



Agilent Technologies

Innovating the HP Way

Mass Spec will not Autotune

Applies to 5971/72A MSD

What could be the problem?

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

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

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 5971A/72A vacuum should be in the range of 5×10^{-5} torr 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 $\times 10^{-6}$ range, depending on column id and column flow rate.

This document is believed to be accurate and up-to-date. However, Agilent Technologies, Inc. cannot assume responsibility for the use of this material. The information contained herein is intended for use by informed individuals who can and must determine its fitness for their purpose.

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, confirm that the rough pump is running and that the diffusion pump is on and hot. The status of the diffusion pump can be determined by going into **vacuum diagnostics** and checking the **vacuum status**.

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 is plugged into the Mass Spec). 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 does not start it may be seized or there could be a electronics problem in the Mass Spec that prevents power from being applied to the Rough pump (if the pump is plugged into the rear of the Mass Spec).

If the Rough pump is plugged into the Mass Spec power outlet (on the rear of the MS), and if there is an adapter available that allows the power cord to be plugged directly into a wall outlet, this may be done to verify if the Rough pump will power up.

NOTE: Make sure the wall outlet is the proper line voltage for the pump or the pump could be damaged.

If the power cord is already plugged into a wall outlet, verify that power still exists at this outlet, and that the circuit breaker for that outlet has not been tripped. If power to the Rough pump checks okay but the Pump does not run, then replacement of the pump or 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 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 is at least 30 meters long to prevent a venting accident.

NOTE: If in doubt about column size and length, go through the complete MASS SPEC vent procedure as outlined in the hardware manual.

This procedure (capping off the column) isolates the Mass Spec from the GC. With the column capped off the Mass Spec should pump down quickly. If a 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 (no-hole) ferrule, then pump the system down. If the problem persists, 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, such as, gauge tube fittings, calibration valve fittings and Manifold seals, etc.

If everything checks okay, and the problem can not be eliminated, Agilent technical support or on-site service is recommended.

3) Perform an Air And Water Check

If the vacuum is looking okay at this point, we can proceed to perform an air and water check. This check can be done in several ways. Depending on which software product and revision is in use, it may be possible to run the air and water check by selecting it from the software menu, which will provide you with a report at the completion of the check. Or, you can simply perform a spectrum scan from manual tune, then evaluate the spectrum scan to determine what percentage air, water and any other contaminants are, relative to ion 69. In performing the spectrum scan, make sure the calibration valve opens, (you should be able to hear an audible click).

What to look for

When evaluating the spectrum scan, you should not see any ion below (less than) mass 69, that is above 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 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, 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 ions are present in the correct ratio. In other words if you have a real 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 a single 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.

4) 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 MS temperature is below 60 degrees C. (this prevents accidental oxidation of the Source).

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.

NOTE: It may be necessary to push down slightly on top of the Analyzer to initially get the Manifold to seal.

5) Calibration valve and calibrant (PFTBA)

Make sure that the calibration valve is operational, (many times you will hear a click when it's opened), and that the calibration vial is filled with the PFTBA calibrant. As another indication that the valve is working properly, when the valve is turned on (at the beginning of a tune or scan) you should notice a jump in pressure when monitoring foreline or manifold pressure.

6) Perform and evaluate Autotune

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 Technical Support Assistant for that particular error.

NOTE: For systems running on 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 are on a software platform that incorporates both Autotune and standard spectrum tune, that you run the Standard Spectrum tune whenever 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 simplify it by focusing on what's important for us determine how the system is performing.

- First, look at the 3 principal peaks of the Autotune, 69, 219 and 502. Observe their peak shapes and peak width. Peak shape should be smooth and symmetrical and peak widths are typically in the 0.6 amu range.
- Next, look at the absolute abundance for ion 69. It should be in the range of approximately 1.5 million to 2.5 million counts for older systems and software. For newer systems and software, the range is approximately 150,000 to 300,000 counts for the absolute abundance of ion 69.
- 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 will be in the range of 1, 4, and 10 per cent respectively, for ions 69, 219 and 502.
- Check the entrance lens offset parameter; it should be in the range of 4.0 to 4.5 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 voltage is 3000 volts.

There are several indicators that may point to a dirty source:

- The abundance of mass 502 will typically start to **decrease** before any other ion, and the 502 peak shape may become irregular or jagged.
- The entrance lens offset parameter may change, either by an increasing or decreasing in voltage away from the 4.0 to 4.5 nominal voltage.

- 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 a good cleaning.

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

7) Remember, if an error message is displayed -

Search the Technical Support Assistant for that particular error message.