

Agilent GCMS Ion Source Cleaning

Agilent Customer Community
25Mar2021



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.

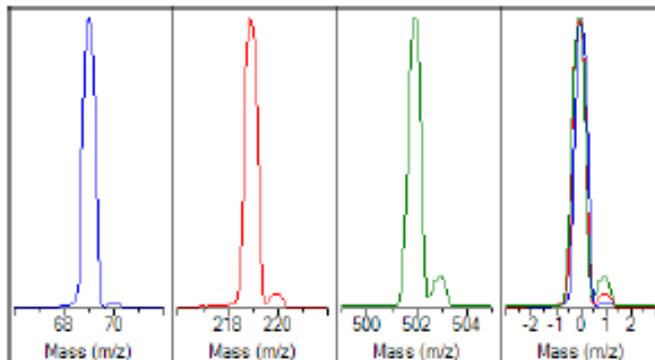
How to clean an Agilent GCMS Ion Source

Autotune - 5977

6/12/2014 12:41:51 PM (UTC-7:00)
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5977A

US1410M217



Ion Polarity	Pos	Mass Gain	-518
Emission	34.6	Mass Offset	-34
Electron Energy	70.0	Amu Gain	1383
Filament	1	Amu Offset	122.63
Repeller	34.90	Width219	-0.006
Ion Focus	84.4	DC Polarity	Pos
Entrance Lens	17.6	HED Enable	On
Ent Lens Offset	15.48	EM Volts	1588.2
Ion Body	0.00	Extractor Lens	0.00
		Scan Speed	3
		Averages	3
PFTBA	Open	Step Size	0.10

Actual m/z	Abund	Rel Abund	Pw50
69.00	378,572	100.0%	0.60
218.90	254,565	67.2%	0.61
501.90	14,430	3.8%	0.60

Temperatures and Pressures

MS Source	230 Turbo Speed	100.0
MS Quad	150 Hi Vac	1.40e-05

Any autotune program adjusts multiple parameters to achieve the desired results. Every time a tune is performed, the parameters may be different and that will affect the system's response.

System Verification - Tune (Detector Optimization) Portion

```

Instrument Name      : 5977A
DC Polarity         : Positive
Filament            : 1
BasePeak should be 69 or 219
Position of mass 69              69.00    OK
Position of mass 219             219.00    OK
Position of mass 502             502.00    OK
Position of isotope mass 70       70.01    OK
Position of isotope mass 220      220.00    OK
Position of isotope mass 503      503.00    OK
Ratio of mass 70 to mass 69(0.5 - 1.6%)  1.10    OK
Ratio of mass 220 to mass 219(3.2 - 5.4%)  4.35    OK
Ratio of mass 503 to mass 502(7.9 - 12.3%) 10.20    OK
Ratio of 219 to 69 should be > 40% and is 121.99  OK
Ratio of 502 to 69 should be > 2.4% and is  8.69    OK

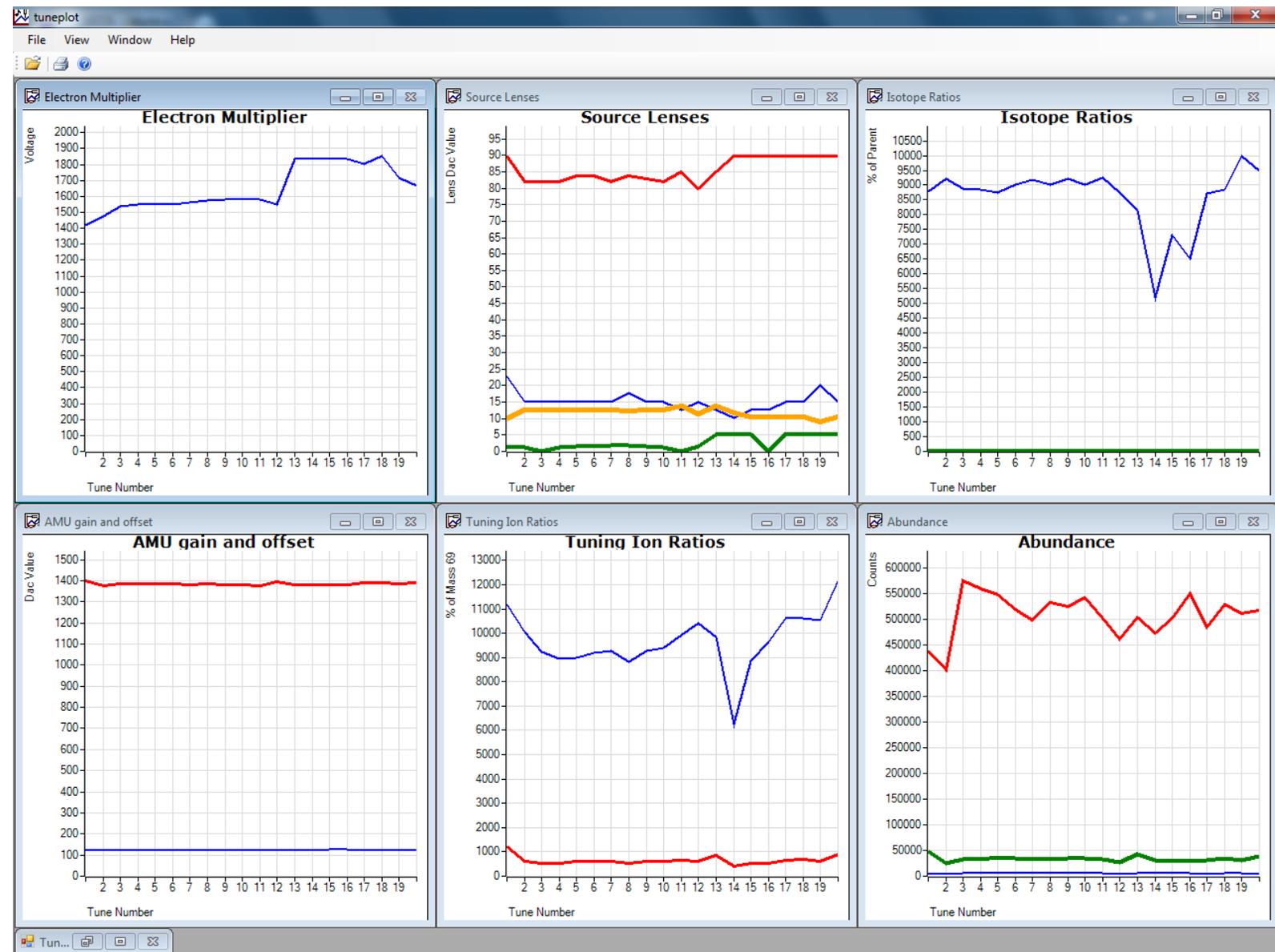
Mass 69 Precursor (<= 3%)           0.06    OK
Mass 219 Precursor (<= 6%)          0.11    OK
Mass 502 Precursor (<= 12%)         0.41    OK

597x Air and Water Check
Mon Aug 25 18:22:12 2014
D:\MassHunter\GCMS\1\5977\air.u
Instrument: 5977A
US1410M217

Testing for a leak in the system
Ratio of 18 to 69 (<20%)             0.64    OK
Ratio of 28 to 69 (<10%)             1.95    OK

Electron Multiplier Voltage          1599    OK
    
```

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°C and with a tune called something like **Prepare to Vent.U** that has the source and the quad(s) temperatures set to 100°C. 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°C 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 slide 17.

How to clean an Agilent GCMS Ion Source

Rinse steps:

- Clean water – rinse until the alumina residue is gone
- 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.

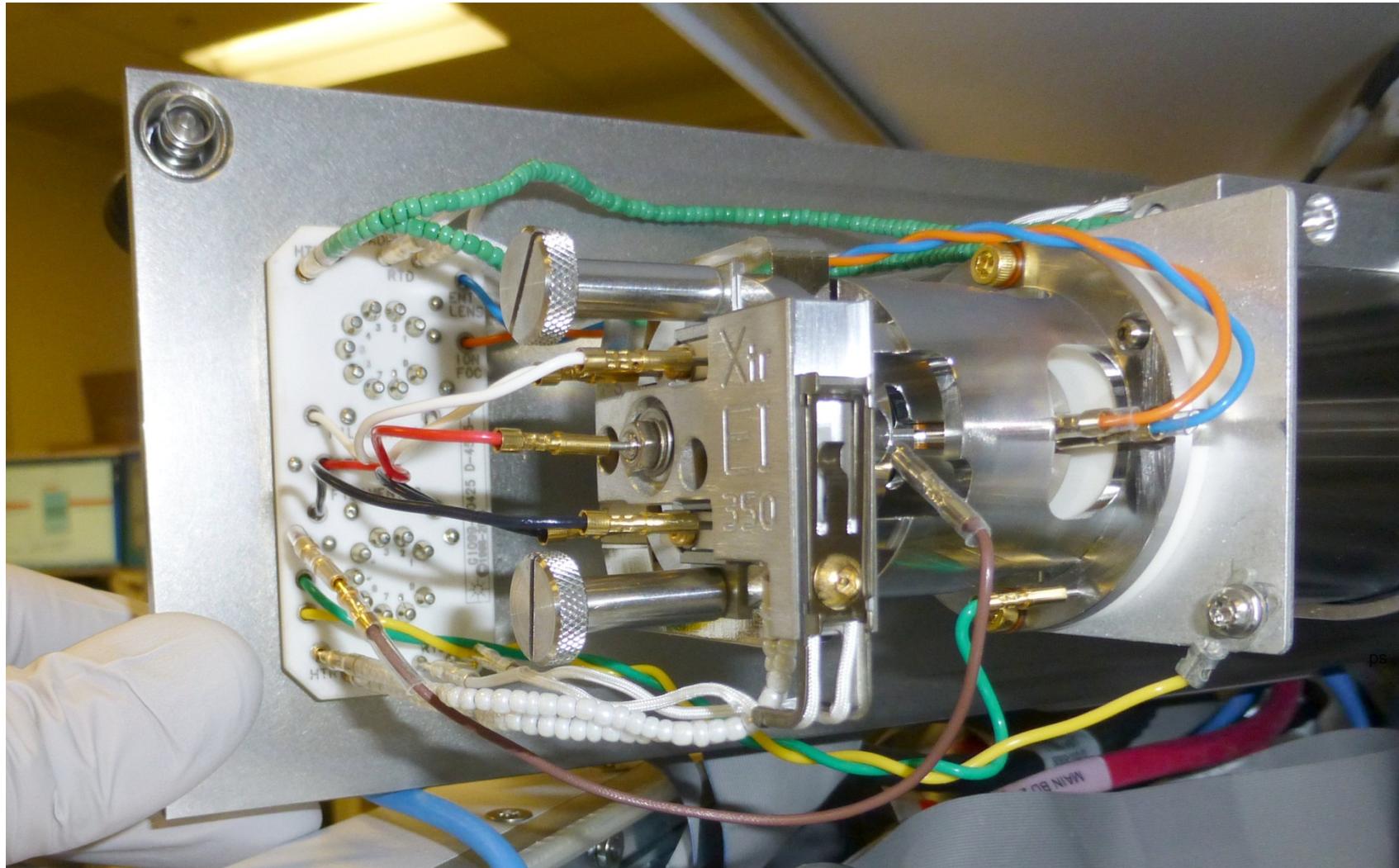
Do not sonicate for a long time in each solvent! Long sonication just bangs the pieces up against each other. Three to five minutes in each solvent is sufficient.

How to clean an Agilent GCMS Ion Source

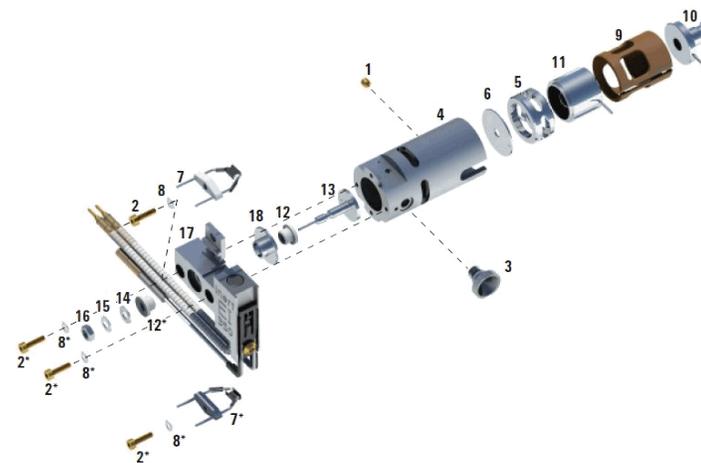
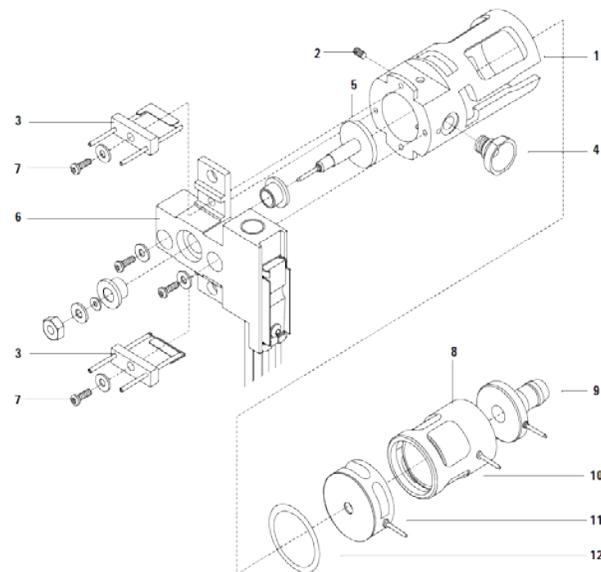
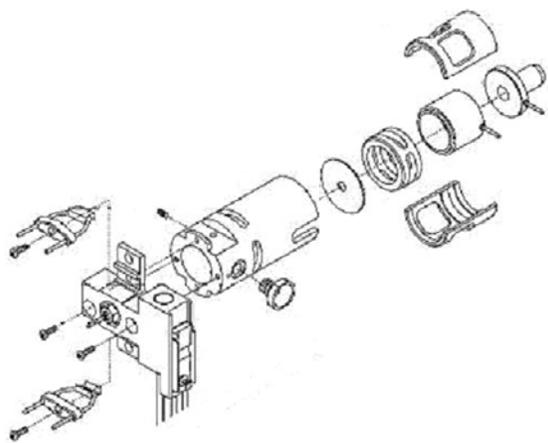
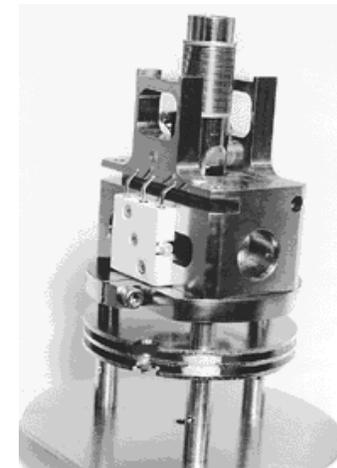
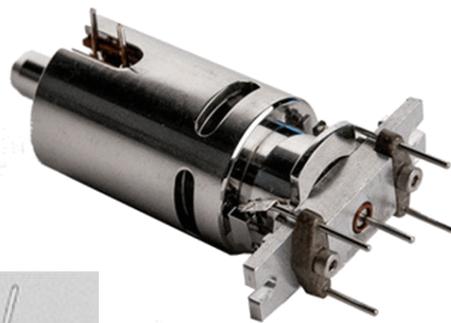
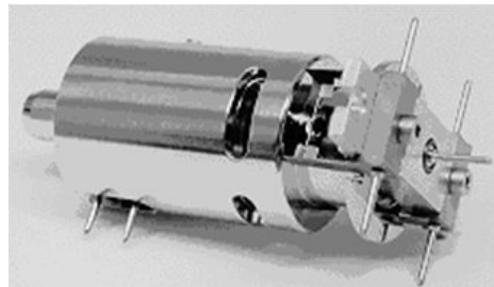
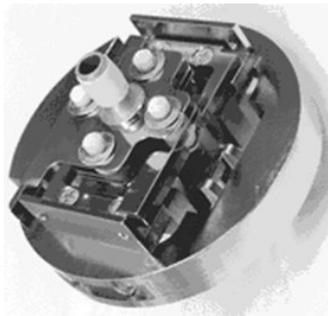
Next:

- DO NOT heat up the parts to try to dry them! Absolutely do not heat the parts up above 70°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.

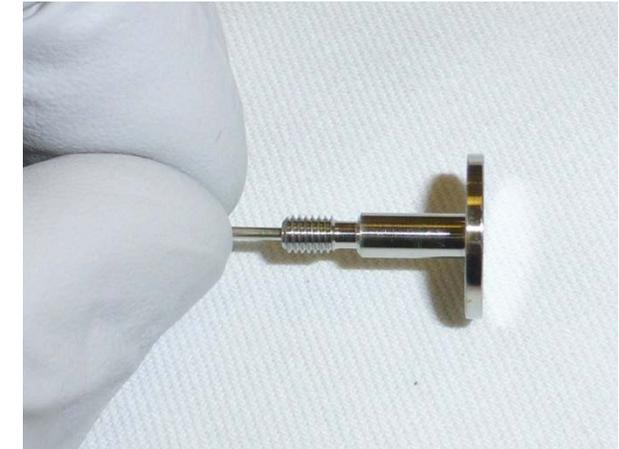
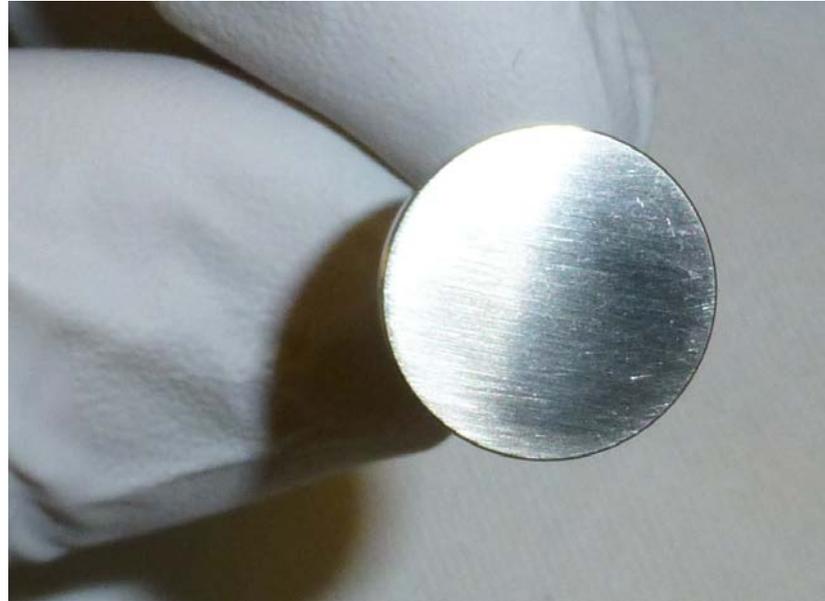
Here's the source.



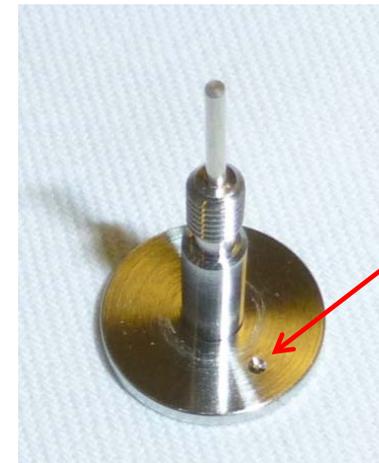
Take it apart.



Repeller

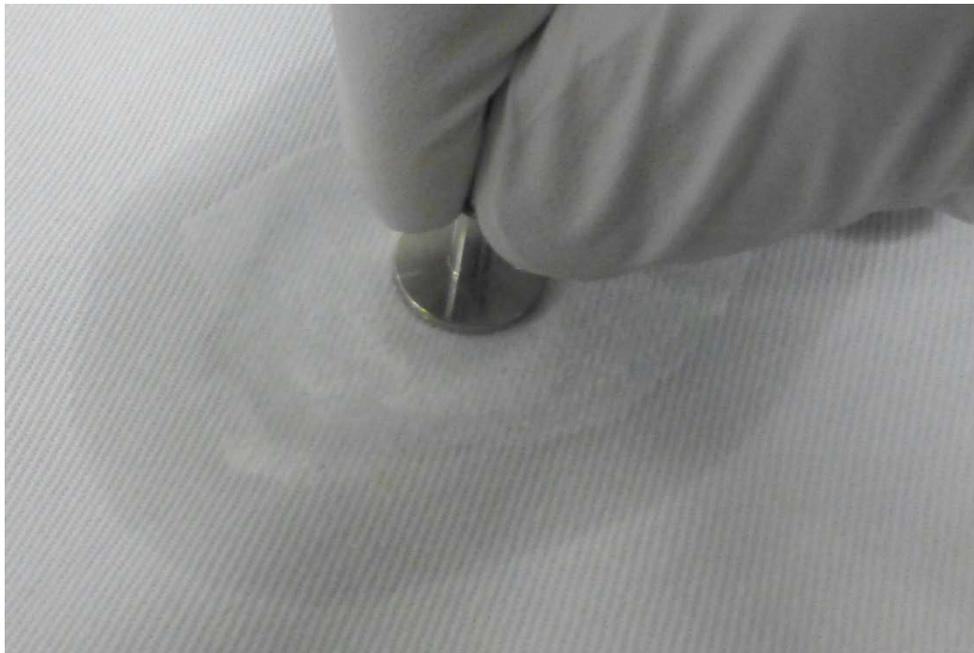


The repeller face must remain flat!



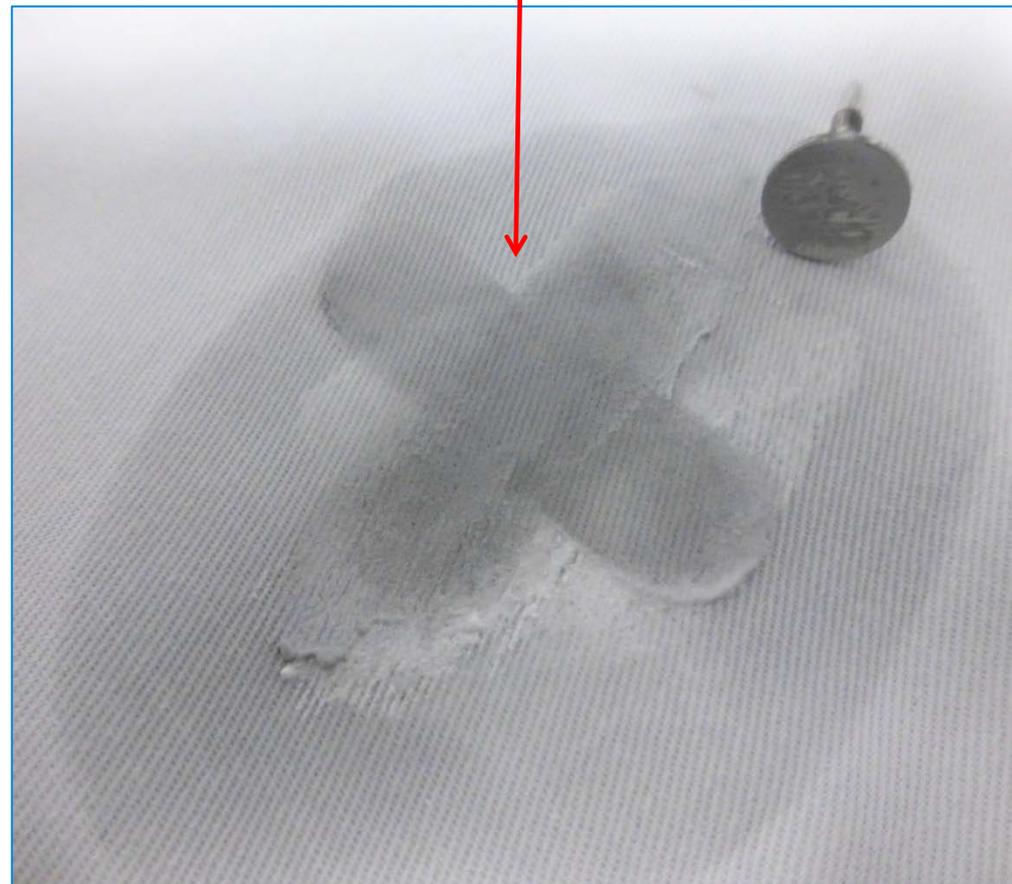
That little dimple denotes that this part is made from the inert metal. Agilent inert and extractor source parts do not have a coating, they are made from an inert alloy.

Repeller

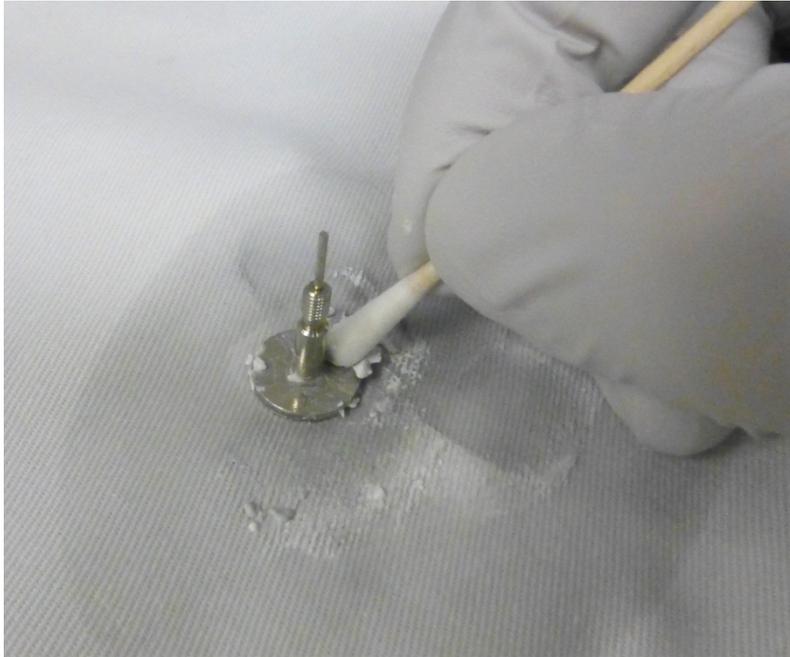


The repeller face must remain flat! Do **not** go around in circles - that will wear down the edges of the face. Press it down flat to the bench and then go back and forth on the lint-free, turn it 90 degrees and do some more. You do not need to do this a lot of times, a handful of times back and forth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



Repeller



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, remembering that any contamination is invisible. 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.

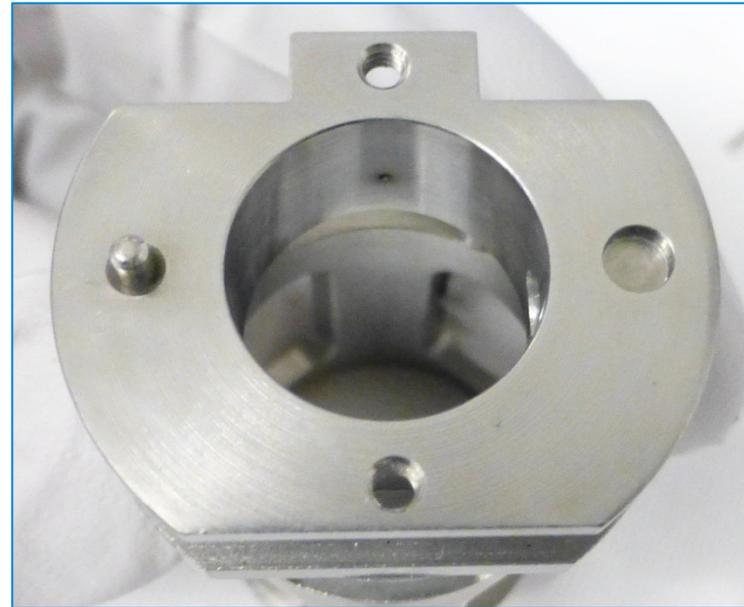


A Belleville washer, or conical washer, is a spring. It is used to hold a very small amount of tension on the repeller to keep it from loosening up and wiggling out of alignment.

Ion Volume.



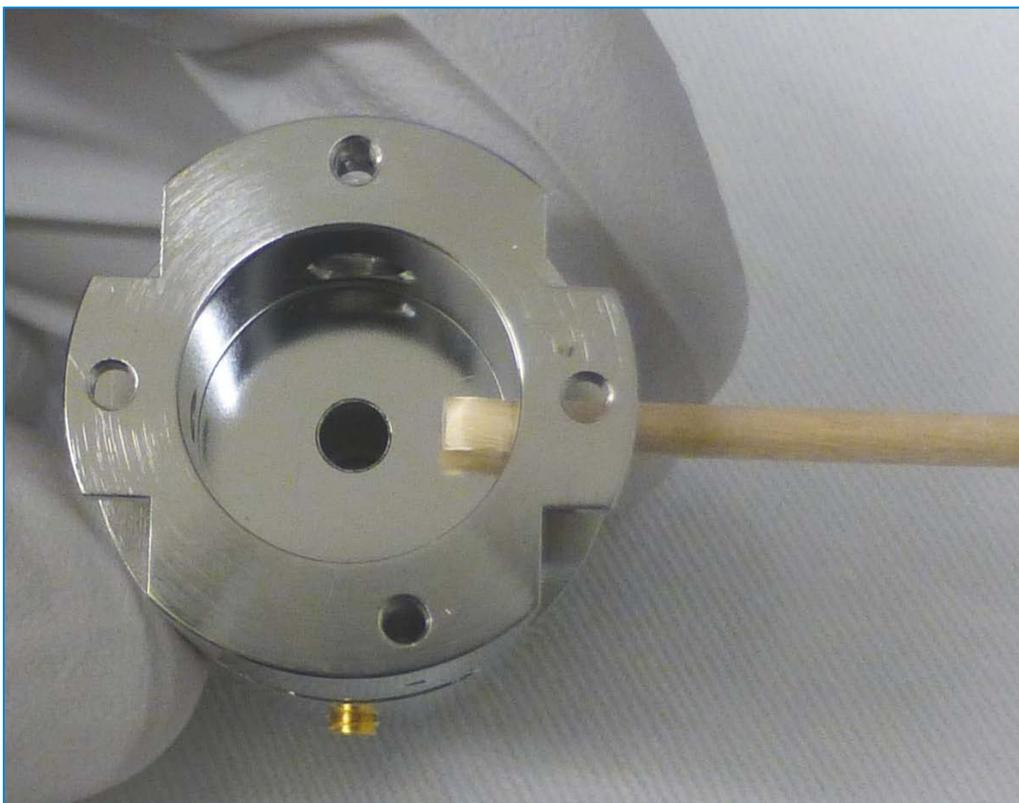
The ion volume, along with the repeller face and the draw out/extractor face, is the most important area to clean. Dip a swab into the DI water and then into the aluminum oxide. Use that to polish the entire inside surface.



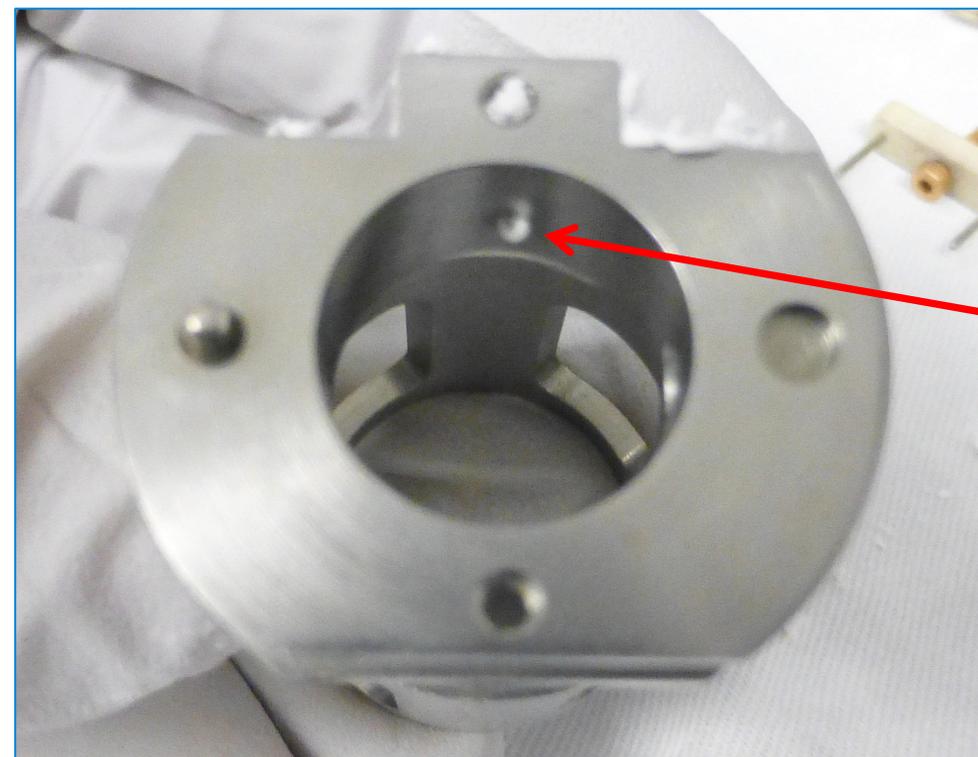
A top tip – use three swabs at a time to get this done a bit quicker.

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.

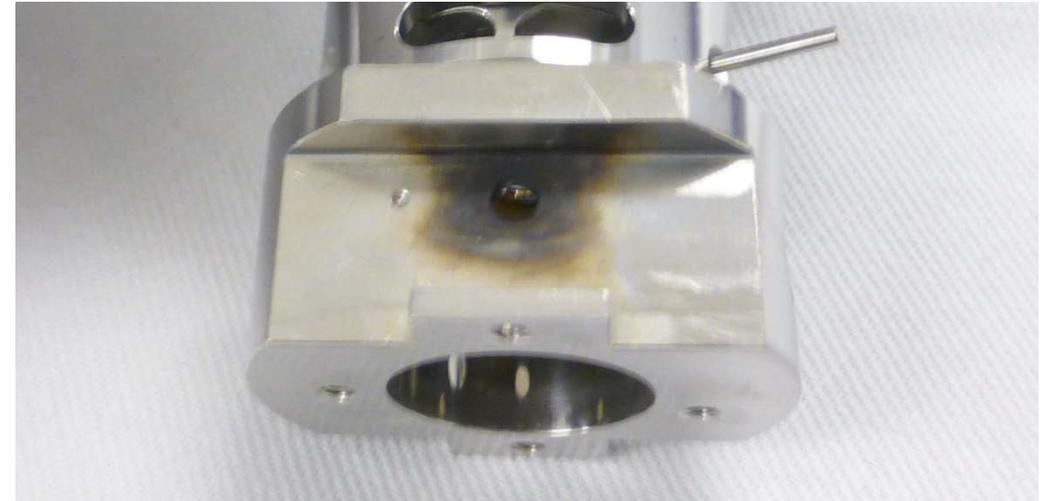


For the CI source, the electron beam hole is too small. It is tempting to break the wood swab and push a sliver through, but it will get stuck! Use the swab to clean the outside and extrude a lot of the slurry through the hole. Alternatively, use a toothpick.



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.

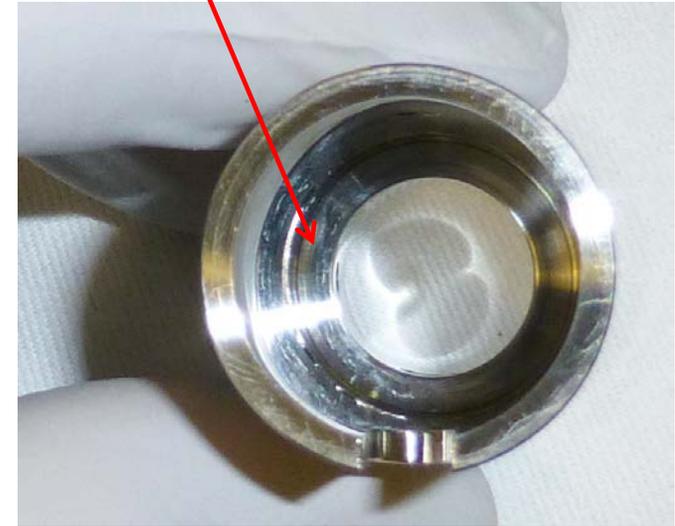


It can be quite bad if there is a leak, if the samples are very concentrated, or if it is not cleaned it very often. It feels good to get this completely perfect, but it is actually not necessary for ultimate performance.

Alumina is ~2400 grit. Green sandpaper is ~1200 grit. Higher number is finer grit.

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 low 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 source body potential). The extractor lens insulator rests there and does not make a tight seal so there is a chance for contamination.

Draw out and spacer or extractor.

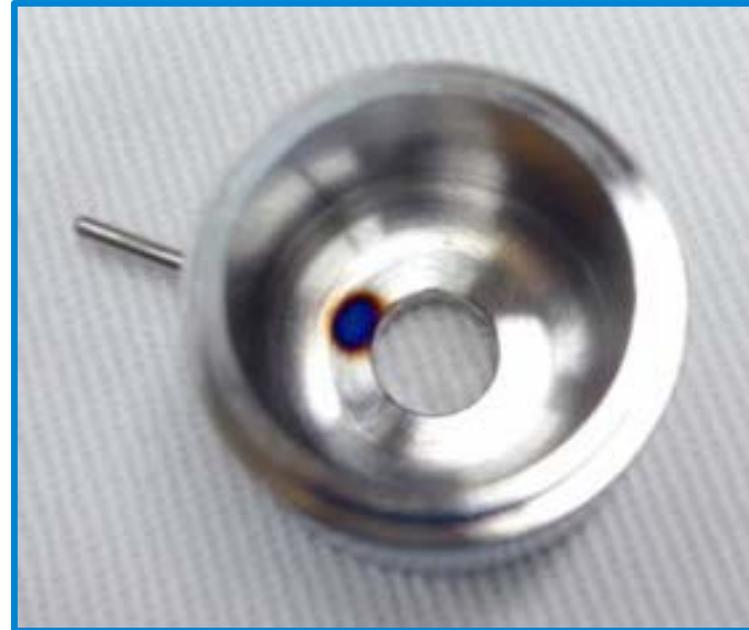
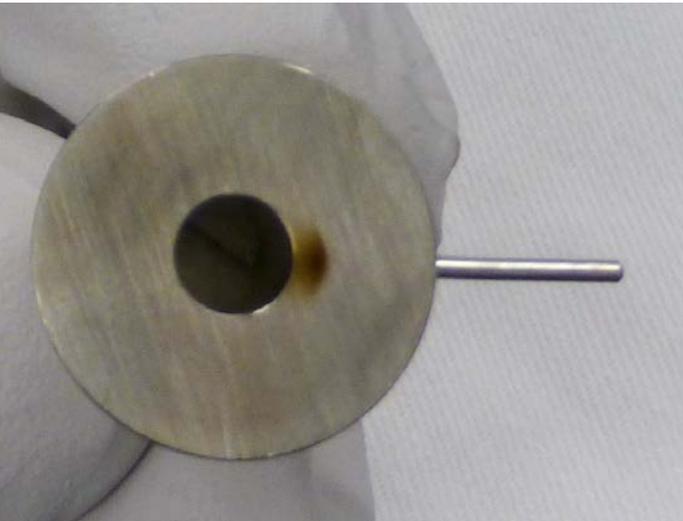
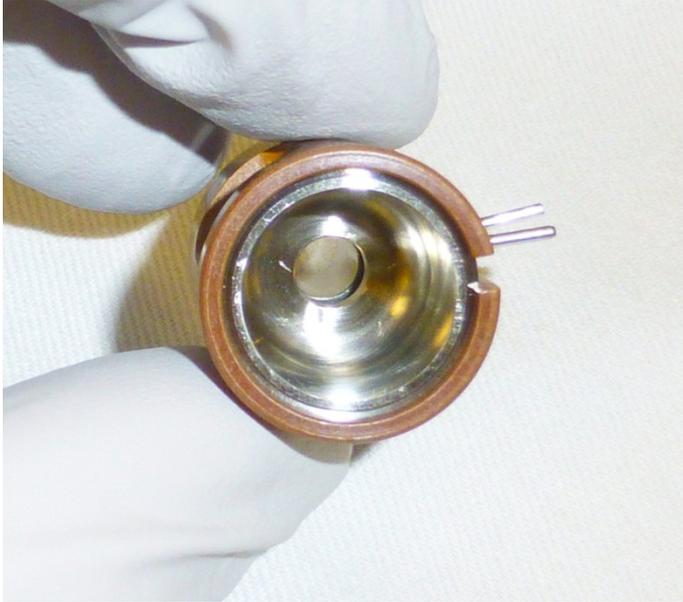


Clean both faces of the drawout – so you don't have to remember which is the cleaner side. Use the swab, either the cotton or the wood stick, and clean the inner edge of the center hole as well. At the very least, sonicate the spacer along with the other source parts. Clean all surfaces if you are having 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. This is caused by very low molecular weight ions that exit the source and hit there. This blue spot may indicate an air leak.

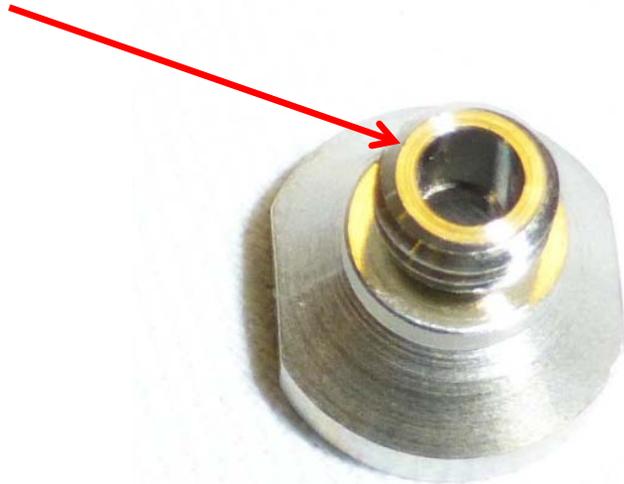
Entrance Lens

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.



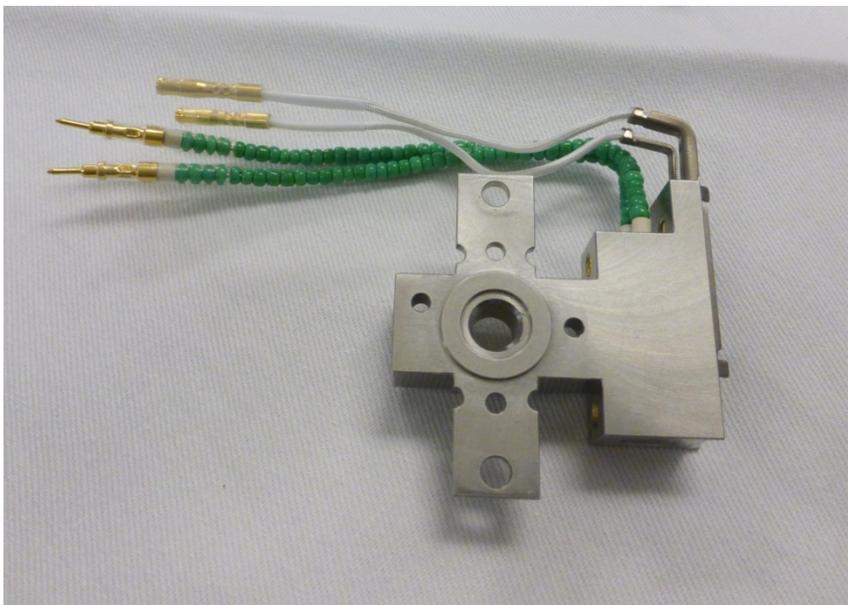
Transferline Socket.

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.

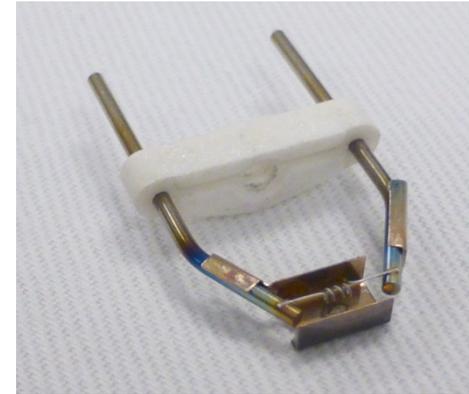
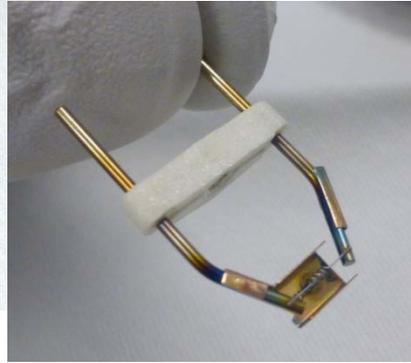
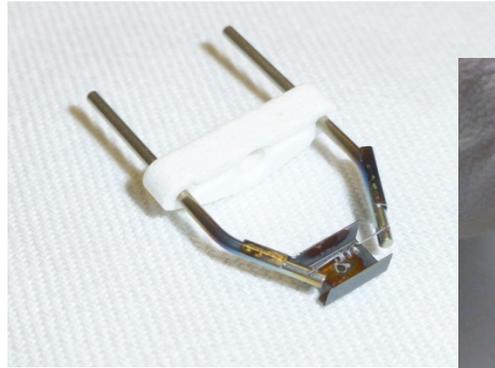
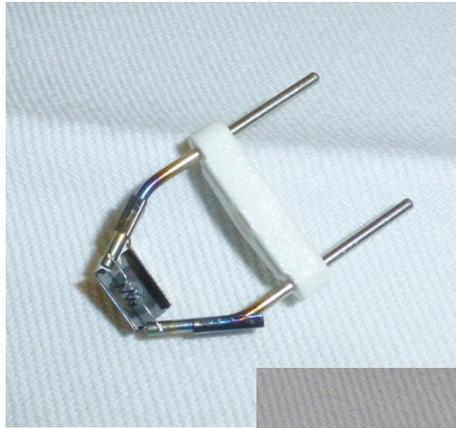
Do not worry too much about any discoloration on the source heater. Wipe all surfaces of it with a hexane dipped lint free cloth. The source heater should measure ~20 ohms and the sensor should measure ~109 ohms at room temperature



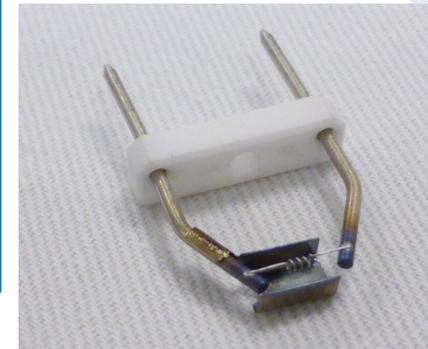
On sources with a drawout plate, squeeze it together during reassembly before you tighten the setscrew to insure good contact between the drawout and the source body.



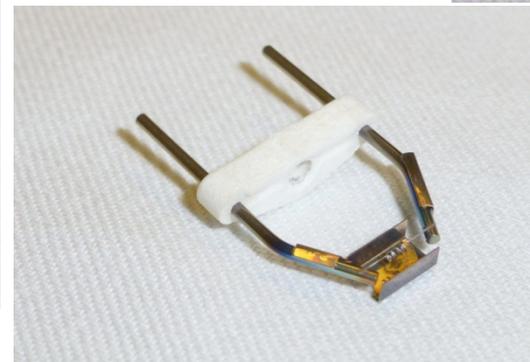
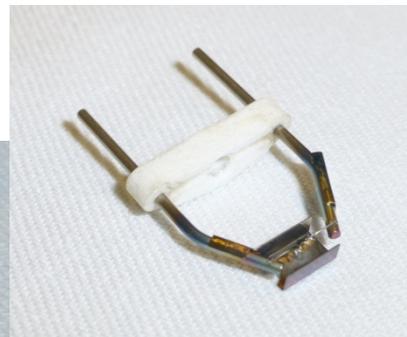
Filaments.



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!



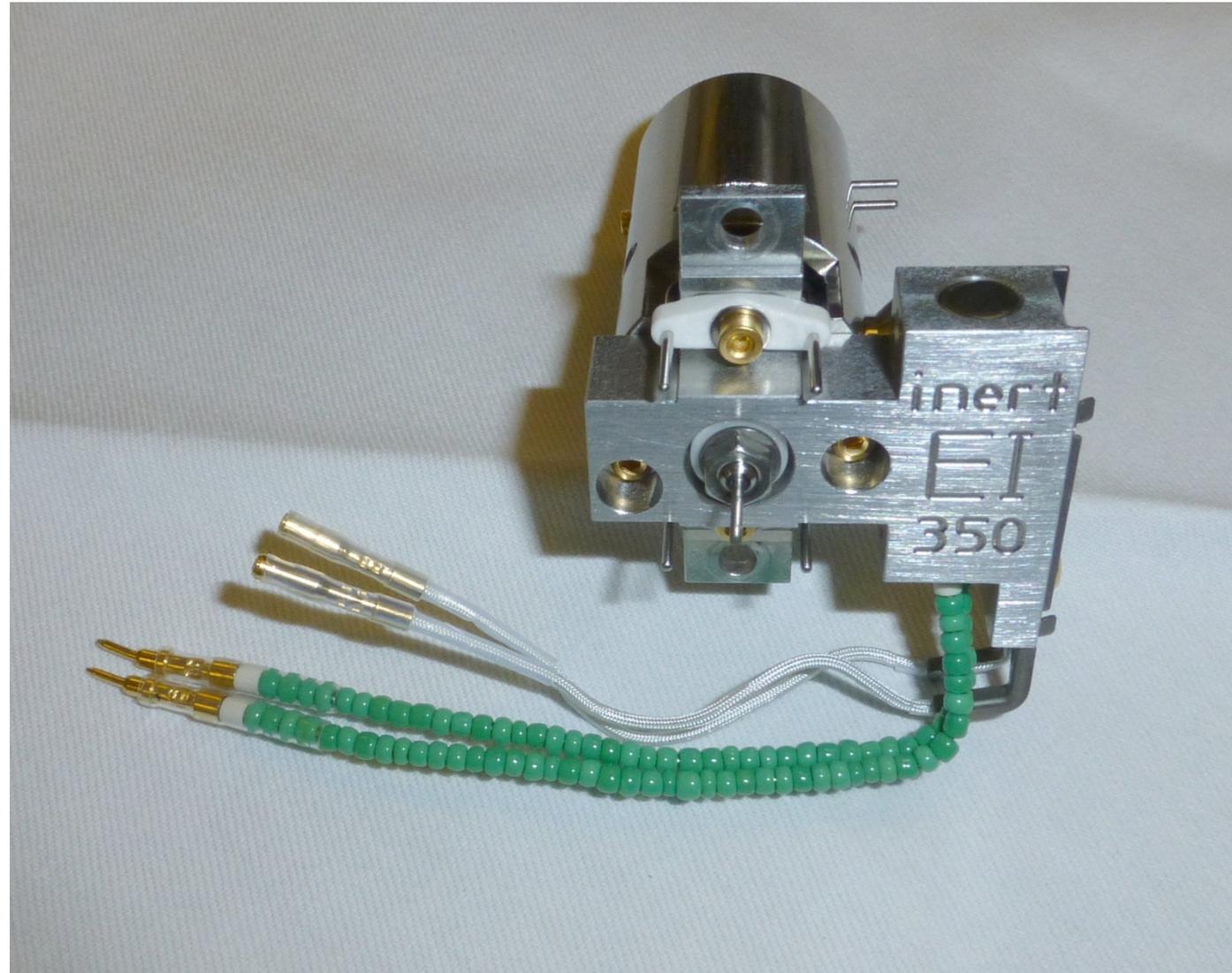
Bent filaments are bad!
Bent or misaligned reflector/targets are bad!
So are cracked insulators.
Inspect the wire – replace if it is not the same thickness everywhere.



Purchase spares!

Ready to reinstall.

A source cleaning is included as part of each preventive maintenance (PM) service. Let your support salesperson know if you'd like it done more than once per year.



Go to the GC front panel:

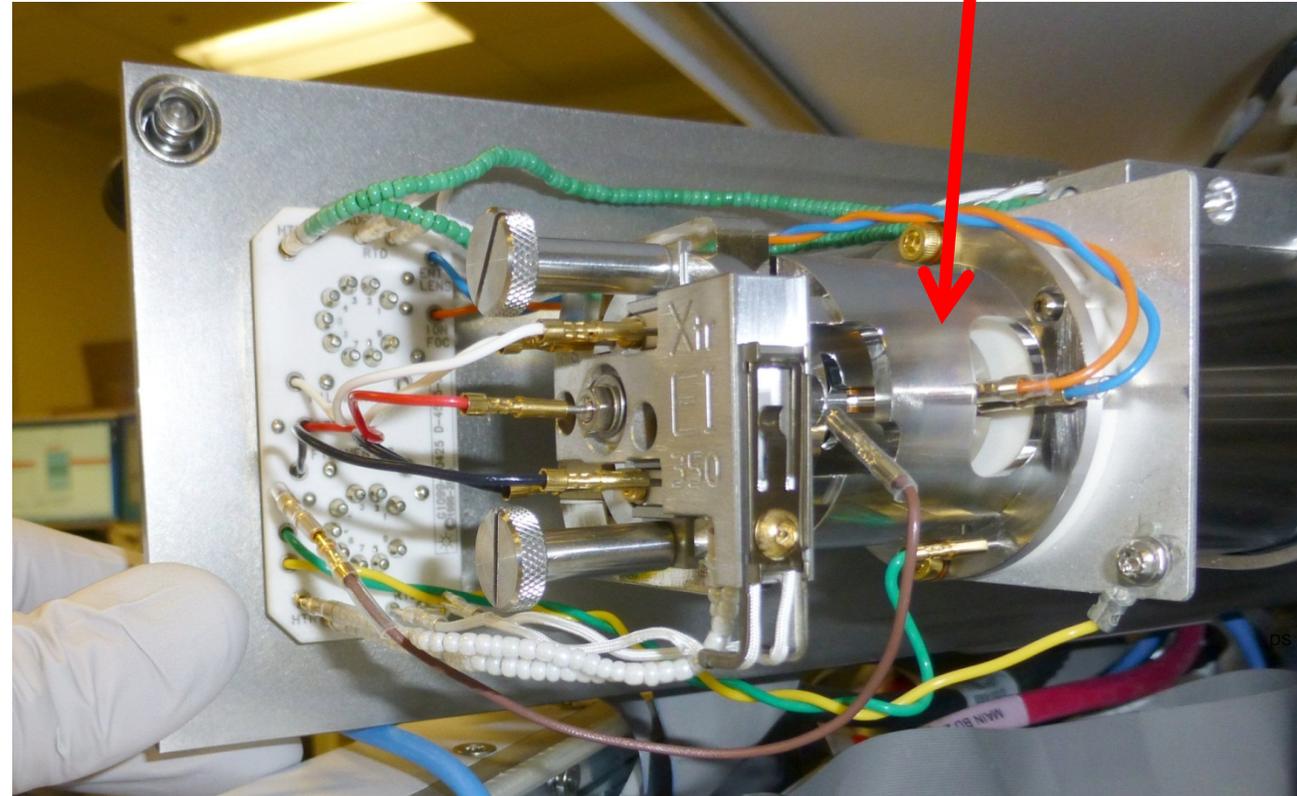
8890 and Intuvo:
Service Mode
Instrument
Usage

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. **You may need to remove and clean all surfaces of the source radiator** (red arrow to the right) – take care not to drop the end of the quad. While it is off, remove the ceramic connector board and wipe clean the side plate. Dampen a lint-free cloth with MeOH and wipe out the inside of the manifold paying particular attention to the right end near the transferline.

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 7×10^{-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°C for two to three hours before proceeding. If you are in a rush, back 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.
- Make sure that **both** filaments will produce a spectrum before running the system.
- Document the maintenance in the instrument logbook.

Cleaning an Agilent GCMS Ion Source

- Clean sooner so you don't have to scrub hard. The source should be easy and fast to clean.
- Clean before the tune degrades significantly.
- Clean thoroughly so you don't have to clean it even more often.
- Clean so that there are no scratches on the critical surfaces.
- Replace the filaments, repeller ceramics, and insulators frequently enough.

Be extra careful and clean everything.

Appendix

You have reached the end of ion source cleaning.

MSD Maintenance schedule

Task	Every week	Every 6 months	Every year	As needed
Tune the MSD				X
Check the foreline pump oil level	X			
Check the calibration vial(s)		X		
Replace the foreline pump oil*		X		
Replace the diffusion pump fluid			X	
Check the dry foreline pump				X
Change the dry foreline pump tip seal			X	
Change the foreline pump exhaust filter				X
Clean the ion source				X
Check the carrier gas trap(s) on the GC and MSD				X
Replace the worn out parts				X
Lubricate sideplate or vent valve O-rings†				X
Replace CI Reagent gas supply				X
Replace GC gas supplies				X

* Every 3 months for CI MSDs using ammonia reagent gas.

† Vacuum seals other than the side plate O-ring and vent valve O-ring do not need to be lubricated. Lubricating other seals can interfere with their correct function.

QQQ Maintenance schedule

Task	Every week	Every 6 months	Every year	As needed
Tune the MSD				X
Check the foreline pump oil level	X			
Check the calibration vial(s)		X		
Replace the foreline pump oil*		X		
Replace the diffusion pump fluid			X	
Check the dry foreline pump				X
Change the dry foreline pump tip seal			X	
Change the foreline pump exhaust filter				X
Clean the ion source				X
Check the carrier gas trap(s) on the GC and MSD				X
Replace the worn out parts				X
Lubricate sideplate or vent valve O-rings†				X
Replace CI Reagent gas supply				X
Replace GC gas supplies				X

* Every 3 months for CI MSDs using ammonia reagent gas.

† Vacuum seals other than the side plate O-ring and vent valve O-ring do not need to be lubricated. Lubricating other seals can interfere with their correct function.

QTOF

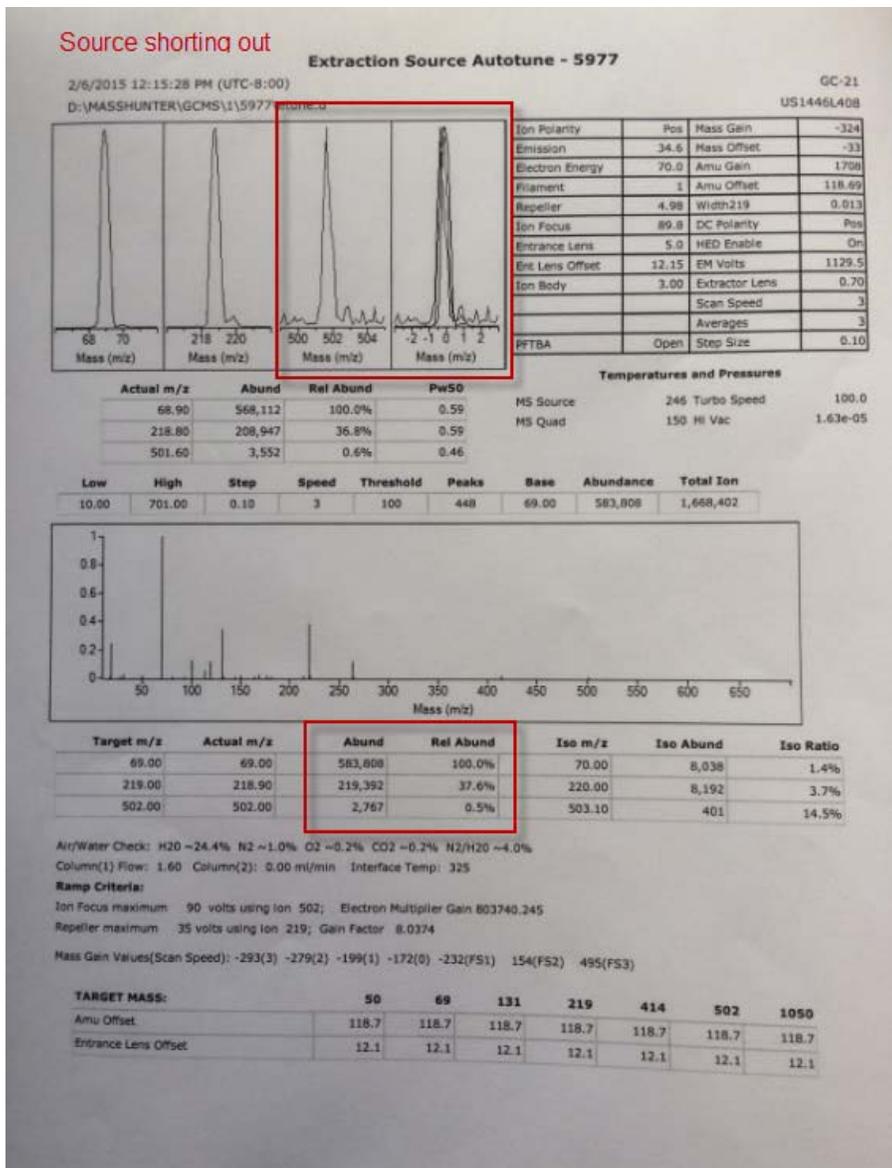
Maintenance schedule

Task	Every week	Every 6 months	Every year	As needed
Tune the MS				X
Check the foreline pump oil level	X			
Check the calibration vial		X		
Replace the foreline pump oil*		X		
Check the foreline pump				X
Clean the ion source				X
Check the carrier gas trap(s) on the GC				X
Replace the worn out parts				X
Replace CI Reagent gas supply				X
Replace GC gas supplies				X
Replace RIS maintenance probe parts			X	

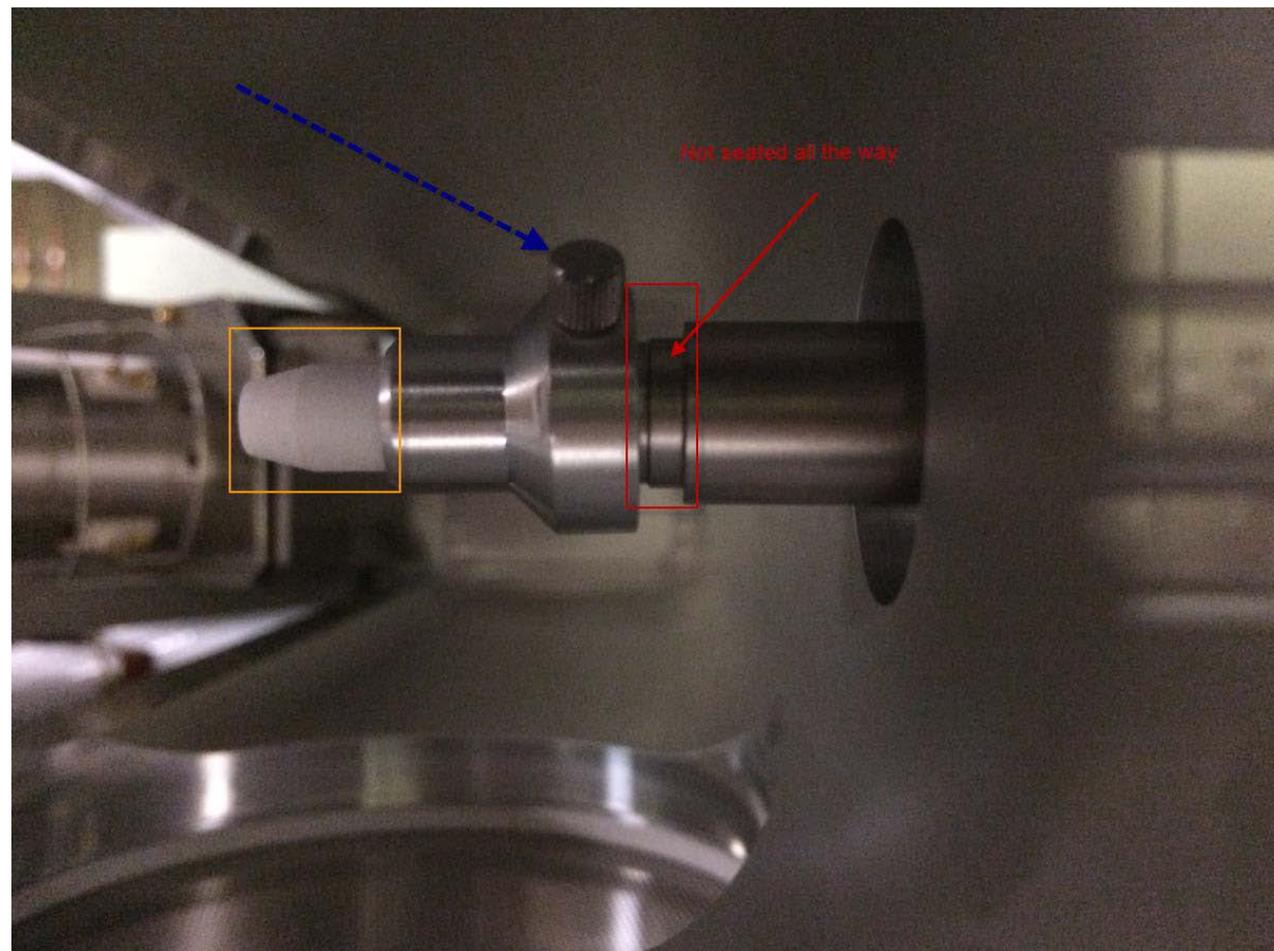
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Every 3 months for CI MSs using ammonia reagent gas.

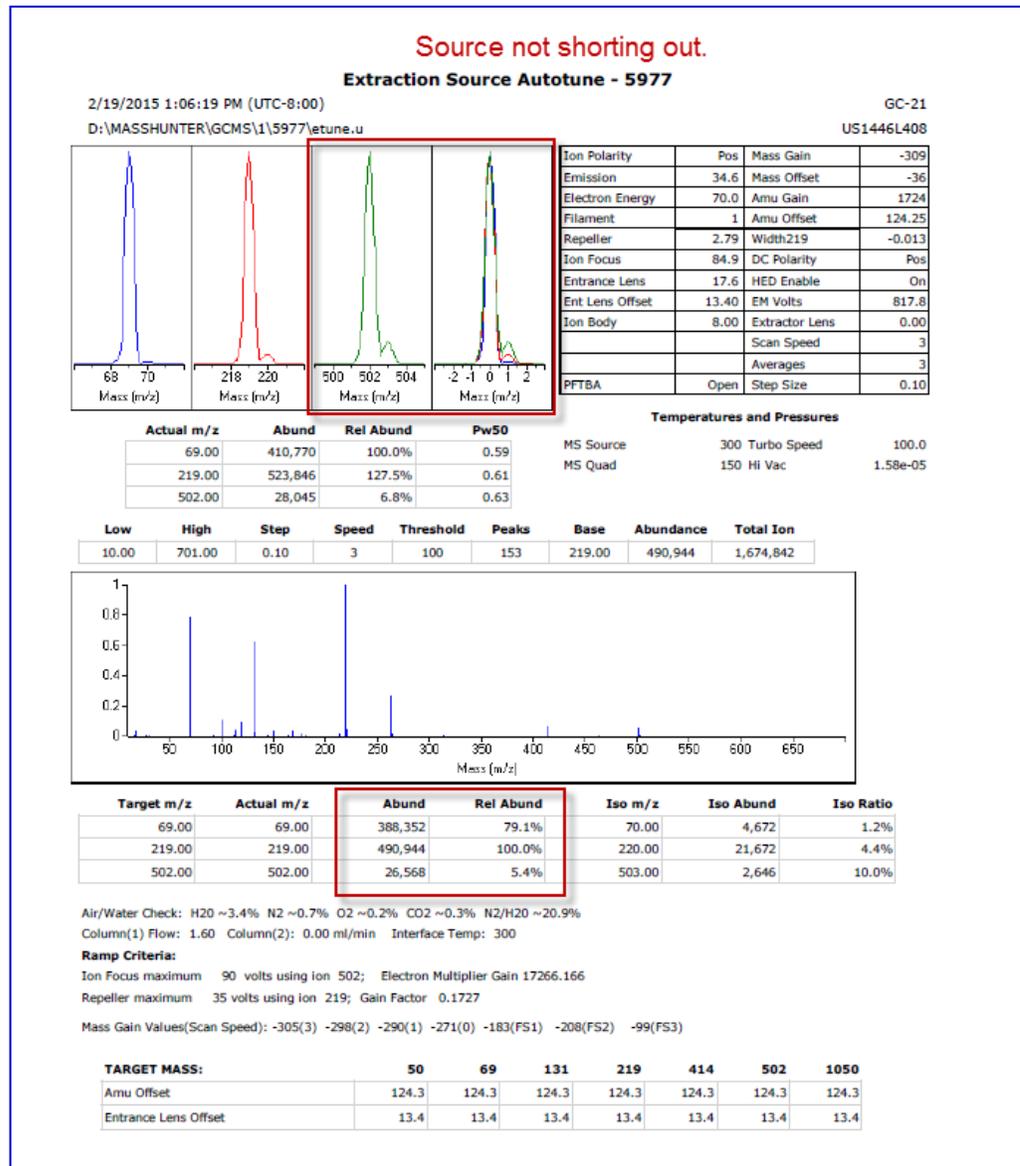
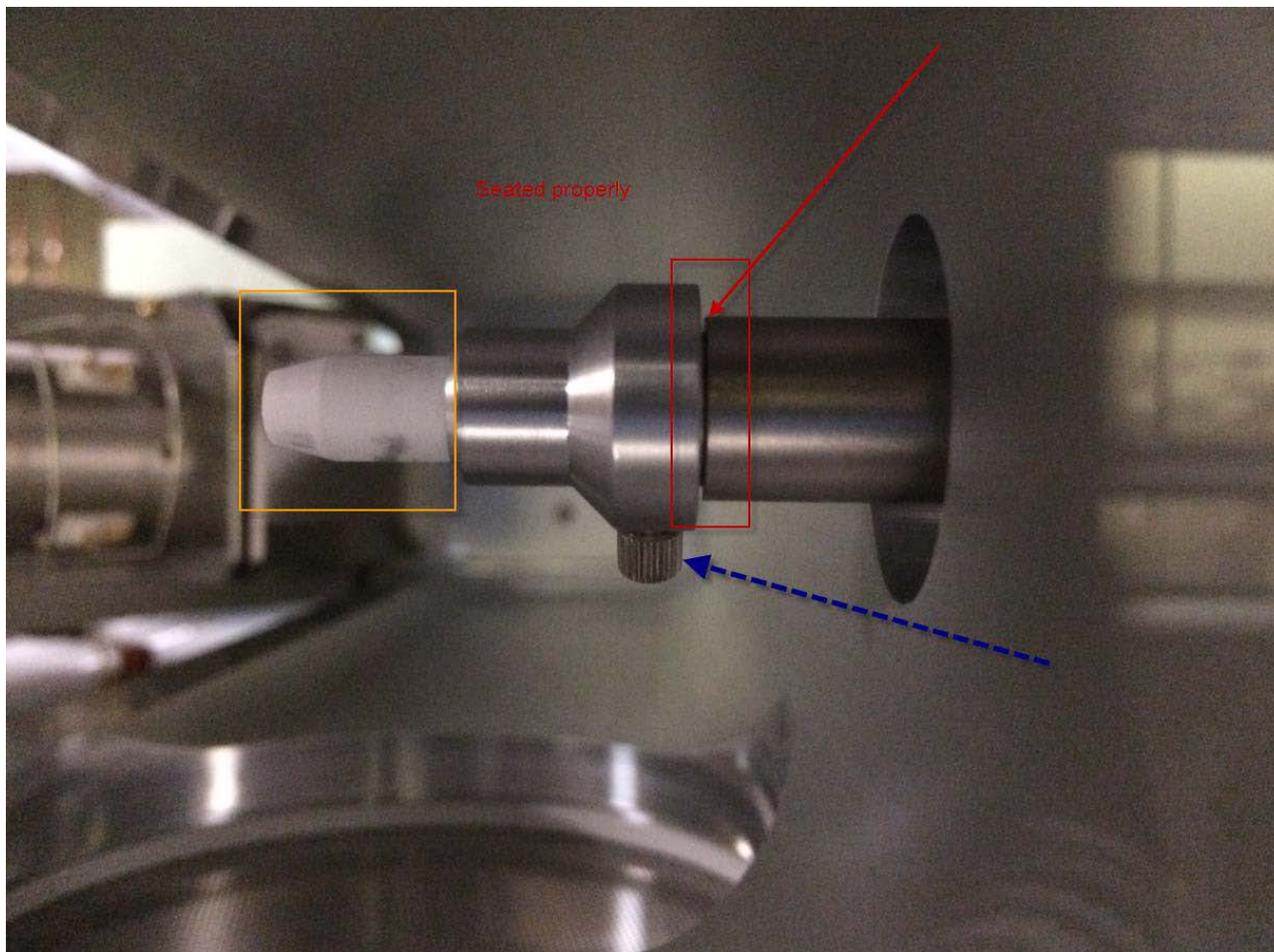
The auto tune looks great but the etune looked bad



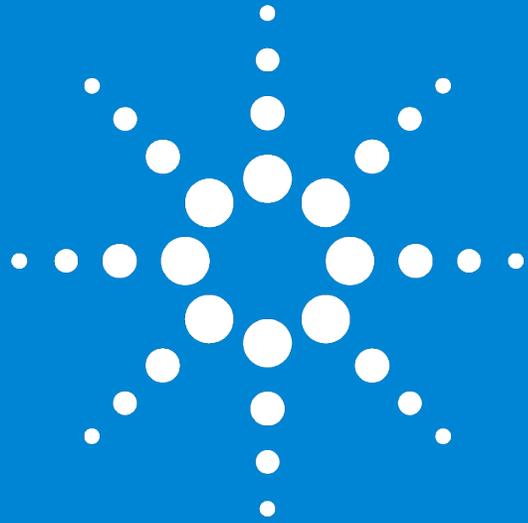
Below is a picture of the transfer line tip installed **incorrectly**. Pay attention to the gap (red box and red arrow) and the set screw in the 2 o'clock position (blue arrow) Also now check the length of the ceramic tip (orange box).



Below of the picture with the tip installed **correctly**. There is almost no gap now between the collar and the transfer line (red box, red arrow), the set screw is in the 7 o'clock position (blue arrow), and the length of the ceramic tip (orange box) figure 3.



Etune after correcting the problem



Agilent

Trusted Answers