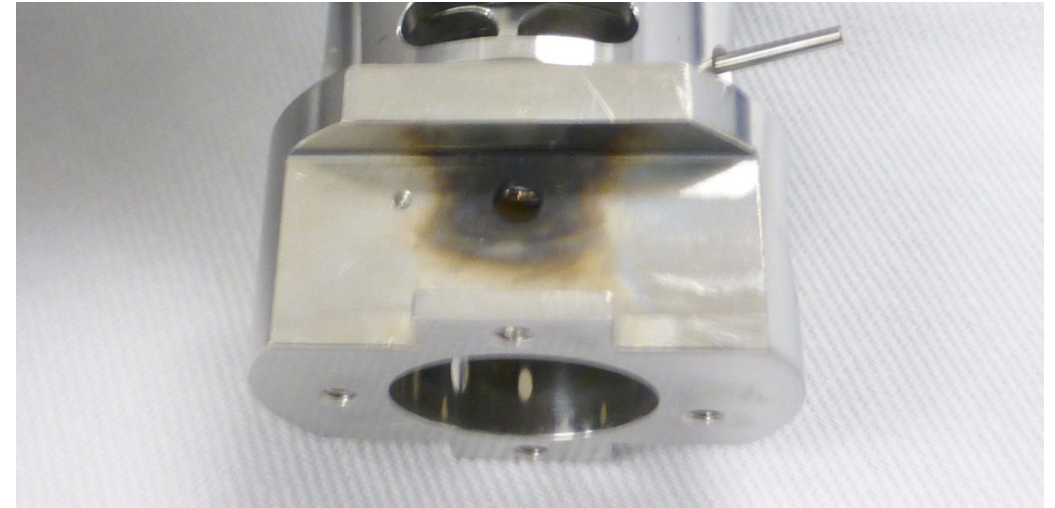


For the CI source, the electron beam hole is too small. It is tempting to break the wood swab and push a sliver through, but it will get stuck! Use the swab to clean the outside and extrude a lot of the slurry through the hole. Alternatively, use a toothpick.



Source Body.

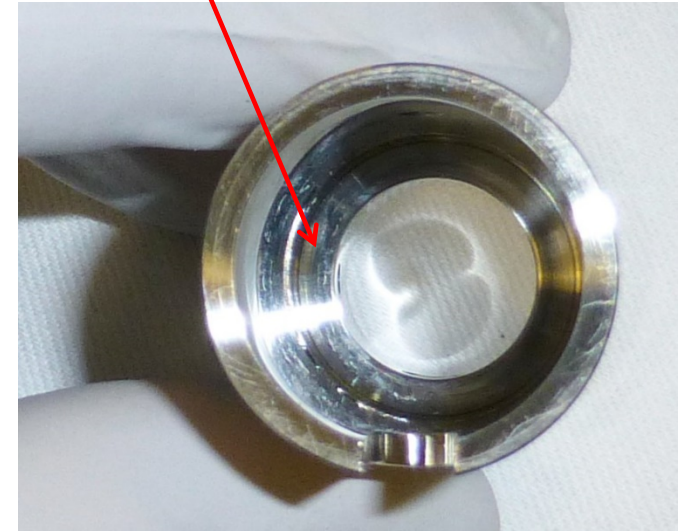
The filament will leave a burn mark on the outside. You may need to use the green sandpaper to get rid of most of it and then polish it with the slurry. This is the only place to use the green sandpaper.



It can be quite bad if there is a leak, if the samples are very concentrated, or if it is not cleaned it very often. It feels good to get this completely perfect, but it is actually not necessary for ultimate performance.

Source Body.

Clean the entire inside and outside of the source body including the slots. This may not be necessary if the system only analyzes clean samples, but can become critical over time. If the source has been cleaned once and still has lower 219 and 502, clean this thoroughly again. Don't lose the setscrew(s)!



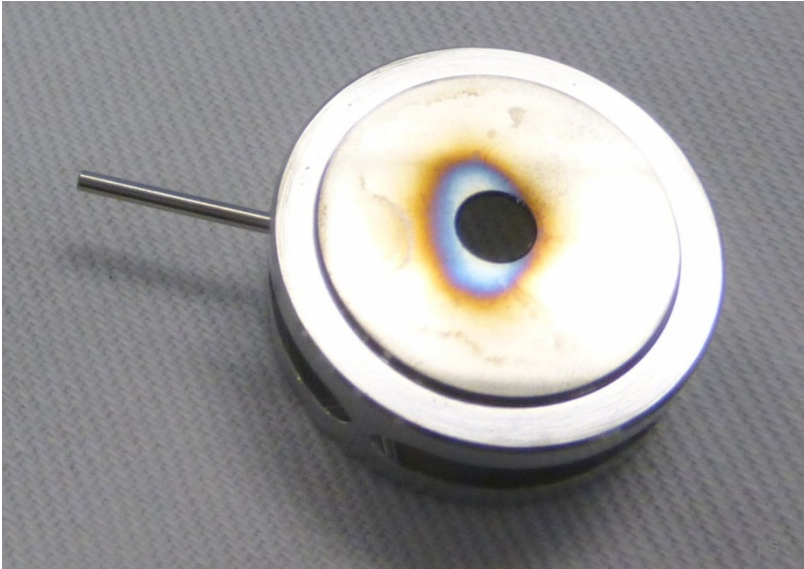
Scrub the area where the drawout plate/extractor lens rests. This area is the electrical connection to the drawout (the drawout is at ground potential). The extractor lens insulator rests there and does not make a seal so there is a chance for contamination.

Draw out and spacer or extractor.

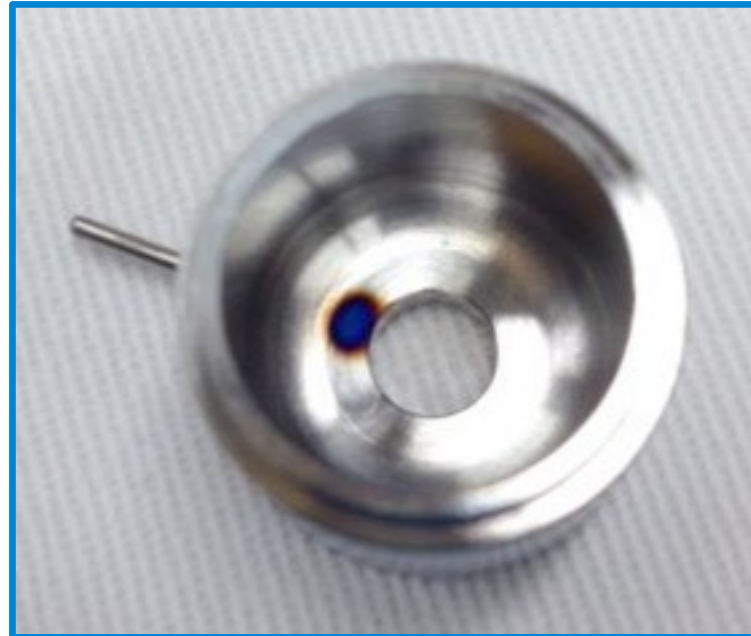
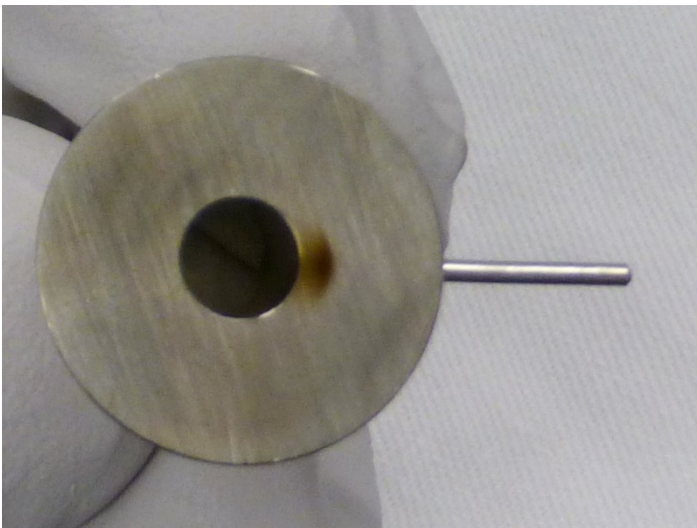
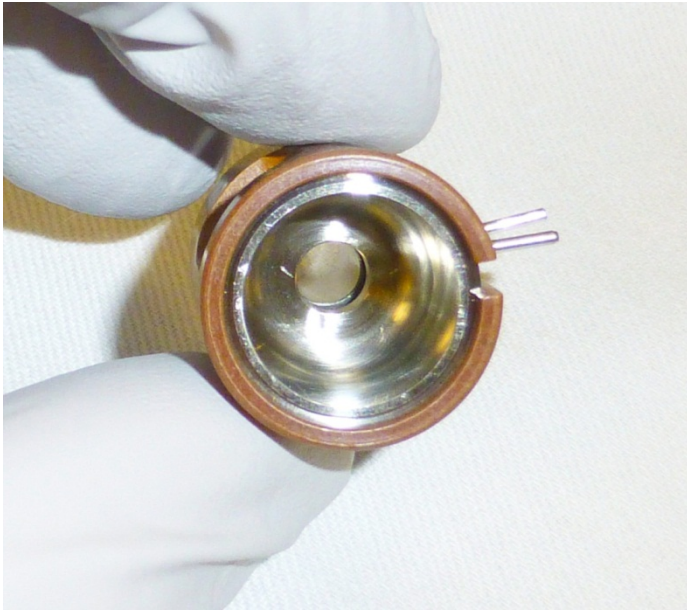


Clean both faces of the drawout – so you don't have to remember which is the cleaner side. Use the swab, either the cotton or the wood stick, and clean the inner edge of the center hole as well. At the very least, sonicate the spacer along with the other source parts. Clean all surfaces if you are having issues.

Clean the face of the extractor, the lip where the insulator sits and the center of the hole. Also, flip it over and clean the entire inside. This may not be necessary if the system only analyzes clean samples, but can become critical over time.



Ion focus



The ion focus needs to have the entire inside of the cup, the edge of the center hole and the backside face cleaned. Try not to round over the lip edge of the center hole.

You may notice a burn mark inside as seen in the center photo. The very low molecular weight ions that exit the source are influenced by the magnet and hit there. This blue spot may indicate an air leak.

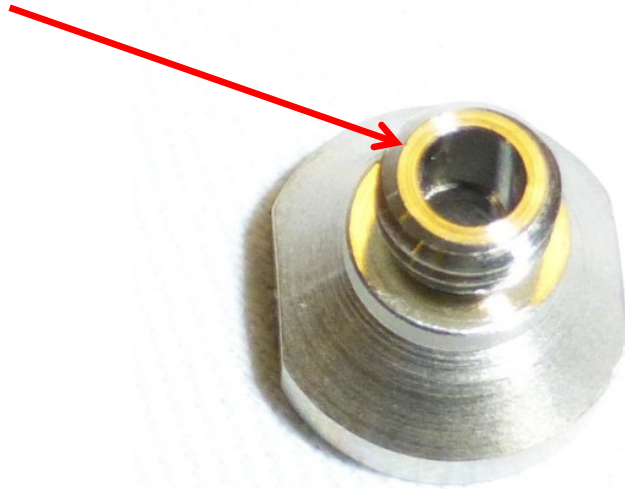
Entrance Lens

The entrance lens reduces fringe magnetic field effects from the end of the quad. Clean the flat face, the center tube and the outside end that goes into the quad.



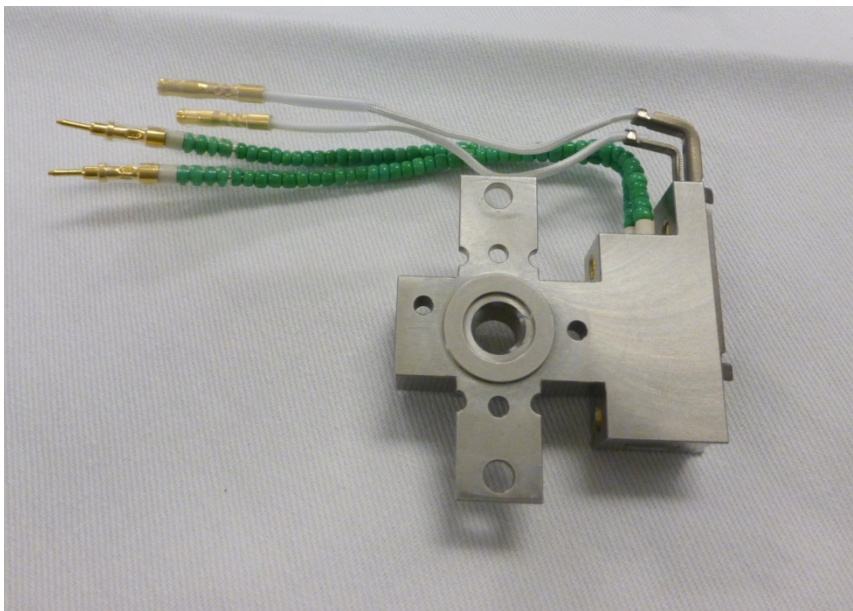
Transferline Socket.

The tip of the transferline socket that is part of some GCMS ion sources sits inside the ion volume. Make sure and clean the end thoroughly. You can clean the inside of the cone if you'd like.



You do not need to clean.

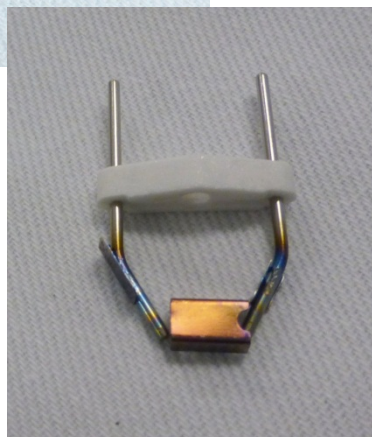
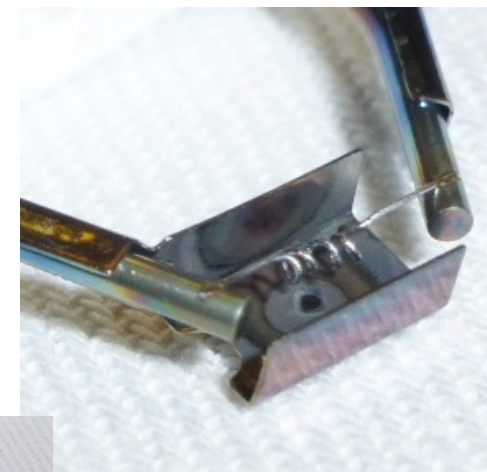
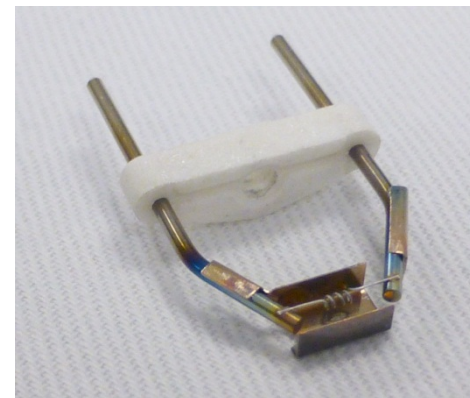
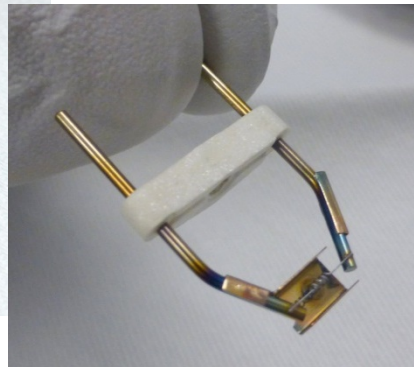
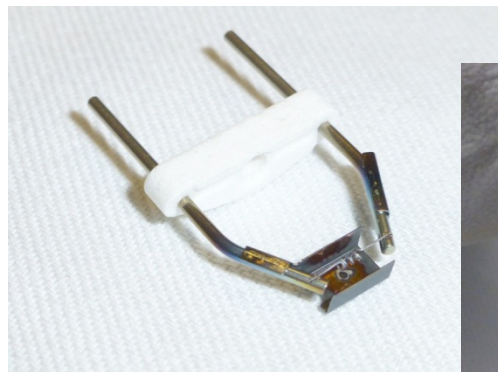
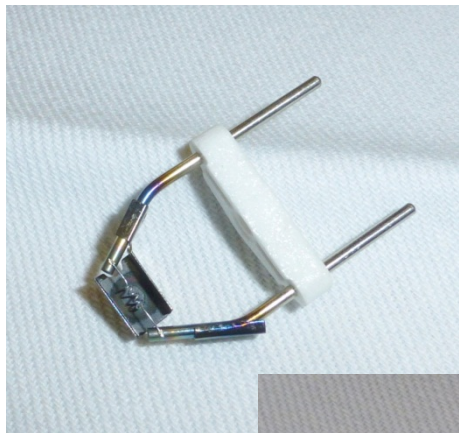
Do not worry about any discoloration on the source heater. The source heater should measure ~20 ohms and the sensor should measure ~109 ohms at room temperature



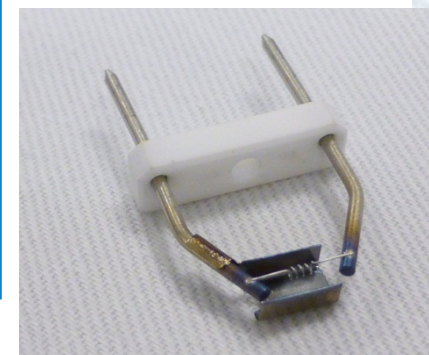
On sources with a drawout plate, squeeze it together during reassembly before you tighten the setscrew to insure good contact between the drawout and the source body.



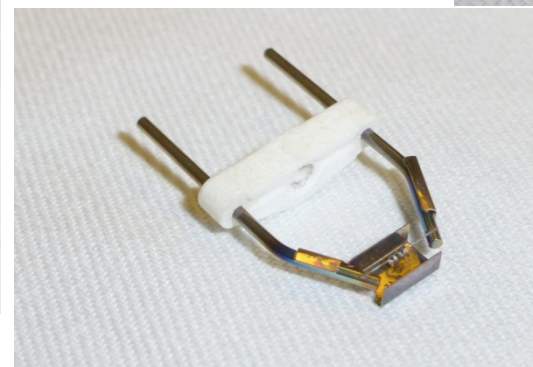
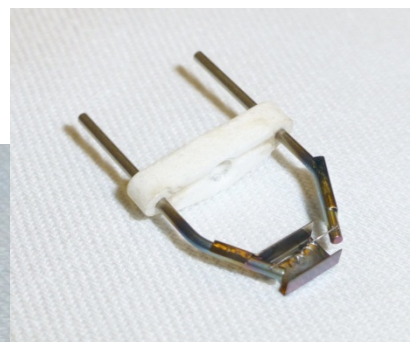
Filaments.



The colors around a filament tell you about the quality of the vacuum and a bit about the samples. Blue indicates that there was air. Blue may still work!

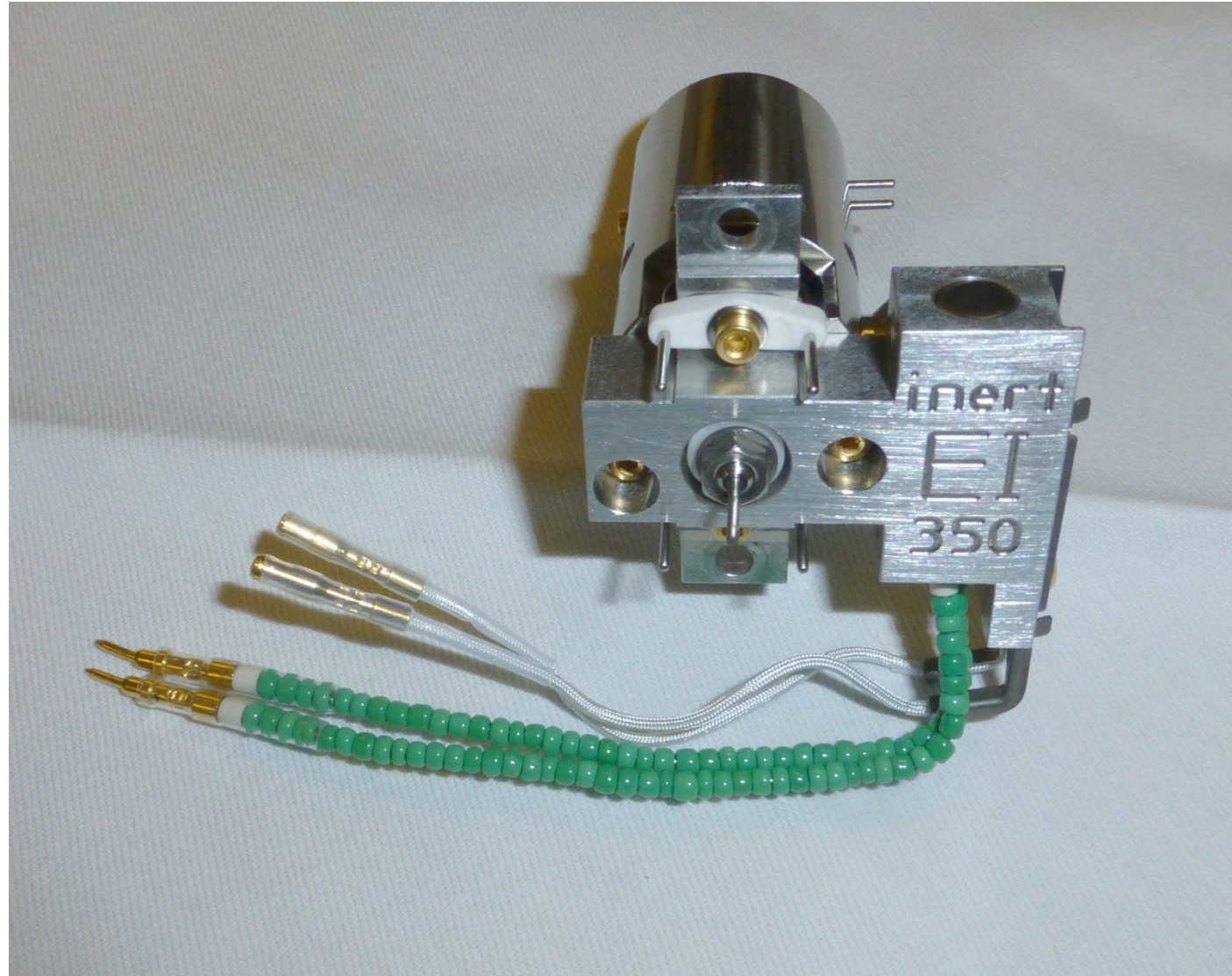


Bent filaments are bad!
Bent or misaligned reflector/targets are bad!
So are cracked insulators.
Inspect the wire – replace if it is not the same thickness everywhere.



Purchase spares!

Ready to reinstall.



Is more cleaning necessary?

Go to the GC front panel:

8890:

- Service Mode
- Instrument Usage

7890:

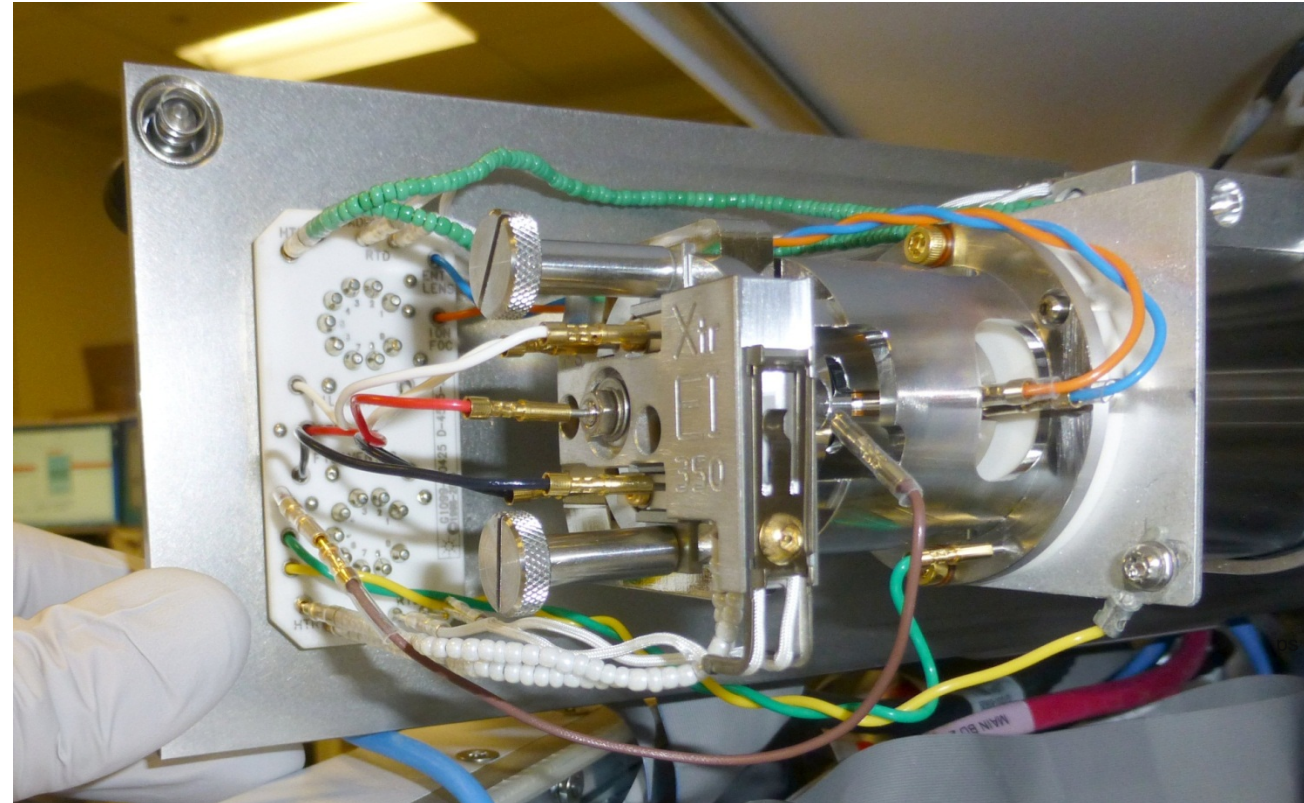
- Service Mode
- Diagnostics
- Instrument Status

6890:

- Options
- Diagnostics
- Instrument Status

If the instrument has had a hard life and done many runs you may need to do a more remedial cleaning once every XX years.

How many runs are a lot? It depends on the application, the sample, the concentration range, the site, and the operator.



You're almost done:

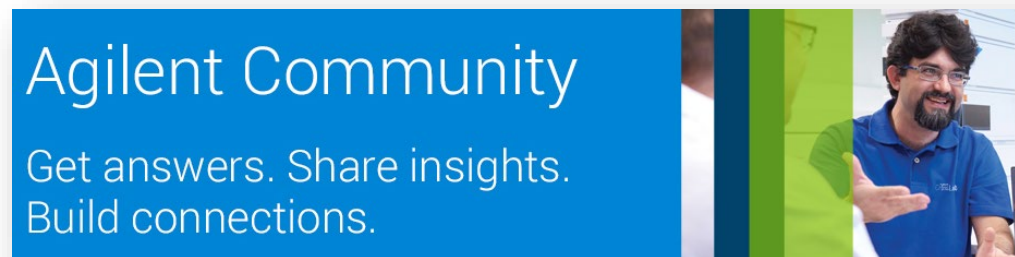
- Your patience will be rewarded. Take your time to ensure that the vacuum is acceptable before you proceed.
- Do not start until the vacuum is less than 6 or 7 x 10⁻⁵! Confirm in Manual Tune that the GCMS is leak tight – with all the zones cold.
- If there is no air leak, heat it up by loading a typical method with the normal tune file.
- Bake – in a perfect world, bake the source at 300 for two to three hours before proceeding. If you are in a rush, bake for 0.1 or 0.2 hours before testing and then set it to bake for a couple of hours when you leave for the day.
- Tune
- Air and Water check.
- Tune evaluation. The water will probably still be high. This is normal. Water will be gone in 8 hours or less.
- Make sure that **both** filaments will produce a spectrum before running the system.
- Document the maintenance in the instrument logbook.

See also: How to clean an Agilent GCMS Ion Source <https://community.agilent.com/docs/DOC-1168>

Resources for Support

<https://community.agilent.com/>

- Agilent support resources:
<https://community.agilent.com/community/resources>
- Collection of support material
<https://community.agilent.com/docs/DOC-1856>
- Agilent chemistries and supplies information:
<http://www.agilent.com/chem/agilentresources>
- Agilent University
<http://www.agilent.com/crosslab/university>
- Youtube – [Agilent Channel](#)



Additional online e-seminars and educational material

<https://www.agilent.com/en/training-events/eseminars>

Agilent Atomic Webinar Series

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<https://www.agilent.com/en/training-events/eseminars/atomic-spectroscopy>

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<https://www.agilent.com/en/training-events/eseminars/openlab2>

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Becoming a Better Chromatographer



<https://www.agilent.com/en/training-events/eseminars/lc-lc-ms-column-e-seminars>
<https://www.agilent.com/en/training-events/eseminars/gc-gc-ms-webinars>

Agilent Chromatography and Mass Spec Educational Webinar Series



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Get individualized assistance. Our remote service engineers are available by **phone or video conference** to answer your questions – including support on compliance issues or performing risk assessments. [Contact us](#) or explore [online resources](#) to do-it-yourself.



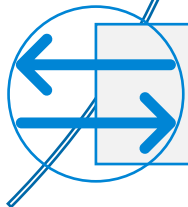
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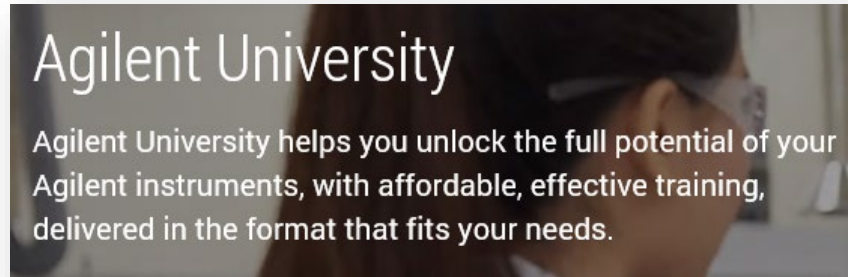
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Contact Agilent Technical Support



1-800-227-9770 Option 3, Option 3:

Option 1 for GC/GCMS Columns and Supplies

Option 2 for LC/LCMS Columns and Supplies

Option 3 for Sample Preparation, Filtration and QuEChERS

Option 4 for Spectroscopy Supplies

Option 5 for Chemical Standards

800 Phone lines available 8-5 in all US time zones

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spp-support@agilent.com

spectro-supplies-support@agilent.com

chem-standards-support@agilent.com

Additional support – Live one-on-one video conference with our service team

- Agilent Service Engineers are currently running live video conferences to remotely support labs around the world.
- To request a one-on-one video conference with one of our service engineers, please send your request through the polling survey and indicate the following:
Name, Company, and instrumentation requiring support or questions
- We will get back to you shortly to schedule a video conference

In Summary



We at Agilent understand the restrictions and hardship many of you are going through because we're experiencing them as well

Given all that we are going through, Agilent remains a stable and continuing resource to meet and exceed your analytical measurement needs

We are open for business and here to help



Any questions?

All unanswered chat questions will be followed up post-event.
Slides will be distributed to the email address you registered with.