



USER MAINTENANCE & DIAGNOSTICS FOR VOLATILE GC/MS SYSTEMS

Whether you service your VOA GC/MS instruments yourself or hire a service company, there are some things all users can do to maximize efficiency of their VOA GC/MS department.

The purpose of this technical tip on user maintenance and diagnostics is to achieve the following 4 objectives:

- ❑ Minimize instrument downtime
- ❑ Reduce the chances for *serious* failure
- ❑ Enable the user to articulate the problem in a clear and concise manner to their service technician or manager
- ❑ Enable you and/or your service technician to diagnose and repair problems more expediently






Consumables

Proper user maintenance begins with having an adequate supply of the right consumables. For most VOA GC/MS departments this includes the following:

- 🔗 VOCARB 3000 Traps- have 2 spares per GC/MS system
- 🔗 Analytical column- have 1 spare per GC/MS system
- 🔗 Filaments- have 2 spares per GC/MS system
- 🔗 Electron Multiplier- have 1 spare per GC/MS system
- 🔗 Injector consumables (liner, disc)- have 2 spares per GC/MS system







Record Keeping

Record Keeping of hardware maintenance issues is also very critical in achieving the 4 stated goals. Examples of information the good GC/MS analyst has at his fingertips include the following:

-  The *exact* date of the last PM (source cleaning, filament change, oil change)
-  The *exact* date that the trap was installed (and the Lot number)
-  The *exact* date that the Electron Multiplier was installed
-  The *exact* date that the column was installed
-  The *exact* date that one of the filaments blew requiring switching to the other

Knowing "optimized run conditions"





Knowing "optimized run conditions" is essential in diagnosing common problems. Examples of information the good GC/MS analyst has at his fingertips include the following:

-  Hard copy printout of Purge and Trap Conditions
-  Purge Flow
-  Desorb Flow
-  GC head pressure (via gauge or GC keypad) at time of desorb
-  Ion Source pressure with the oven at 150°C at column flow of 1.0 mL/min.
-  Rough pump Foreline pressure with the oven at 150°C

Tuning Reports

Tuning Reports give a "history" of Mass Spec conditions which enable us to determine what's wrong. The good GC/MS analyst has copies of all manual tune and AUTOTUNE reports run during the past 24 months.

Key things to check *each day* in Manual Tune:

-  Peak Widths (0.45- 0.55 amu)
-  Mass Axis (integer +/- 0.1 amu)
-  Relative ratios of 69-131-219 (historical ratios that pass BFB)
-  Raw abundance of Ion 69 (historical sensitivity for your curve's dynamic range)

- Once the EM Voltage reaches 2400 in Manual Tune you should plan to either perform a PM or replace the EM.
- Background contamination (ions 18, 28, 32, 40, 44 all below 5% of ion 69)

You should run AUTOTUNE after service to the MSD has been done and save the report in a notebook. Always run AUTOTUNE with the oven and carrier flow the same. I suggest oven at 150°C and the carrier flow to 1.0 mL/minute

Changing The Trap

You MUST know how to properly change the trap as it is done on a periodic basis. Countless problems have occurred at labs over the years because this task is done incorrectly.

Always check the Purge and Split/Desorb Flows immediately BEFORE and AFTER changing the trap. When checking the Desorb Flow the system must be checked in both standby and in Desorb (the values should be within 1 mL/min of each other). Always leak check the trap fittings after changing.

Condition the trap properly prior to use as follows

- Set the oven to the oven max
- Step the concentrator to "Purge" for 10 minutes
- Step the concentrator to "Bake" for 10 minutes (skip over Desorb)
- Step the concentrator to "Purge" for 2 minutes
- Step the concentrator to "Desorb" for 3 minutes
- Step the concentrator to "Bake" for 15 minutes

Leak Checking

Keep in mind something very important that many GC/MS analysts overlook:

✌ There are 2 Kinds of leaks:

The first type is where air gets sucked into the MSD. This can be detected very easily through manual tune where you will see elevated amounts of ions 18,

28, 40 and 44. This is one reason why Manual Reports are useful- so you can see what typical amounts are these background ions are.

The second type is where Helium and analytes are lost from the system. There can best be detected with a Helium leak detector or by monitoring changes in flow

Common areas where leaks can occur:

- ☞ Trap fittings (Helium/Analytes out)
- ☞ Interior Purge and Trap fittings (Helium/Analytes out)
- ☞ column fitting into Mass Spec (Air gets sucked in)
- ☞ column fitting into injector (both)
- ☞ O-ring on MSD (Air gets sucked in)
- ☞ Glassware of Concentrator (Helium/Analytes out)

Active Sites

Detecting Active Sites is critical in maintaining linearity and hitting MDLs.

What is an active site? An active site is any contamination in the system, other than air, which can cause Volatile Organic Compounds to react with or adsorb to, hence reducing the response and/or creating Organic artifacts. Active sites in Volatile systems usually develop in the Purge and Trap or Injection Port.


☞ With active sites, certain compounds will exhibit greater breakdown based on their reactivity. Usually by reviewing "good data" (obtained via Quant reports, TICs and ICAL reports) vs. "bad data" as well as checking run conditions one can determine if active sites exist and where they are.


Things the good analyst can do to prevent active site formation:


- ☆ guard against soapy, foamy samples with every fiber of his being
- ☆ guard against water overflow by checking the concentrator sparge tube before starting a sequence
- ☆ minimize Acid preservative
- ☆ minimize Methanol and water vapor entering the system


If You DO Need Service

Things to check and have ready if you do need service:

 Please be able articulate the problem clearly and analytically. For example, if the sensitivity has dropped, tell the service technician to what extent (e.g. 30%, 50%, etc.). If the retention times have shifted, tell the service technician to what extent (e.g. they are all eluting later by 2 minutes or 5%)


 Check to be sure the Helium hasn't run out and if you have a helium scrubber be sure it isn't shot.

 If you are seeing elevated ion 40 (Argon), it could be a contamination in the Helium so check the EXACT DATE when the Helium tank was last changed.

 Check to be sure the correct Internal Standard solution is loaded- this means having the correct mix at the correct concentration.

Note: if ISTD areas are drifting the technician may want you to add the solution manually and run a batch so be prepared for that.

 Check to see if the column has broken.

 Have current information about the issues discussed here such as tune reports, recent quant reports, TICs and ICAL reports and a list of run conditions you have been using.