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User Diagnostics for GC/MS Systems

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USER DIAGNOSTICS FOR GC/MS SYSTEMS

INTRODUCTION

By "User Diagnostics" I mean those tests, measurements and records which are the first step in resolving instrument problems and which **all GC/MS analysts** should be able to perform as needed. Even today, when I provide technical support, I routinely ask the GC/MS analyst to perform certain User Diagnostics described in this issue.

The purpose of becoming proficient at User Diagnostics as well as all other training on user maintenance and diagnostics, is to achieve the following 4 objectives:

1. Minimize instrument downtime.
2. Reduce the chances for serious failure.
3. Enable the user to pinpoint the problem in a clear and concise manner.
4. Enable repairs to proceed more expediently by the user or service technician.

So, even if you have a hardware service contract or hire GC/MS technicians as needed to do the service, it's still in your best interest to become proficient at User Diagnostics as this will greatly assist in achieving all 4 of the above goals. OK, having said that, let's begin.

THE MASTER PLAN FOR GC/MS ANALYSTS AND MANAGERS

- Maintain an adequate supply of the right consumables.
- Be diligent in your record keeping.
- Know the "optimized" run conditions.
- Run AUTOTUNE as needed and Manual Tune each day.
- Check for leaks and active sites as needed.

Proper User Diagnostics begins with having **an adequate supply of the right consumables**. For the typical GC/MS Dept. this includes the following:

- VOCARB 3000 Traps (or whatever you are using) - have 2 spares per VOA GC/MS system.

  VERY IMPORTANT - keep track of the Lot number of the VOCARB 3000 traps you have. This is because the lot to lot variation in the VOCARB 3000 traps can be great so keeping close tabs on the lot numbers can prove helpful down the road.

  HAVE A KNOWN "GOOD TRAP". One of the best pieces of advice I can give is that you should always have a spare trap (one per instrument) that you keep in a drawer strictly for troubleshooting purposes. When you order traps, if you should receive a really good lot which performs well, set aside one of those traps and mark it as "KNOWN GOOD TRAP". The next time you are having problems and you want to eliminate the trap as a possible problem, you can install your "KNOWN GOOD TRAP". If the problem subsides, then you know the old trap was the problem and you replace it.

  If not, at least you can perform further diagnostics without the uncertainty of the condition of the trap.

- Analytical column - have 1 spare per GC/MS system.
- Filaments - have 2 spares per GC/MS system. Avoid rebuilt filaments as they have been reported to be far more problematic than brand new ones. This is not an area to try to save a few dollars because in doing so you are risking major down time.
- Electron Multiplier - have 1 spare per GC/MS system.
- Injector consumables (liners, disc) - have 2 spares per GC/MS system.
- Fresh silanizing reagents .

Record keeping of hardware maintenance issues is also very critical in achieving the 4 stated goals. Many times analysts will be having a problem and I'll ask them questions about the system and they either don't know or don't even know where to look in their lab for the answers.

Examples of information the good GC/MS analyst has **at his fingertips ready on-demand** include the following:

- The exact date of the last PM (source cleaning, filament change, oil change).

- The exact date that the Oil Diffusion Pump Fluid was added (for those GC/MS systems with Oil Diffusion Pumps).
- The exact date that the turbo pump was installed (for those GC/MS systems with Turbo Pumps).
- The exact date that the trap was installed (and the Lot number).
- The exact date that the Electron Multiplier was installed - keep the paperwork that accompanies the Electron Multiplier which lists the manufacturer, model and serial number.
- The exact date that the Purge and Trap was last refurbished. By refurbished we mean lines and valves replaced.
- The exact date that the Injector Pneumatics were last replaced.
- The exact date that the column was installed. You should also know the specifications of the column (manufacturer, length, internal diameter, phase and film thickness).
- The exact date that one of the filaments blew requiring switching to the other.
- The exact date that any service was done to the instrument and what exactly was done.

Keeping your instrument log accurate and up to date is an essential part of a good GC/MS analysts job.

Knowing “optimized run conditions” is essential in diagnosing common problems. When the system is performing well, i.e. passes tune consistently, sensitivity is good, curves are linear, etc., you should document those conditions that lead to such good performance.

Examples of information the good GC/MS analyst has at his fingertips include the following:

Volatile GC/MS systems

- Hard copy printout of Purge and Trap Conditions (in case of power failure) for all methods.
- Purge Flow.
- Desorb Flow.
- GC head pressure (via gauge or GC keypad) at time of desorb.
- Column flow.
- Ion Source pressure (measure this with the column flow the same as in your analytical run).

For BNA Systems, virtually all of which employ a split/splitless injector you will need to measure the split flow in the Purge ON mode as well as the Purge OFF mode.

1. Set the oven to your starting temperature (typically 40o C).
2. Step the Injection Port to the Purge OFF mode.
3. Measure the split vent flow (typically 20-40 mL/min).
4. Step the Injection Port to the Purge ON mode.
5. Measure the split vent flow again. It should be no more than 1 mL/min different than it was in Step #3.

Tuning Reports give a "history" of Mass Spec conditions which often enable us to determine what's wrong more quickly than we would without such reports. The good GC/MS analyst has copies of all manual tune and AUTOTUNE reports run during the past 5 years (or better yet for the entire life of the system).

Key things to check each day in Manual Tune:

- Peak Widths (0.45-0.55 amu) with 0.50-0.52 being ideal.
- Mass Axis (integer ± 0.1 amu).
- VOA GC/MS: Relative ratios of 69-131-219 (historical ratios that pass BFB)
- BNA GC/MS: Relative ratios of 69-131-219-414 (historical ratios that pass DFTPP).
- 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 clean the source or change the electron multiplier.
- Background contamination (ions 18, 28, 32, 40, 44 all below 5%).

You should run AUTOTUNE and save the report upon any of the following situations:

- Every 30 days if no service has been done.
- If the system has been vented for any reason.
- If any electronics have been changed.
- If you are having mass spec problems.

Changing the Trap

Many times problems occur immediately after changing the trap. Hence, always check the Purge Flow immediately BEFORE and AFTER changing the trap. If you've done it correctly and no leaks are present then the flow before and after should be identical. Always leak check the trap fittings after changing and use fresh ferrules if the old ones look worn out.

Condition the trap properly prior to use as per manufacturers specifications.

Leak checking the system

There are two completely separate kinds of leaks:

1. The kind of leak where air gets sucked in (can detect through manual tune by the presence of ions 28, 32 and 40).

2. The kind of leak where Helium and analytes lost (can detect with leak detector or changes in flow).

Keep in mind that if leak type #2 exists, you will NOT be able to detect this in manual tune. The manual tune only detects leak type #1. Many time I hear GC/MS analysts rule out a leak because they don't see ions 28, 32 or 40. This is a major mistake to do this!!!!

Common areas where leaks can occur in Volatile Systems:

- 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).

Common areas where leaks can occur in Semivolatile Systems:

- Bottom on liner that seals against inlet disc (Helium/Analytes out).
- Column fitting into Mass Spec (Air gets sucked in).
- Column fitting into injector (both).
- O-ring on liner (Helium/Analytes out).
- Retainer nut on inlet (Helium/Analytes out).

Detecting Active Sites

What is an active site? An active site is any contamination in the system, other than air, which can cause Volatile or Semivolatile 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. Active sites in Semivolatile systems usually develop in the Injection Port. With active sites, certain compounds will exhibit greater breakdown based on their reactivity. Usually by reviewing good data vs. bad data as well as checking run conditions and leak checking, we can determine if active sites exist and where they are.

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

Volatiles

- Guard against soapy, foamy samples with every fiber of his being.
- Minimize Acid preservative (just add what the method stipulates- nothing more).
- Minimize Methanol and water vapor entering the system.

Semivolatiles

- Don't inject highly viscous extracts. Dilute them and run them first; you can always inject the un-diluted extract later.
- Use silanized fused silica wool and liners in the injection port.
- Avoid injecting extracts from samples which contain high levels of organometallic substances.

For Volatiles, if a water spill-over or soap foamover occurs, there is a good chance that water may be still be in the lines. Dry the Purge and Trap system out by conditioning the system as you would if a new trap were installed and then run 2 blanks and then a standard. If responses still look poor you should change and condition the trap. Run another 2 blanks and then a standard. If performance is still unsatisfactory then a refurb of the purge and trap may be indicated.

Questions or comments on this or any issue of OPTIMIZE may be emailed to the author at MFerry@SPEX.com

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