

Agilent GCMS High Efficiency Source Cleaning HES

Agilent Customer Community 25Mar2021





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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.
- Increasing tune Electron Multiplier voltage.

Proper cleaning, assembly, and installation are essential to robust and reliable operation. Source cleaning is <u>normal user maintenance</u>. Operators are responsible for this recurrent task.

Source cleaning is not covered under warranty or hardware maintenance contracts except during covered PM services.

Sample residue, gas contaminants, and pump oil vapor 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 the metal surfaces of specific parts of the source removes this coating. Non metal parts must be replaced if dirty.

PROFILE



TARGET MASS: 50 131 219 414 502 1050 Amu Offset 137.7 137.7 137.7 137.7 137.7 137.7 137.7 Entrance Lens Offset 15.6 15.6 15.6 15.6 15.6 15.6 15.6 Any autotune program adjusts multiple parameters to achieve the desired results. <u>Every</u> time a tune is performed, the parameters may be different. This is normal and should not affect the system's response





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 tunes that have 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.



Have these on hand before you begin:

- Consumables such as filament assembly, lens stack insulator, ferrules, etc.
- Four clean beakers large enough to lay the ion body down inside.
- DI water
- <u>Pesticide Residue Analysis Grade</u> Methanol, Acetone, and Hexane.
- Aluminum Oxide powder
- Lint free cloths
- Cotton tipped swabs
- Necessary tools

The Consumables and Supplies Required for Repair option does not provide consumables and supplies for the routine maintenance or normal operation of your instruments.

These are normal usage parts. Have extras on hand all the time!

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.

Cleaning steps:

- In a small beaker, make a thick paste with DI water and Aluminum oxide.
 - [393706201 ALUMINUM OXIDE POWDER 100g]
- Use cotton-tipped swabs, dip them in DI water and then Aluminum oxide to make a paste on the swab or dip them into the pre-made paste in the beaker.
 - [8520-0023 Swab 6.0 in LG; cotton tipped,100/bag]
- Put a small amount of the Aluminum oxide paste on a lint-free cloth laying flat on the bench.
 - [05980-60051 Cloth, lint free 15/PK]
- Use the swabs to scrub all faces of the critical parts. Fronts, edges, backs, in the holes, everywhere.
- Follow all of the rinse and sonication steps when complete.

You are NOT trying to remove metal, only surface contamination. Mechanically, this happens quickly. You are trying to clean <u>all</u> the surfaces of the critical parts.

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

Rinsing and Sonication:

- 1. Clean water rinse until all the alumina residue is gone
- 2. DI water Water removes salts. Sonicate.
- 3. Methanol this step removes the water. Sonicate.
- 4. Acetone Sonicate.
- 5. Hexane this step removes any hydrocarbon residue Sonicate.

Use a separate beaker for each different type of solvent – four beakers. Keep a special 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 the analysis does 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.

Do not heat up the sonication bath.

Make sure to use beakers that will allow the source body to lay down flat so as not to bend the alignment pin.





Next:

- DO NOT heat up the parts to try to dry them! Absolutely do not heat the parts up above 70°C.
- Do <u>NOT</u> bake 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 High Efficiency Source mounted in the analyzer.







Remove T6 screw.



Hold the filament and holder in one hand, invert the rest of the source with the other and carefully lift it up to the source. If you do it this way, the filament assembly stays in the filament block and has less chance of damage.





Lift out filament



The filament sticks out past the target and is easily damaged.

Filament holder – note the flat so that the filament can only go in one way.







Remove the two T6 gold screws

NOTE:

The flat edge of the repeller, repeller insulator and source mount goes towards the interface socket. The heater/sensor goes to the flat of the ion source body.



Note the insulators. Remove the source mount.





Remove the repeller.

Remove heater/sensor. The pins are quite fragile.

This heater/sensor is a very expensive part. Please handle with care.





Remove T6 screw and lens stack retainer.

Remove lens stack and ceramic. This insulator is rarely replaced.







Pull lenses from insulator. This insulator is rarely replaced. It can be broken with effort, though, so have a spare available. (p/n G7002-20074)





HES exploded diagram

12 17 15 0100/ 10



These are the parts that will need to be abrasively cleaned.





Repeller



The repeller face must remain flat! Do <u>not</u> go around in circles. 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 <u>not</u> 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 edge. 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 inside. There will be filament burn. It is <u>not</u> necessary to remove every trace of burn here. It feels good to get this completely perfect, but it is not necessary for ultimate performance.





Repeller



To clean the hole, pull the end of the swab to extend it a bit.



Dip the tip of the swab into the DI water and then into the aluminum oxide then put it in the hole and twist it. Do this from both sides.



Remember this is not sample residue but metal. This is a new source that has never been installed.



Ion Volume.



The ion volume along with the repeller face and the extractor face are the most important areas to clean. Dip a swab into the DI water and then into the aluminum oxide to make a paste on the swab. Use that to polish the entire inside surface.







Ion Volume.

Clean the interface hole.



Clean from the lens side also, including the insulator seat area.



Source Body.

Clean the <u>entire inside and outside</u> of the source body including the slots and edges. This may not be necessary if the analysis is only clean samples, but can become critical over time. If the source has already been cleaned and the tune still shows low 219 and 502, clean this thoroughly.





Extractor Lens – Lens 5.



Clean the face of the extractor, the edge, and the center of the hole. Also, flip it over and clean the entire inside.



Why does it say 5 on the outside edge but 3 inside on the back face?

It's lens 5 with a 3mm hole.



Post Extractor 1 – Lens 4 and Post Extractor 2 – Lens 3



Clean the two Post Extractor Lenses the same way as the Extractor Lens.

Clean the faces, the edges, and the center of the holes. Also, flip them over and clean the entire insides.





Ion Focus – Lens 2.



The ion focus is a flat cylinder.

Clean both faces, the edge, and the edge of the of the hole. Pay particular attention to the hole.

Make sure that the tiny holes on the reverse do not hold residue after the sonication and rinsing steps.



Entrance Lens – Lens 1.

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.







Make sure that the tiny holes on the reverse do not hold residue after the sonication and rinsing steps.







You do not need to abrasively clean... Source mount







Do <u>NOT</u> try to remove the yellow insulators! They are pressed in and should never be removed.



This part should not need any cleaning or rinsing and sonicating at all.

It can be wiped off with a clean solvent dipped lint-free cloth.



You do not need to abrasively clean...Heater/Sensor, Lens Retainer, Filament Block/Finger Grip

Do not worry about any discoloration on the source heater. Do not rinse or sonicate this part.

The source heater should measure ~20 ohms and the sensor should measure ~109 ohms at room temperature



The lens retainer and filament block/finger grip do <u>not</u> require abrasive cleaning. You can add them to the rinse steps.



This is an incredibly expensive part! Do not break it by dropping, mishandling, or squeezing the leads.



Filaments.



Bent filaments are bad!

- A bent or misaligned reflector/target is bad!
- So is a cracked insulator.

Inspect the wires – The filament should be replaced if they are not the same thickness everywhere.



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!



These are normal usage parts. Have extras on hand all the time!



Parts to solvent rinse and sonicate.

Rinse steps:

- 1. Clean water rinse until all alumina residue is gone.
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Ready to reinstall.







You're almost done:

- Your patience will be rewarded. Take your time to ensure that the vacuum is acceptable before you proceed.
- Do not turn on the filament or try to tune at all until the vacuum is nearly typical for your system wait an hour or more! Only then should you even think about confirming 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 the source at 300°C and the quad(s) at 175°C for two to three hours before proceeding.
- 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 you put the instrument back into operation.
- Perform several injections of standard solutions to condition the GC system and source before they start running samples.
- Enter the details into the instrument logbook and maintenance schedule!



Cleaning an Agilent GCMS Ion Source

- Clean <u>sooner</u> so you don't have to scrub hard. The source should be easy and fast to clean.
- Clean <u>before</u> 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.

You have reached the end of High Efficiency Source cleaning.



