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## Diagnosing and Resolving Mass Spec Problems

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# DIAGNOSING AND RESOLVING MASS SPEC PROBLEMS

**NOTE:** It is not my intent to try to turn the readers into mass spec service technicians. I provide this information to help educate the readers. I realize that it's a full time job just to run a GC/MS-what with tuning, calibrating, running samples, reviewing data, etc. Some of you may even run multiple instruments so I realize you don't have the time nor inclination to start fixing the systems yourself once the problem becomes complicated. Many of you have service contract agreements or hire service technicians as needed. The information provided here is intended to allow you to solve relatively simple problems yourself and, when larger problems arise, be able to communicate with your service company in a more intelligent and articulate manner. For those of you without a background in electronics it would be impractical for you to start swapping electronic components. It is helpful, however, for you to do all the service short of swapping electronic components and to be able to relay the nature of the problem articulately to your service company. Also, my experience is limited to Agilent 5970-5973 MSDs so that is what I will mention, although I am sure most of this information applies to the newer units as well.

## Mass Spectrometer Problem #1

Error Message: "No Emission Current"

### What can cause this (in order of likelihood):

1. Blown filament
2. Weak filament
3. Shorted filament
4. Shorted out source
5. Electronics problem

### Discussion of problem and what to do:

The first thing to do is to switch to the other filament. This is why it's important to keep track of when filaments are installed and when filaments are switched. Once both filaments blow, the system MUST be vented and two new filaments must be installed. Unless you keep track, if you switch to the other filament and get the same error message, you won't know whether it's because both filaments are blown or some other reason. This is especially important for those analysts who run more than one instrument - it becomes difficult to remember which system has one blown filament and which doesn't. The solution is to record the installation and switching from a blown filament in a log book so you'll be sure.

If you switch to the other filament and the system is fine you know the problem was just a blown filament. If you switch to the other filament and the system is still not scanning then you should vent the system. Examine both filaments. Are they broken? If so, replace them (you might as well clean the ion source and change the rough pump oil as well while the system is down). If not, a different problem may exist. Sometimes, a weak or sagging filament (especially on H-P 5970 and 5971 model MSDs) can give you no emission current - even if the filament is not completely severed. It is best to put in two brand new filaments. Hence, for each GC/MS system you have, have two brand new filaments on hand at all times. It is a huge error not to have them on hand at all times!

In addition to a blown or weak/sagging filament, you'll get the no emission current error if the filament is not making proper contact on the filament block, thereby shorting it out. If this is the case, it will get resolved when you install the two fresh filaments. Be sure that the filaments are properly aligned on the filament block (this is especially true for H-P 5970 model MSDs). Also, be sure that the wires contacting the filaments are making good connection (this is especially true for H-P 5971/2 and 5973 model MSDs).

**IMPORTANT NOTE:** I strongly suggest that you always use NEW filaments, NOT rebuilt ones as new filaments are more reliable.

If the no emission current error is not caused by a filament problem (broken, weak, sagging, or shorted) then perhaps the source has been shorted out. For 5971/72 systems, if the source is contacting the transfer line it will short out and all kinds of error messages can occur. If you check the source and it seems fine, then an electronics problem probably exists. Generally, the electronics component that drives the filament circuitry would be the first place to look. The likely components, if it is an electronics problem, are as follows:

- 5970 MSD: RFPA Board, Motherboard
- 5971 MSD: Top Board, Power Supply Board, Power Distribution Board
- 5972 MSD: Top Board, Power Supply Board, Power Distribution Board
- 5973 MSD: Side Board, Main Board

## Mass Spectrometer Problem #2

An ion at every mass (grass)

### What can cause this (in order of likelihood):

1. Contaminated source
2. Dirty source
3. Contaminated/degraded electron multiplier
4. Contaminated quadrupole
5. Pumping problem
6. Electronics problem

### Discussion of problem and what to do:

The first thing to do is to switch to the other filament (assuming the other filament isn't blown). If this doesn't remedy the problem, then it's probably one of the first six things listed above. Run AUTOTUNE and evaluate the report. Notice the number of peaks listed. For a properly functioning mass spec, the number of peaks (ions) for PFTBA, assuming you are scanning from 10-650 AMUs, should generally be between 100-200 peaks. If you are seeing an ion at every mass, that means that the mass spec cannot properly distinguish between signal and noise, hence *everything* shows up as a signal, which is obviously not correct.

If you notice that this happens only in manual tune, but NOT in your analytical run, then I would suspect a pumping problem. You can usually rule out any other possibility. The reason for this is that in manual tune, the PFTBA valve is open to allow the calibration gas to enter the source. During an analytical run, this valve is closed. Ion source pressure generally is slightly higher with the PFTBA valve open as opposed to when it is closed. In a properly functioning pumping system, this slight increase in pressures should not affect the mass spec's operation. However, if the pumping system is borderline, then the increased pressure from the valve being open may be enough to cause a problem. If this is the case, replacement of the E2M2 (or equivalent) roughing pump would be indicated.

What if the "ion at every mass" occurs even in your analytical run? You will end up with a very noisy baseline and, very likely, poor sensitivity. The problem could still be the rough pump, but it's more likely to be sourced related. There is a good chance some foreign matter exists in the ion source. The first thing I would do is a thorough source cleaning. If this problem occurs immediately after you have cleaned the source, then I suggest venting the system, dismantling the source, rinsing it in Methanol, and re-assembling. There is a difference between a "contaminated" source and a "dirty" source. A "dirty" source is a source whose lenses have become stained with a thin film through the normal use and operation of the system. A "contaminated" source is

one that has some foreign matter present somewhere (a piece of ferrule, column, lint, piece of metal, etc.). You can have a brand new or immaculately cleaned source that's "contaminated" if some foreign matter is present. If the source is "dirty", it needs to be cleaned. If the source is "contaminated", all you need to do is disassemble it, rinse it with Methanol, dry it and re-assemble it. I will be talking frequently about a "contaminated" source and a "dirty" source so it is important to recognize the difference.

Another possible cause is the electron multiplier. If you've cleaned the source and checked it for contamination, the problem could be the electron multiplier. You can clean and decontaminate the electron multiplier by sonicating it in Methanol for 3 minutes. Then let it air dry for 30 minutes. Alternatively, the electron multiplier could be shot. If the multiplier is old (more than 2 years) and you've noticed that your voltage in AUTOTUNE or Manual Tune is above 2,400 volts (even with a clean source), I suggest replacing the multiplier.

I want to make an important point. Many times in troubleshooting, there are a variety of factors that can cause a certain problem. Many times we'll suggest cleaning the ion source and/or changing the multiplier as a prelude to swapping electronic components. The reason for this is two-fold: first, a dirty source is often the problem so cleaning it makes sense as a first step. Secondly, if the problem is elsewhere, doesn't it make sense to do any future diagnostics and service on a mass spec with a clean source? I certainly think so.

Another possible cause of the problem is a contaminated quadrupole. If this ends up being the problem you may have a very expensive repair on your hands. For the 5970 MSDs, we have been able to successfully dismantle the quadrupoles, aqua regia and muffle furnace the ceramics, clean the rods and re-assemble it. We only do this as a last resort because it's a very time consuming process (and hence expensive) and there exists the definite possibility that the system may never be as good as it was before it was disassembled. For the 5971 and 5972 quadrupoles, we have been able to clean them by soaking them in a solvent. This is also a risky and time consuming process and should only be done as a last resort.

Servicing the quadrupoles is very risky business and I would advise against doing it. I list it here so you know that the quadrupoles could be the problem and they indeed can be cleaned (in some cases). As you will see, dirty quadrupoles are the cause of several problems, so we can't just ignore it. However, if dirty quadrupoles are the problem, you may be better doing a whole analyzer swap with Agilent. This is necessary because Agilent does NOT sell a quadrupole assembly by itself; only as part of a whole analyzer swap. This is unfortunate but that's the way it is.

The last possibility is that there's an electronics problem. The likely components if it is an electronics problem are as follows:

- 5970 MSD: RFPA Board
- 5971 MSD: Top Board, Main Board
- 5972 MSD: Top Board, Main Board
- 5973 MSD: Side Board, Main Board

### Mass Spectrometer Problem #3

Poor high end sensitivity

#### What can cause this (in order of likelihood):

1. Contaminated source
2. Dirty source
3. Contaminated/degraded electron multiplier
4. Mis-assembled source (5971, 5972, 5973)
5. Contaminated ceramics (5970)
6. Pumping problem/excessive water
7. Mass spec temperature incorrect
8. Electronics problem

#### Discussion of problem and what to do:

As is usually the case, the first thing we check is the source. For items #1, #2 and #3 above you can follow the same procedure as in Problem #2. The term "Poor High End Sensitivity" is a relative term. What, exactly, is "Poor High End Sensitivity"? In it's mildest case, it would be a system whereby AUTOTUNE generated only 1.5% of ion 502 and only 40% of ion 219. If this were the case it would certainly be fair to say that the system is exhibiting "Poor High End Sensitivity" (and "Poor Mid-Range Sensitivity" as well) but, it's probably not so bad that you couldn't run. In fact, for volatiles analyses, who cares about ion 502 being low - after all you're only scanning from 10-300 AMU anyway. The low ion 219 is of more concern because it may make passing BFB difficult (or impossible) with such poor mid-range sensitivity. If this is the situation, I suggest running with the system until you can't pass BFB or DFTPP anymore and then clean the source. If the electron multiplier is tuning above 2,400 volts, then change that as well.

In a more extreme case, "Poor High End Sensitivity" means having a mass spec where ion 502 is below 0.5% and ion 219 is below 20%. It is virtually assured that a system like this will fail DFTPP or BFB. In this case you'll need to service the instrument.

If this problem occurs right after you cleaned the source, you may have a mis-assembled source. You should keep printouts of your AUTOTUNE and Manual Tune reports before and after you took the source apart so as to check for any problems that arose because of incorrect reassembly of the source. For those users of 5971, 5972 and 5973, be very careful when reassembling the ion source to be sure the Ion Focus Lens, Drawout Cylinder and Drawout Plate are all making good connection inside the ion source body chamber.

For you 5970 users, the problem may very well be caused by contaminated ceramics. If that is indeed the case, you can clean the ceramics parts (dowels, spacers, drawout sleeve, and filament block) as follows:

1. Soak in aqua regia for 1 hour. Aqua regia is made by combining 3 parts HCl + 1 part HNO<sub>3</sub>
2. Rinse the parts in distilled/deionized water to get rid of the acid
3. Dry the parts at 150 °C
4. Fire in a muffle furnace at 1,000 °C for 4 hours

Allow the ceramics to cool and reinstall into the source and see what happens.

Up to this point, we've addressed the various solutions if the problem is the ion source. But what else can cause this problem besides a source-related issue? Well, there are several other things that can cause this.

There may be a pumping problem. If you have a turbo pump system, the problem is probably NOT the turbo pump, assuming that the vacuum is in the 10<sup>-5</sup> range or lower. Instead, the problem may be in the rough pump. If you have an oil diffusion pump system, the problem may be the oil diffusion pump system even if the vacuum is in the 10<sup>-5</sup> range or lower. Just as with turbo pump systems, the problem may be in the rough pump. How do you determine what to do?

Here's a good test to run: remove the carrier flow from the mass spec and see what happens. Remove the column from the injector and stick it into the side of a septum. This will stop the flow of helium into the system. Wait 30 minutes. Then run the AUTOTUNE again. If the tune looks a lot better, then that would indicate either a pumping problem or a shorted out source. If the tune looks the same, then I would suspect an electronics problem.

In addition, poor low end sensitivity is often accompanied by what appears to be an increase in water content. Since water is ion 18, it may just look higher than usual. What's happening is that as pumping problems can cause the low end masses to appear higher than normal, so the

amount of water in your system may be normal, but the amount of ion 18 relative to ion 69 in manual tune may appear much higher than normal. Usually the ratio of 18 to 69 is below 5%, but it is common for systems with pumping problems to have this ratio be 30% or more. So, don't be fooled into thinking you have excessive water in the system - you may just as easily have a distorted mass spectrum due to pumping problems.

Another variable that can cause poor high end sensitivity is incorrect mass spec temperature. If the ion source is too hot, high end sensitivity will be reduced. We recommend the following temperatures:

MSD	Source temp	Quad temp	Transfer line temp
5970:	200 °C	same as source	250 °C
5971:	N/A	N/A	280 °C
5972:	N/A	N/A	280 °C
5973:	200 °C	150 °C	280 °C

Keep the transfer line at 250 °C when using the 5970 as higher temperatures will degrade the magnet.

For the 5971 and 5972, the transfer line heater also heats the source and quad. This is why the transfer line needs to be kept at 280 °C with these models.

The 5973 is more like the 5970 in that the transfer line is a separate heated zone from the source/quad. The table lists the recommended temperatures we suggest.

The final factor that can cause high end problems would be electronic problems. You can refer to Problem #2 for a list of the likely culprits if the problem is indeed electronic.

## Mass Spectrometer Problem #4

Poor overall sensitivity

### What can cause this (in order of likelihood):

1. Leak
2. Contaminated/degraded electron multiplier
3. Weak filament
4. Contaminated source
5. Dirty source
6. Excessive water/pumping problem
7. Electronics problem

### Discussion of problem and what to do:

For this problem, the most likely cause is a leak. If your sensitivity in your GC/MS run is low (i.e. the areas of all the compounds are much lower across the board), you should immediately check manual tune. Remember that there are 2 kinds of leaks:

- The kind where air is being sucked into the Mass Spec (shows up as ion 28 in manual tune)
- The kind where helium and analytes are lost from the system

If it's the kind where air is being sucked into the mass spec it will be obvious when you check manual tune. The abundance of ion 28, which should be less than 5% of ion 69, will be greater than 50% and sometimes much higher. This will cause reduced sensitivity. Should this be the case, check the following:

1. O-ring on the mass spec (this is especially true for 5971 model, which has a grooved rectangular o-ring and is VERY susceptible to leaks).
2. The fitting where the column goes into the mass spec. We strongly suggest using stainless steel nuts rather than the brass ones, especially on semivolatiles systems where the oven cycles to 300 °C or more. The brass nuts tend to leak and eventually can strip the fitting.
3. The fitting where the column goes into the injection port.

If it's the kind where helium and analytes are lost from the system, it will be harder to detect. You'll know this is the case because your manual tune will look fine, yet your areas in your GC/MS runs will be significantly reduced. Here's where a handheld helium leak detector is invaluable.

If no leak in manual tune exists, yet the sensitivity in manual tune is low, I would next look to the ion source and/or the electron multiplier. Check the EM voltage - if it is tuning above 2,400 volts, you will often notice that sensitivity is lost. Adding more voltage above 2,400 (to 2,600 for example) many times will not improve sensitivity that much. That's a good indication that the multiplier needs replacing.

Of course, this is only true if the ion source is clean. Many times, high EM voltage is a by-product of a dirty source. Here's where record keeping helps us out. If we know the age of the multiplier, then that will help us to determine if it needs replacement. Certainly a 3 year old EM that has been in constant production is more likely to be "dead" than a one year old EM under the same production. In addition, a history of AUTOTUNE reports can help us. If our last 3 AUTOTUNE reports shown an EM voltage of only 2,000 volts and now it's up to 1,500 volts, it's unlikely that the EM is the culprit. It is more likely that some other problem (typically a contaminated or dirty source) is causing the system to run with a high EM voltage.

Try switching filaments. Many times, if one filament has been in use for several months, it will begin to “sag” as it weakens. This can lead to reduced overall sensitivity.

So, our next step would be to clean the source. We suggest installing two fresh filaments whenever the source is disassembled for cleaning. If the EM is old or has been tuning at higher voltages even with a clean source- we’d replace the EM as well.

Another factor that can cause poor overall sensitivity is the presence of excessive water. This is rare in semivolatiles systems but much more common in volatile systems where water vapor is constantly being desorbed from the purge and trap, through the GC column and into the mass spec. This can translate to a loss of overall sensitivity. If you have a volatile system and are seeing ion 18 (water) at high levels- try “drying out” the system. Bake your column at 200 °C for 15 minutes (being sure NOT to desorb your purge and trap while doing this). If ion 18 persists, then either you’re overloading your system with water or the rough pump is not functioning properly.

You can do the same test we mentioned before- i.e. plug off the mass spec from any carrier flow. If the sensitivity improves, then that would support the belief that the pumping system is faulty. If nothing changes, it’s either source or electronics related.

How do you know if you’re overloading your system with water or the rough pump is not functioning properly? Well, assuming you’re using a VOCARB 3000 trap and are employing dry purge in the 2-5 minute range and a desorb time in the 2-4 minute range, then that should be sufficient to remove the excess water and not overload the system. The mass spec pumping system should be able to remove the rest. So double check your dry purge/desorb settings and if they are in range and the purge and trap is indeed working correctly then you can then assume the problem is the pump.

OK, but what if you have reduced sensitivity even though you have a clean source, new filaments, new EM, no leaks and a new pump - well then the problem is electronic. Once again, you can refer to the earlier problems in directing you as to which board could be the problem.

## Mass Spectrometer Problem #5

Poor peak shape in manual tune

**What can cause this (in order of likelihood):**

1. Improperly set AMU gain/AMU offset
2. Incorrect polarity setting
3. Dirty source (especially at the entrance lens)

4. Dirty quadrupole
5. Faulty quadrupole wiring
6. Pumping problem
7. Electronics problem

### Discussion of problem and what to do:

Don’t confuse poor peak shape in manual tune with poor GC peak shape. Poor GC peak shape is a function of your column, your flows, pressures, etc. What I’m referring to is the peak shape of the three key ions in manual tune or AUTOTUNE. Once again, look at your AUTOTUNE. You should notice nice, sharp, Gaussian (symmetrical) peak shapes of ions 69, 219 and 502. The common examples of poor peak shape are:

- Peak splitting
- Fronting
- Very narrow peak widths
- Very wide peak widths

These problems need to be resolved because if not, the mass spec will have difficulty resolving one ion from the next. This will translate into failing for DFTPP/BFB, especially for the minor isotope ions. So pay close attention to peak shape when you generate your manual tune reports. Think of profile scan as a 2-dimensional version of the bar chart that makes up a spectrum scan. The 2-dimensional profile scan enables us to readily see how well the mass spec resolves one ion from the next- especially one large ion next to one smaller ion.

To resolve the problem of very narrow peak widths or very wide peak widths, adjust the AMU gain and AMU offset in manual tune. The AMU gain affects high masses more than low masses, and the AMU offset affects all masses equally.

As you increase the AMU gain or AMU offset, the peak widths should narrow. As you decrease the AMU gain or AMU offset, the peak widths should widen. For all MSDs other than the 5970, you can also adjust peak width via the “219Wid” setting. The default setting here is 0.000 but you can adjust it either slightly positive or negative to help improve peak widths. This setting affects the mid-masses (131 and 219) mainly.

So, if you adjust the peak widths to 0.50 AMU and the peak shape still is unsatisfactory, move on to the next step. The next step is to change the polarity on the quadrupole. For all MSDs, other than the 5970, you can also adjust the polarity by the “DC POL” setting. It’s either positive (POS) or negative (NEG). Try switching to the other polarity, then re-running AUTOTUNE. If the

peak shapes improve, you can then manually tune the system (using the new polarity setting) and you're back in business. For the 5970, changing the polarity involves switching 2 wires on the RFPA Board. You will probably have to tune the RF coils should you do, this so that might be best left to a service technician. More information on Tuning the RF coils is given later in this issue. If changing the polarity doesn't resolve the problem, then it's safe to say the problem isn't with the tune file, but with some underlying hardware.

The next step would be to clean the ion source, paying extra close attention to the entrance lens. The reason for this is the entrance lens is the component of the ion source that comes in contact with the quadrupole. It's the entrance to the quad which is why it's called the "entrance" lens. Any contamination on the entrance lens can cause distorted peak shape in manual tune.

If, after cleaning the source and replacing the entrance lens, the peak shape is still poor, the next likely cause is the quadrupole itself.

The quadrupole (also called the mass filter) is the component of the mass spec that selectively filters one ion at a time such that the ion stream created in the source can be analyzed, one ion at a time, by the electron multiplier. Suffice it to say that contamination on the quadrupole can lead to distorted peak shape. Should this be the case, this is definitely a service issue. In some cases, the quadrupoles can be cleaned (or in the case of the 5971 and 5972 they can be rotated 180°) so as to improve peak shape. This is a risky procedure and disassembly of the quadrupole is a last ditch effort because many times the system will not work at all once the quad has been disassembled. So, contaminated quadrupoles is about as serious a problem as one can have because it may not be repairable. We have had success cleaning quadrupoles but, as I said, it's something we do only in extreme cases.

Another common problem, especially among 5971 models, is faulty quadrupole wiring. We ran into this problem circa 1991-1993 when the 5971's first came out. Faulty quad wiring caused somewhat odd peak shape which translated into many systems failing DFTPP because ion 441 could never be detected. This problem was addressed when H-P/Agilent introduced the 5972 model MSD, whose quadrupole wiring was far superior to the 5971 and remedied the peak shape/missing 441 ion problem. Should you have an older 5971 with the original wiring and are having problems passing DFTPP you may wish to have a service tech re-wire your quad.

Another problem that can cause poor peak shape is a pumping problem- especially rough pump problems. If the vacuum isn't as low and stable as it should be, the peak shapes will often suffer. Replacing the rough pump with a new or rebuilt pump often solves the problem. If there is a pumping problem, poor peak shape will usually be accompanied by the other problems that accompany rough pump problems- such as poor high end sensitivity, exaggerated low end sensitivity, or poor overall sensitivity. It's rare that a pumping problem would cause poor peak shape alone without the other problems as well.

The final factor that can cause poor peak shapes is an electronics problem. Since peak shape is a quadrupole-related issue, the electronics that drive the quad, RF or DC amplifiers, would be the things to check and replace.

## Mass Spectrometer Problem #6

Error message: "Excessive source pressure"

### What can cause this (in order of likelihood):

1. Ion source chamber pressure in the mid 10<sup>-4</sup> range or higher due to a massive leak
2. Ion source chamber pressure in the mid 10<sup>-4</sup> range or higher due to a pumping problem
3. Ion source chamber pressure in the mid 10<sup>-4</sup> range or higher due to excessive column flow
4. Shorted out ion source

### Discussion of problem and what to do:

The first thing to do is to check the ion source chamber pressure with the PFTBA valve CLOSED. We have seen the error message "Excessive source pressure" even though source pressure was in the 10<sup>-5</sup> range. The point is that you can receive misleading and erroneous error messages so you have to know what to check for.

If you have an ion gauge controller and tube connected to your mass spec, check the source pressure. If the source pressure is higher than about 3 x 10<sup>-4</sup> torr then indeed the source pressure is too high. OK, what do you do? Assuming that the system gave you this error message while the system was operative (or 1 hour after you started the pump down cycle) then you should check the following:

1. Broken column or transfer line. If you are running capillary direct into the source, check for any breaks in the column. Often a column will break and the air getting sucked into the MSD will elevate the source pressure to the point where it cannot scan.



2. Loose fitting on the MSD inlet. Also check the fitting where the column/transfer line enters the MSD. If it is loose, massive amounts of air will get sucked in. Also, if it seems tight it may be that the ferrule has cracked, in which case massive amounts of air will get sucked in even though you cannot tighten it any further.
3. Check the hose and clamp that connect to the MSD rough pump. If it is loose, tighten it up.

Keep in mind that a MASSIVE leak must exist for you to get “Excessive source pressure”. If you have checked all the fittings and don’t think a leak exists, it may be a pumping or flow problem. Here’s what to do.

Vent the system and cap off the MSD. You need a blank ferrule and nut to accomplish this. Once the MSD is capped off try pumping it down. The system should pump down to the mid 10-5 range very quickly and low 10-6 range after about 2 hours. If not, a pumping problem is likely. If you have a turbo pump, then the turbo is probably not the problem (although it could be). It’s more likely to be the rough pump. Swap rough pumps and see what happens. If that fixes it fine; if not then look to the turbo pump or oil diffusion pump.

If you have an oil diffusion pump, then the problem could be either the rough pump or the oil diffusion pump (it’s 50-50). Swap rough pumps and see what happens. If that fixes it fine; if not then the problem is likely to be the oil diffusion pump. It’s possible the pump isn’t shot, but rather that it’s low on oil diffusion pump fluid. A full charge of oil diffusion pump is inexpensive and can be done in a short time. It’s important to keep track of when oil diffusion pump fluid was added. Unless your lab is excessively hot, oil diffusion pump fluid only needs to be recharged every 5 years or so.

If it’s not a leak or pumping problem, possibly there is excessive flow into the system. Obviously, if you capped off the mass spec and the problem still exists, then carrier flow cannot be the cause. However, if it pumps down quickly when capped off but cannot pump down when under flow, double check your column flow. Keep in mind that we STRONGLY suggest using 0.18-0.25 mm ID columns for Agilent MSD models 5970, 5971, 5972, and 5973 because those columns keep the flow between 0.4 and 1.2 mL/minute which is ideal for the benchtop models. Wider bore columns (e.g. 0.28 and 0.32 mm ID) can be used, but we advise against it. Keep the flow between 0.4 and 1.2 mL/minute and flow will never be a problem for you.

“Excessive source pressure” can also be given if the source has been shorted out. For 5971/72 systems, if the source is contacting the transfer line it will short out and all kinds of error messages can occur.

## Mass Spectrometer Problem #7

Air leak

### What can cause this (in order of likelihood):

1. Leaking O-ring (especially with 5971 models)
2. Leaking PFTBA valve
3. Tuning file biased towards low mass
4. Leaky fitting into the MSD
5. Leaky fitting at the injector

### Discussion of problem and what to do:

First, let’s define an air leak. For air leak to truly exist, you must see the following ions:

- Ion 28 Nitrogen (N<sub>2</sub>)
- Ion 32 Oxygen (O<sub>2</sub>)
- Ion 40 Argon (Ar)

Nitrogen is the major component of air, with Oxygen the second largest component . Argon is present in small amounts.

If an air leak really exists, you will see BOTH ion 28 and ion 32. If you see JUST ion 28, then that means your Helium tank is contaminated with Nitrogen, rather than having an air leak. Change tanks and see if that solves the problem.

OK - so let’s say you see ions 28 and 32 with small amounts of ion 40. Chances are that you will also see ion 18 (water) and ion 44 (CO<sub>2</sub>) since they are present in the air at low levels. There’s always some humidity in the air and we breathe CO<sub>2</sub> into the air constantly. If indeed you are seeing air, then that means you have the kind of leak where air is being sucked into the system.

If you have a 5971 MSD, the chances are very good that the O-ring is leaking. The O-rings of the other Agilent models seal reliably but the grooved 5971 O-ring is notorious for leaking. Vent the MSD, reseal the 5971 and see if that fixes it.

You might ask “what level of air is OK and what level is excessive”? Well, that’s a good question and to some extent is determined by each user. Certainly, if the abundance of ion 28 is below 5% of ion 69 in manual tune then you’re OK. If it’s above 20% then that would certainly warrant service. The real determination is the baseline you get in your chromatogram. While you’d like to see the air as low as possible in manual tune, if the baseline in your chromatogram is excellent, then don’t worry about the air leak in manual tune. If you are

scanning from 35 AMU in your method, only ions 40 and 44 will appear in your run. So, it's up to your discretion as to how high a baseline you can tolerate.

OK, let's proceed on the assumption that the air leak is excessive and must be minimized.

Go into manual tune. The FIRST thing to do is monitor the raw counts of ion 28. Set ion 28 as one of the three ions to be monitored. Then close the PFTBA valve and continue scanning. If the raw counts of ion 28 stay essentially the same then you can rule out a leaking PFTBA valve. If the raw counts of ion 28 drop after the valve is closed, then you know the leak is due to a faulty PFTBA valve and you should arrange to have it replaced as soon as possible. If it's not a leaking PFTBA valve move on to the next step.

Sometimes, your tune file can be such that it's "low mass biased". This means that the combination of settings you have to obtain adequate mid-range and high-mass sensitivity also magnifies the low end sensitivity. You may notice that the abundance of ion 28 rises and falls as you make changes in your manual tune file (especially with the ion focus, repeller and entrance lens offset). So, a small air leak can be tuned to seem magnified, and a large air leak can be tuned to seem minimized. Keep that in mind. If possible, try to achieve the normal mid-range and high-range abundances and, at the same time, keep the 28 to a minimum. I know that's a little tricky but sometimes it can be done. It's worth spending a little extra time in manual tune so as to end up with a file that keeps ion 28 to a minimum.

After all efforts in manual tune have been exhausted, then you have to look elsewhere. As I said in the previous problem, excessive source pressure, you may have a leaky fitting somewhere inside the GC. I would suggest venting the system and re-doing the fitting going into the MSD. Always use 85% Vespel/15% Graphite ferrules at the MSD end. They are less likely to crack or get compressed out of shape than the 60% Vespel/40% Graphite which are good to use at the injector end. NEVER, EVER use 100% Graphite ferrules at the detector end. For the MSD end, use the larger triangular ferrules and always remember to put the wider part of the ferrule towards the MSD end and the tapered side of the ferrule towards the interior of the GC.

If you get stuck in your leak checking try the following:

For capillary direct systems, remove the column from the injector and stick it into the side of a septum. This will cut off flow to the MSD and remove the injector from the loop. Wait 30 minutes. If the leak subsides, you know the problem is in the injector. If the leak stays, you know it's in the mass spectrometer.

Air leaks can be a real pain to diagnose, especially in VOA systems where the presence of the purge and trap complicates matters. In a subsequent issue we'll discuss troubleshooting the purge and trap.

## Mass Spectrometer Problem #8

Excessive water levels

**What can cause this (in order of likelihood):**

1. Water in the system
2. Dirty source
3. Pumping problem
4. Tuning file biased towards low mass
5. Source/analyzer temperature too high

**Discussion of problem and what to do:**

First, let's define "excessive water levels" as the presence of ion 18 to be greater than 10% of ion 69 in Manual Tune. Since we are scanning from 35 AMU in our EPA methods, excessive water will not show up in our TIC, but can cause the mass spec to behave erratically which will make it difficult for us to maintain linearity and sensitivity. So, it should be corrected.

If you have a semivolatile system you can rule out #1 above (water in the system) because there's no way any significant amount of water enters the system. For volatile systems, there's a constant introduction of water into the system from the repeated desorption in the purge and trap cycle. If you are seeing lots of ion 18 in manual tune, make sure the purge and trap is in STANDBY. Give it 30 minutes to stabilize and try scanning again. By then, all the water should be pumped out of the system. If not, continue on with the troubleshooting.

The next thing to check is the same thing I mentioned in the previous problem - i.e. that your tune file isn't biased so as to magnify the low end.

After all efforts in manual tune have been exhausted, then you have to look elsewhere. The problem could be a dirty source. One of the problems that accompanies a dirty source is reduced high-end sensitivity. That often translates to artificially enhanced low-end sensitivity. Since ion 18 is as low as we see (we scan in manual tune to 10 AMUs but ion 18 is the lowest significant ion we see), it would be most affected if the low end has been artificially enhanced. Try cleaning the source and see if that fixes it. Sometimes, if excessive air hits the mass spec over extended time, the source can become oxidized and that will create the artificially enhanced ion 18 effect.

Assuming that the problem is not the tune file nor a dirty source, the next most likely culprit is the mass spec rough pump. Even though your source pressure might be excellent, your rough pump might need replacing. For those of you with 5971, 5972 and Oil-Diffusion type 5973 MSDs, check the “Vacuum” reading in your manual tune report. This reading is the foreline pressure (pressure through the rough pump) in millitorr. Typical settings are 20-60 millitorr depending on the flow and configuration. Here’s where having a history on manual tune reports is helpful as you can compare your current reading to historical readings. Elevated water levels, in conjunction to an elevated foreline pressure, strongly suggests a rough pump problem.

For those of you with oil diffusion pumps, the problem could be depleted diffusion pump fluid (as we mentioned when air levels are high) so that might be the culprit.

Finally, source/analyzer temperatures that are too high also exhibit artificially enhanced low end sensitivity. See Mass Spectrometer Problem #3 for our recommended temperatures.

## Mass Spectrometer Problem #9

RF coils out of tune

### What can cause this (in order of likelihood):

1. Normal drift over time
2. Contamination build-up on the quadrupoles
3. Polarity change on the quadrupoles
4. Replacement of certain electronics

### Discussion of problem and what to do:

Actually, the need to tune the RF coils in and of itself is not indicative of a problem. Think of this procedure as a “tune-up” for your quadrupole. First, let’s define what we’re doing by tuning the RF coils. Basically, what we do is adjust the RF (radio frequency) power being applied to the quadrupoles. Think of the quadrupole as a radio antenna, which needs to be tuned to balance the radio energy being transmitted by the quads into the space between them. This power is used to separate or “mass filter” the ions being produced in the source so that the electron multiplier at the opposite end only sees one ion at a time. There is an electrical circuit which measures the amount of RF energy not transmitted, or reflected, back from the quadrupole cavity and reads this out as the RFPA (Radio Frequency Power Amplifier) voltage. The lower the voltage, the less energy is reflected, and the better tuned the quads are. Check out your mass spec manual for the specific details of the procedure for measuring this voltage. Tuning coils located on the RFPA

electronics are then adjusted, if necessary, to achieve the proper RFPA voltage.

You would suspect that the RF coils need tuning if the system is indeed having difficulty performing mass axis and peak width calibrations, or if you’ve changed the polarity due to poor peak shape in manual tune profile scan.

Sometimes, buildup of contamination on the quadrupole surface will cause the RF to become out of tune, so it’s a good idea to check it if you are failing DFTPP or BFB for the minor precursor/isotope ions (ion 96, 175 and 177 in BFB and 68, 70 199, 441, and 443 in DFTPP).

You will also need to tune the RF coils if you replace your analyzer or swap certain electronic components (especially those which drive the quadrupole electronics). In general, tuning the RF coils is simple enough (and important enough) that it is often done anytime there is a source or quadrupole related problem. That’s why it’s good to learn how to do it because being able to do it may end up saving you a lot of diagnostic time somewhere down the road.

## Mass Spectrometer Problem #10

High foreline pressure

### What can cause this (in order of likelihood):

1. Excessive flow into the mass spec
2. Problems with the rough pump
3. Leak in the mass spec

### Discussion of problem and what to do:

First off, let’s define “foreline pressure” as the pressure through the mass spectrometer rough pump expressed as millitorr. Typical foreline pressure is in the 20-60 millitorr range. Only H-P/Agilent systems with oil diffusion pumps (i.e. 5971, 5972 and some 5973) enable you to monitor the foreline pressure via the software. The reading in millitorr is listed as “Vacuum” in the tune reports. For turbo pump systems (i.e. 5970 and some 5973) there is no readout so you’re on your own in trying to evaluate foreline pressure.

For this discussion let’s make the assumption that you have a GC/MS system with an oil diffusion pump (i.e. 5971, 5972 and some 5973). The next question becomes “what is normal or good foreline pressure and at what point would it be considered excessive”? Since the software doesn’t notify you when the foreline pressure is too high, you have to determine it yourself. Here’s where having a history of manual tunes is very helpful because you can check to see what the foreline pressure

has historically been. Since the foreline pressure will vary depending on the configuration of the system, it's difficult to know when it is excessively high. The usual range is fairly wide; we've seen some systems as low as 10 millitorr and others as high as 80 millitorr. Generally, the lower the flow into the mass spec the lower the foreline pressure will be. Volatile systems using a 0.18 mm ID column directly into the source generally have foreline pressures at the lower end of this range due to the sub-1.0 mL/minute flow into the source. Semivolatile or volatile systems using a 0.25 mm ID column directly into the source generally have foreline pressures in the high end of this range due to the greater than 1.0 mL/minute flow into the source. These are good guidelines but knowing the historical foreline pressure (especially when the system was running optimally) is vastly superior to any ballpark estimates.

What can occur if the foreline pressure is too high? The biggest problems are:

1. Instability of the mass spectrometer
2. Failure of BFB/DFTPP
3. Poor high end sensitivity

What should you do if this happens to your system:

1. Try lowering the flow. Remember to keep the total flow into the mass spec below 1.5 mL/minute, and below 1.2 wherever possible.
2. Change the oil in the rough pump. Vent the system, and drain the oil. Refill using INLAND 45 mechanical pump oil or whatever the manufacturer recommends.
3. Replace the mass spec rough pump. If you were stuck with an Edwards E2M1.5 pump you should consider upgrading to either an E2M2 or RV3.
4. If you already have either an E2M2 or RV3, you should look at having the rough pump rebuilt.
5. Check for leaks. If you have a leaking O-ring or some other leak that is allowing air to get sucked into the mass spec, that can also cause high foreline pressure. You will know if this is the case because you will see ions 28 and 32 in your manual tune (ions indicative of a leak whereby air gets sucked into the mass spec).

One final note: High foreline pressure is a tricky problem. Many times failure of BFB/DFTPP can be attributed to very minor problems with the rough pump. If the manual tune seems erratic or inconsistent even after you've cleaned the source, you might suspect the rough pump. A good idea would be to have a spare Edwards E2M2

pump in your lab, because it typically takes 2 weeks to get them rebuilt and you don't want to be down for that length of time.

## Mass Spectrometer Problem #11

Mass spec times out

**What can cause this (in order of likelihood):**

1. Loose cable somewhere
2. Entire system needs to be reset
3. Problem with the PC I/O board
4. Problem with the mass spec smartcard
5. Problems with the hard drive
6. Corrupt file in the MS Chemstation

**Discussion of problem and what to do:**

When I use the term "Mass Spec Times Out" I mean that some communications problem has occurred to prematurely abort a run or sequence. I'm NOT talking about a mass spec problem that would stop a run (such as a sample overload or something along those lines), but rather something external to the operation of the source, quadrupole and electron multiplier.

Let me distinguish between a "timeout" and no communications at all. A "timeout" is one where the mass spec temporarily shuts down, usually over and over, and needs to continually be restarted. That is different from a situation where you can't get a run to start at all. The problems I've listed can cause both "timeouts" and no communications at all.

The first thing to check is all the cabling between the mass spec, GC and PC. If a cable has come loose or is not snug against the electronic component to which it connects you can get the timing out problem.

If all the cabling looks fine, you are likely to either have a defective board or problem with the computer. If you are not having any other computer problems then it's possible that you just need to reset the entire system. To do this proceed as follows:

- A. Close out of the Chemstation
- B. Close out of Enviroquant
- C. Close all other applications programs, leaving you with just the desktop open
- D. Shut down the PC
- E. While the PC is off, power cycle the mass spec

(turn the power off, wait 2 seconds and turn it back on. For 5970s, you can just power cycle the Heater/Electronics switch)

- F. While the PC is off, power cycle the GC (turn the power off, wait 2 seconds and turn it back on)
- G. Reboot the PC
- H. Run manual tune to download the Chemstation software and verify that the system can scan
- I. Resume your sequence from where you left off

This procedure often works; if not you have to move to the next step. The order that you do the next 3 steps is up to you. We would first swap each or both of these boards to see if either restores communications. The SmartCard is the board inside the mass spec that enables it to communicate with the rest of the system. The PC I/O (Input/Output) board is a small board which connects to the mother board of the PC enabling the PC to run the GC/MS. We have seen numerous failures of both of these parts which cause the mass spec to “time out” or not to work at all.

If the problem isn't one of the above mentioned items, it may reside with the PC. Another possible solution would be to reload and reconfigure the Chemstation software. Sometimes, a file gets corrupt and reloading and reconfiguring resolves the problem.

The final possibility is a hardware problem with the PC hard drive itself. In this case, back up ALL your methods and data files and any other custom programs and install a new hard drive or install a different PC and see if that resolves it.

### Mass Spectrometer Problem #12

Diffusion pump too hot/too cold

#### What can cause this (in order of likelihood):

1. Faulty diffusion pump switch and cable assembly
2. Insufficient oil diffusion pump fluid in the pump
3. Faulty diffusion pump

#### Discussion of problem and what to do:

Obviously, this problem can only occur on systems with oil diffusion pumps. When you see the Diffusion pump too hot/too cold error message the mass spec will have already put itself into vent mode. If the oil diffusion pump can maintain the proper temperature it will shut down for sure.

The simplest thing to do is to replace the diffusion pump

switch and cable assembly. Often that solves the problem without any further action necessary. If not, a good idea would be to drain the fluid out of the diffusion pump, and replenish with a full charge of new fluid.

If the problem is neither the diffusion pump switch and cable assembly nor insufficient oil diffusion pump fluid, then it's likely the diffusion pump heater itself is faulty. Since this is rare, when this happens we usually replace the diffusion pump with a new one and that resolves the problem.

### Mass Spectrometer Problem #13

Sensitivity drift/decline over time

#### What can cause this (in order of likelihood):

1. Weak filament
2. Dirty source
3. Aging/degrading electron multiplier
4. Problems with the purge and trap
5. Problems with the injection port

#### Discussion of problem and what to do:

Many customers report that as they run their sequence, they notice the areas of the internal standards decline from run to run. The decline usually is gradual, but significant. Then, the next day after sitting idle for several hours, the response returns to normal, only to fall off again during the next sequence (typically of 10-20 runs). This cycle repeats itself and makes passing ISTD and Surrogate recoveries as well as spikes very difficult. To resolve this, we have to identify the source of the problem.

The problem is that the problem could be JUST the mass spec, JUST the purge and trap or JUST the injection port...or maybe more than one of these. That's the tricky part of this problem many things can cause it. So, how do we go about resolving it.

First, let's determine how stable the mass spec is. With the purge and trap idle and with the oven at 150 °C, start scanning in manual tune. Just sit and watch it for about 15 minutes.

Let's assume that your target abundance for ion 69 in manual tune is 1 million counts. As you begin scanning and allowing 60 seconds for the system to full stabilize, the abundance is almost exactly 1 million counts. In a properly functioning mass spec, if you were to walk away and come back 24 hours later, the abundance for ion 69 should be within 10% of where it was originally. Now, it's impractical to perform this test, as you would certainly

not want to lose a day's production while evaluating the mass spec's stability. However, you might want to spend 2 hours doing this test as the same information can be obtained. If, after 2 hours, the abundance for ion 69 is rocksteady, then you can rule out the area drift as being related to the mass spec. You would then look to the purge and trap or injection port as likely culprits.

However, let's say that 2 hours later the abundance for ion 69 is now 850,000 counts. That's a 15% decline. Wait another hour. If it continues to drop then you've found the problem it's the mass spec because this kind of decline should NOT occur. OK, so now what? If the sensitivity declines but pretty much everything else stays constant (i.e. peak widths, mass axes, relative ratios), then the problem is very likely a weak filament, or possibly a dirty source or aging/degraded electron multiplier, although the filament is far more likely the problem.

**KEY POINT:** If the sensitivity decline is resolved by doing nothing more than letting the mass spec "rest" for 2 hours, you can be reasonably sure the problem is in the mass spec itself.

You have to check each filament separately and thoroughly. The next step would be to switch to the other filament. Many GC/MS analysts believe that either a filament is blown, or working optimally with no in-between. This is not true. As filaments age, they can sag and become weak. When this happens, you can see a sensitivity decline over a 10-12 hour period that the filament is energized. Then, after the sequence ends and the filament has a chance to cool, the sensitivity jumps back up, only to repeat the problem with the next sequence.

If you determine that both filaments are weak or blown and thus the system needs to be vented, you might as well clean the source and change the rough pump oil anyway. You should also replace the electron multiplier if it was tuning above 2,400 volts. In fact, usually we change filaments and change rough pump oil whenever a source cleaning is indicated, and clean the source and change rough pump oil whenever changing the filaments is indicated. I mean, why not - the system is vented so you might as well do all the PM (preventative maintenance) tasks. The only exception would be if we have to change filaments on a volatiles system (especially a 524.2 system) which has been stable and passing BFB reliably, in which case we might be inclined just to change filaments as the source and rough pump oil tend to stay clean for extended periods on such systems.

If, after doing the PM, the problem STILL occurs, then changing the electron multiplier is indicated. In rare cases a sensitivity decline (in the absence of other mass spec problems) can be caused by an electronics problem. I

would suggest that the electronics that drive the filament circuitry could possibly be a cause but when this happens you usually see other instabilities in the mass spec, not just sensitivity decline.

If the problem is NOT in the source, then you need to look elsewhere. At this point, how you proceed depends on how your system is configured.

**Semivolatiles:** Sensitivity decline problems not related to the mass spec would then likely be related to the injection port. If you are running sediment or other samples with non-volatile/non-semivolatile residue (that black material that ends up all over your inlet liner), then you can see sensitivity decline. What happens is that with each successive injection, some of this residue clogs the column and/or adsorbs to the liner and inlet seal. This in turn reduces the amount of analytes entering the column (either by reducing vaporization and/or restricting entrance into the column), so if you run a batch of 12 of these extracts you can see lower and lower areas. Once you do the injector maintenance the sensitivity is restored.

**KEY POINT:** If the sensitivity decline is resolved by doing nothing more than performing the routine injector maintenance, you can be reasonably sure the problem is contamination caused by extracts.

**Volatiles:** Sensitivity decline problems not related to the mass spec would then likely be either purge and trap or autosampler related. The first thing to do would be to change the trap. Don't discard your old trap just yet - only do so if changing the trap resolves the problem (which would show that the previous trap was indeed the problem). Leaks or contamination in the purge and trap or autosampler would lead to haphazard and inconsistent results rather than a slow but steady decline over the course of a sequence. Generally, only a degraded trap would cause sensitivity decline during a sequence. Then, after the trap has had sufficient time to rid itself of water and methanol buildup, it is sometimes able to restore sensitivity.

**KEY POINT:** If the sensitivity decline is resolved by doing nothing more than replacing the trap, you can be reasonably sure the problem is degradation of the trap itself.

**FINAL NOTE:** Keep in mind that the problem could be the sum of multiple problems. This is why you need to test the mass spec first, then only after you've ruled out the mass spec as the culprit you can check other parts of the system.

## Mass Spectrometer Problem #14

Excessive amount of ion 44 in your TIC

### What can cause this (in order of likelihood):

1. Air leak (means the ion 44 is CO<sub>2</sub>)
2. Contaminated helium supply (means the ion 44 is CO<sub>2</sub>)
3. Glass/column fragments in the source (means the ion 44 is SiO)
4. Degraded trap (means the ion 44 is CO<sub>2</sub>)
5. Leak (means the ion 44 is CO<sub>2</sub>)

### Discussion of problem and what to do:

The first thing we have to do is determine what the ion 44 indicates and the source of it. Usually, ion 44 indicates the presence of CO<sub>2</sub>, but in some cases it could be SiO, which is what glass and fused silica are made of.

The first step is to use manual tune to determine if the problem is in the mass spec.

First, determine if an air leak (the kind where air gets sucked into the system) exists. With the purge and trap idle and with the oven at 150 °C, start scanning in manual tune. If there is an air leak, then the 44 is attributable to the CO<sub>2</sub> that's in the ambient air. For this to be the case, there would have to be large amounts of ion 28 (nitrogen) and 32 (oxygen) as well as trace amounts of 18 (water) and 40 (argon) in addition to the 44 (CO<sub>2</sub>). If this is the case you need to resolve the air leak. (How to do this was addressed earlier in this issue.)

If you see mainly ion 44 and little else of ions 18, 28, 32 and 40, then there is NOT an air leak. Instead you have one of the following occurring:

- A. CO<sub>2</sub> contamination in your helium supply. Check your supply. Was a new tank recently installed? CO<sub>2</sub> and Argon (ion 40) are common contaminants in helium, so don't be shocked if it happens to you. Even though you may be using UHP (ultra high pure) helium, you can still get a bad tank every now and then. There are no scrubbers that I know of that scrub CO<sub>2</sub> or argon so if you get a bad tank you'll have no choice but to replace it.
- B. If the ion 44 is NOT linked to the gas supply, it may be that it's NOT CO<sub>2</sub> at all, but rather SiO, which is basically sand, which is the building block of glass and fused silica capillary columns. To check for this, plug off the column with a septum at the injector end (i.e. remove the column from the

injector and stick it into the side of a septum to remove the flow of helium to the mass spec). Wait about an hour. If ion 44 still remains high then you can rule out the gas supply as the problem and assume the 44 is from SiO. Sometimes a small piece of glass or column fragment can enter the source and get continually ionized. Vent the system, clean the source, and the problem should be remedied.

If you have a volatiles system and see ion 44 ONLY in your chromatogram but NOT in manual tune, then that means the 44 is CO<sub>2</sub> and is likely being created by the trap and entering the system during the desorb mode. The carbon in the trap combined with heat, water and air can create excessive amounts of CO<sub>2</sub>. When this occurs you might see elevated amounts of ion 44 for the first 10 minutes or so of your run, and then the baseline returns to normal. The first thing to do is change the trap, and you might want to leak check the system as well while you're at it.

Another possibility is that a leak exists (the kind where helium and analytes are lost from the system) which is reducing the response of your analytes. Hence, normal amounts of CO<sub>2</sub> will seem larger by comparison in the TIC (total ion chromatogram) as the software scales the peaks. If this is the case, a thorough leak checking of the purge and trap (for volatiles) and injection port (for volatiles and semivolatiles). Once the leak is resolved and the areas of the analytes returns to normal, the signal-to-noise will be greatly improved.

**Questions or comments on this or any issue of OPTIMIZE may be emailed to the author, Mark Ferry, at [MFerry@SPEX.com](mailto:MFerry@SPEX.com).**

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