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Innovating the HP Way

Mass Spec will not Autotune Applies to 5989A/B MS

What could be the problem?

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

- Vacuum
- Perform And Evaluate Air And Water Check
- Perform And Evaluate Autotune Report
- Calibration Valve And Calibrant

Vacuum

One thing that should be checked constantly when operating the mass spec is the vacuum, and vacuum should be the first thing you check when having or suspecting problems. When everything is working as designed, the 5989A/B vacuum should be in the range of 5×10^{-6} range in the source or front manifold, and slightly better in the rear, or quad section of the manifold. It's important to know the vacuum in both the front and

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the rear manifold. As stated above the vacuum in the rear manifold should be slightly better than the front.

What to do if the vacuum is bad

1. Verify that the pumping system is operating

Check that the oil level is correct in the sight glass of the foreline and auxiliary pumps and confirm that the pumps are running and that the diffusion pump is on and hot, (but DO NOT TOUCH the diffusion pump, because it will burn). If the foreline or auxiliary pump is not running, verify that pump circuit breaker is on, (reset if in doubt by putting the breaker in the off position then back to the on position). If the foreline and auxiliary 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 pump or pumps do not start it may be seized or there could be a electronics problem in the Mass Spec that is preventing power from being applied to the pump or pumps. If this is so, 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 cap it off by pressing that end into a septum. If your column is 0.25mm id or smaller and at least 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 is at least 30 meters long to prevent a venting accident.

CAUTION: If in doubt vent the MASS SPEC.

This procedure separates 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, (that is, a ferrule without the hole), then pump the system down.

If the problem still exists, then 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 the mass spec won't pump down at all (diffusion pumps will not turn on)

3. Check for foreline pressure, if it's excessive (greater than 300 milli-torr) then it's possible that O-RINGS at bottom of DIFFUSION PUMPS (four in all) have become hard and brittle, preventing them from sealing. This is especially true if the pump down problem occurred after shutting down the system for normal maintenance. The O-RINGS in question are located at the bottom of the diffusion pumps beneath caps (2 per pump) on the drain/fill spouts. The O-RINGS over time will become hard and brittle, because of the harsh environment they have to operate in, (mainly the high temperatures). When the system is cooled down everything contracts, causing the O-RINGS to break their seal with the cap and the drain/fill spout. When you attempt pump down again the O-rings are not pliable enough to seal and the foreline pressure remains too high to allow the diffusion pumps to turn on. When replacing these O-RINGS, make sure that all four are replaced (2 per pump).

NOTE: It's especially IMPORTANT to CHANGE these O-RINGS if the MASS-SPEC has been running for a long time and has been shut down for maintenance. These O-RINGS operate in an harsh environment and will become brittle and non-pliable over time.

4. Verify MANIFOLD O-RING and MANIFOLD glass cover is in good condition and that the O-ring is seated properly in its groove.

NOTE: The manifold O-RING should be inspected for cuts, flat spots and general good condition. The O-ring groove should be checked for cleanliness, there should be no signs of dirt or debris in the o-ring groove.

The O-ring, O-ring groove and the sealing surface of the glass manifold cover can all be cleaned with a length free cloth, dampened with isopropyl alcohol or methanol.

5. Verify that the glass manifold cover is warp free by placing on a clean level surface.

If vacuum is good but Autotune problem persists

6. Perform air and water check

If the vacuum is looking okay, we can proceed to perform an Air and Water Check. This check can be done in several ways, depending on what software product and revision you are 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

When evaluating the spectrum scan, you should not see any ion below (less than) ion 69, that is above 10 percent of scale, relative to ion 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 69 ion. You may also want to check the overall background of the baseline against backgrounds that you have had in the past. If the present background is significantly different (higher) than what has been normal for this particular Mass Spec, as this could be an indication of carrier gas, column, or ion source contamination.

Ions 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 true 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 18 (water) in some amount representative of the humidity at the time. These are the signs of a leak. If you just have an abnormally high ion, meaning ion 28 is high or ion 18 being high, 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.

7. If a leak is suspected

Troubleshoot the leak by:

Split 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 or the GC side of the system 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 .025mm 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 not sure about column length and diameter, it may be safer to vent completely using the vent procedure in the hardware manual.

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

CAUTION: DO NOT OPEN THE Mass Spec until the source and quad temperatures are below 60 degrees C. (to prevent accidental oxidation).

After capping the Mass Spec, pump the system down and verify if the leak has been 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 like described above. Pump the system down long enough to determine that the system is okay, then remove the septum and install it into the GC injection port and re-verify that the system is okay.

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

8. Calibration valve and calibrant (PFTBA) -

Make sure that the calibration valve is operational and that the calibration vial is filled with the calibrant, PFTBA. An indication that the valve is working properly is that, when the valve is turned on (at the beginning of a tune or scan), there should be a noticeable jump in pressure when monitoring manifold pressure.

9. Perform and evaluate Autotune (standard spectra tune) -

If the system will complete the Autotune without generating a system error, it could contain information that will help you isolate your problem by evaluating the information contained in the tune. Even if the Mass Spec can not complete the tune, the resultant error message that is typically generated will often be an excellent guide to determining the actual source of the problem.

If an error message is displayed during tune operations, stop and search the Agilent Technical Support Assistant (on the web) for that particular error.

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 widths. 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 relative to the ion 69 absolute abundance. Isotope ratios will vary but typically they are in the range of 1, 4, and 10 respectively for ions 69, 219 and 502. Repeller voltage should be relative low, between approximately 4 to 8 volts.

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.

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.

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 being replaced. 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, and there is no other error message being displayed and all other suggestions have been investigated, Agilent Technologies technical support or on-site service is recommended!

10. **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.