Agilent Technologies

Innovating the HP Way

Mass Spec will not Autotune

Applies to 5970A/B MSD

What could be my problem?

There could be several things that would cause your Mass Spec not to Autotune. The most common conditions that typically cause Autotune problems and are easily corrected are listed in scenarios below. By following these suggestions most Autotune problems can be corrected, providing that no hardware-electronics problems exist.

- **Vaccuum**
- **Perform and evaluate air and water check**
- **Calibration valve and calibrant**
- **Perform and evaluate Autotune report**

What should I observe or do?

Vacuum

One thing that should be checked constantly when operating the Mass Spec is the vacuum, and should be the first thing you check when having or suspecting problems.

When everything is working as designed, the 5970A/B the vacuum should be in the range of $5X10^{-5}$ range or lower, if the mass spec is being used as a capillary direct system, (column inserted directly into mass spec interface). If the Mass Spec is being used with a jet separator, the vacuum will typically be 6 to 8 $X10^{-6}$ range, depending on column id, length and flow rate.

What to do if vacuum is bad:

1) **Verify That The Pumping System Is Operating**

Check that the oil level is correct in the sight glass of the rough pump and confirm that the rough pump and turbo pump are running. The 5970A is equipped with a turbo speed indicator gauge, which can show visually the approximate rotational speed of the turbo pump. The 5970B is not equipped with this gauge, but is designed with a light that will illuminate in the heater/electronics switch to indicate that the turbo is up to speed. If the rough pump is not running, verify that it is plugged into the rear of the Mass Spec or into a wall outlet. If the Mass Spec power switch is on, turn it off, wait a minute then turn it back on. If the rough pump restarts, observe to see if the system will complete the pump down sequence. If the system completes the pump down sequence and the vacuum is okay, the system may have shut down because of some voltage glitch. If the rough pump shuts down after 5 to 10 minutes, it could indicate that the turbo pump is not coming online (getting up to speed), a bad Turbo pump or Turbo controller can cause this symptom.

If you are not familiar or comfortable with troubleshooting this problem, on-site service is recommended.

2) **Pumping System Is Okay But Vacuum Is Still Bad**

In this case the best way to troubleshoot the problem is to split the system in half. You do this by removing the column from the injection port and capping it off by pressing that end into a septum. If your column is 0.25mm id or smaller and at least 10 meters long, you should be able to remove the column without venting the system. If the column is 0.32mm id or larger it is recommended that the column be at least 30 meters long to prevent a venting accident.

CAUTION: If in doubt vent the MASS SPEC.

This procedure isolates the Mass Spec from the GC. With the column capped off the Mass Spec should pump down quickly. If the vacuum problem still exists, the next step requires you to use the vent procedure and vent the Mass Spec. After the Mass Spec has been vented, cap off the Mass Spec interface with a blank ferrule, (*i.e.,* a ferrule without the hole), then pump the system down.

If the vacuum problem still exists, there is a problem at the Mass Spec, and you may at this time elect to check all other fittings in the vacuum manifold that can leak to the outside of the manifold.

If everything checks good and vacuum problems still exist, Agilent Technical Support or on-site service is recommended.

3) **Perform Air and Water Check**

If your vacuum is looking okay, at this point we can proceed to performing an air and water check. This check can be done in several ways, depending on what software product and revision you running, it could be possible to run the air and water check by selecting it from a menu in the software. Another way is to simply perform a spectrum scan from manual tune, then evaluate the spectrum scan to determine what percentage the air (ion 28 and 32) and water (ion 18) are, relative to ion 69. In performing the spectrum scan MAKE SURE THE CALIBRATION VALVE IS OPEN.

What to look for in the Air & Water Check:

When evaluating the spectrum scan, you should not see any ion below (less than) mass 69, that is higher than 10 percent of the abundance of mass 69. That means if there is ion 18 (water), ion 28 (nitrogen), ion 32 (oxygen) or any other background ion, they should be below 10 percent of the mass 69 abundance. You may also want to check the overall background of the spectrum against backgrounds that you have had in the past to verify if the current background is significantly different (usually higher) than before. High background can be a sign of a source of contamination.

Masses 18, 28 and 32 being higher than normal could indicate a leak, but only if the abundances are in the correct ratio. In other words if you have a leak, the resultant spectrum will be representative of the atmosphere's make-up. In the event of a leak you should have ions 28 (nitrogen), and 32 (oxygen). The ratio of ion 28 to ion 32 should be approximately 4:1, or four times more nitrogen than oxygen, you will also have mass 18 (water) in some amount representative of the

humidity at the time. These are the signs of a true leak. If you have a single abnormally high ion, for instance ion 28 is high or ion 18 being high individually, this is not a leak but more likely a sign of contamination, either from the gas source (tank) or chemical filter traps that have become saturated.

If Autotune parameters and peak ratios seem normal, but abundances appear low across the spectrum with a high multiplier voltage, (2600 to 3000 volts), that's usually a strong sign that the multiplier is going bad and in need of replacement. If you have a multiplier that was taken out before, you may elect to try it just to verify the problem, (assuming that the old multiplier is not in bad condition).

4) **If A Leak Is Suspected**

Split the system in half by removing the column from the injection port of the GC and capping off (plugging) the column, by pressing the injection port end of the column into a septum. If the leak was in the injection port then the indications of the leak should go away while the column is capped off.

NOTE: If your column is at least 30 meters long or longer and .25mm id or smaller, you should be able to remove the column without venting the system. However don't take your time plugging off the column. If you're not sure, VENT.

If the problem still exists then the Mass spec should be vented, (using the vent procedure). Remove the column and cap off the Mass Spec interface by using a blank (no-hole) ferrule.

After capping the Mass Spec, pump the system down and verify if the problem is corrected. If the problem is corrected then you should vent the Mass Spec and reinstall column using a new ferrule at the Mass Spec Interface.

CAUTION: Never use an all graphite ferrule at the mass spec interface, this ferrule should be 85 percent vespel and 15 percent graphite.

It's recommended to pump the system down in stages to continuously monitor if the problem is corrected. To pump system down in stages, simply reinstall column into Mass Spec interface, using a new ferrule, and cap of the injection port end of the column with a septum as described above. Pump the system down long enough to

determine that the system is okay. Then remove the septum from the end of the column and install the column into the GC injection port and re-verify that the system is still okay. Use the guidelines given above to determine if you need to vent the Mass Spec before removing the septum from the column.

If you are still experiencing a problem, Agilent technical support or on-site service is recommended.

5) **Calibration valve and calibrant (PFTBA)**

Make sure that the calibration valve is operational, if a guage controller is installed, you should notice a jump in pressure when the mechanical valve is opened. Also insure that the calibration vial is filled with the PFTBA calibrant.

6) Perform and Evaluate Autotune

If the system will complete the Autoune without generating a system error, it could contain information that will help you isolate your problem.

NOTE: On system's running newer software, "Windows 95 or Windows NT based Chemstation platforms", the names and functions of some tunes have changed. For instance Autotune on these systems, as we are referring to it in this document is actually called Standard Spectrum Tune, while the Autotune in this newer software is actually a MAXIMUM SENSITIVITY TUNE.

IT IS IMPORTANT THAT IF YOU'RE ON A SOFTWARE PLATFORM THAT INCORPORATES BOTH AUTOTUNE AND STANDARD SPECTRUM TUNE AS A TUNE OF CHOICE, THAT YOU RUN THE STANDARD SPECTRUM TUNE WHEN EVER THIS DOCUMENT REFERS TO AUTOTUNE FOR THE RESULTS TO BE CORRELATED CORRECTLY.

What to look for

There is a lot of info on the Autotune report, which can make it confusing, but we can make it simple by narrowing it down to what's important for us to make a determination on how the system is performing. First look at the 3 principal peaks of the Autotune, 69, 219 and 502. Observe their peak shapes and their peak width.

Peak shape should be smooth and symmetrical, and peak widths are typically in the 0.5 amu range. Next look at the absolute abundance of ion 69, it should be in the range of approximately 150,000 to 450,000 counts for the absolute value of ion 69. Absolute abundance could be different depending on exact hardware and software/ software revisions in use.

Relative to ion 69, ion 219 should be 35 percent or greater, and 502 should be 2 percent or greater. Isotope ratios will vary, but typically they are in the range of 1, 4, and 10 percent for ions 69, 219 and 502 respectively. The ion focus parameter should be in the range of 0 to approximately 10 volts for systems that have a good vacuum, low background and a good clean source. Multiplier or EM voltage should be reasonable, depending on EM age, between 1400 and 2600 volts, (the maximum is 3000 volts), as the multiplier voltage surpasses 2600, it's anybody's guess as to how long it will last.

There are several indications that you could expect as the source gets dirty. Typically the 502 ion will start to decrease before any other ion, and the 502 peak becomes jagged in appearance. In many cases the ion focus parameter will increase drastically, to between 20 to 40 volts or more.

If Autotune parameters and peak ratios seem normal, but abundances appear low across the spectrum with a high multiplier voltage, (2600 to 3000 volts), that’s usually a good sign that the multiplier is going bad and in need of replacement.

If you have a multiplier that was taken out before, you may elect to try it just to verify the problem (assuming that the old multiplier is not in bad condition). Since multiplier voltage does not increase to compensate for low 219 or 502 ion abundance, the EM or multiplier voltage may or may not increase unless the 69 ion abundance has dropped below its target abundance mentioned above. If all or some combination of the above symptoms appear, typically it's an indication that the source is in need of good cleaning.

If tune problems still exist after cleaning the source, there is no other error message being displayed, and all other suggestions have been investigated, Agilent technical support or on-site service is recommended.

7) **Remember, If An Error Message Is Displayed …**

If an error message is displayed during the tune process, Search the Technical Support Assistant for that particular error message.