Evaluating Instrument Status for Evolved Gas Analysis

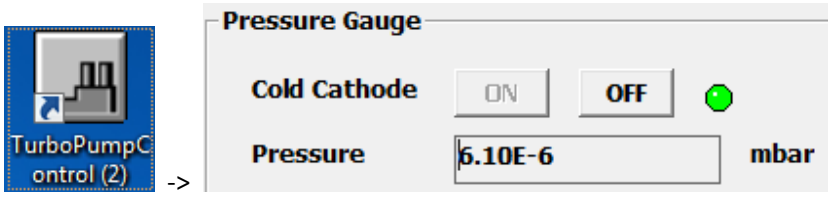
07/25/17 SWV

Often the samples that are run in the TGA/DSC furnace evolve gases at higher temperatures. Some of the evolved gases may be small molecules like CO2, CH4, H2O, et cetera, that travel through the MS fused-silica transfer line and the IR Teflon transfer line without condensing. Those transfer lines, and the IR light pipe assembly are kept at 200C. However, some gases evolved at higher temperatures do condense in the transfer lines and in the light pipe, eventually leading to partially or fully clogged lines and deposits on the light pipe windows. This significantly reduces IR and/or MS measurement sensitivity. Soot formation is especially likely to occur if the decomposition temperature of material is reached in the absence of oxygen. There are several steps you can do to minimize your contribution to these problems:

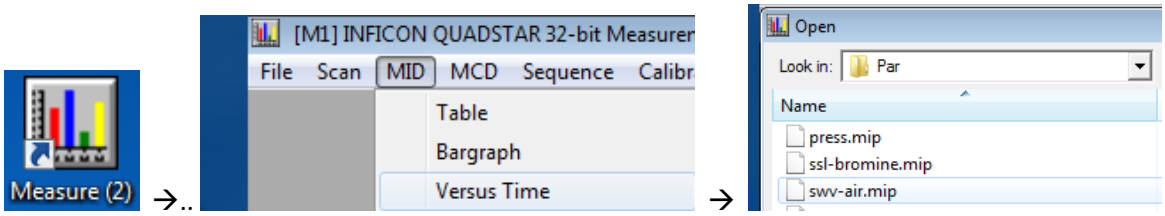
- Use the minimum necessary sample size. Two to five milligrams is more than enough for most applications.
- Don't heat your sample to the decomposition or melting point unless these are the thermodynamic events you are investigating.
- Do your measurements with air purge gas whenever possible. We offer both breathable (moist) and synthetic (dry) air.
- If you are not using air as the purge gas, test the melting/boiling/decomposition properties of a representative sample by using our external furnace configured with nitrogen purge. I can show you how to do that. If the external surface of the crucible darkens significantly in this test, then you know that species are being evolved that condense outside the crucible and will cause contamination of surfaces and transfer lines in the TGA instrument.

Testing MS Performance

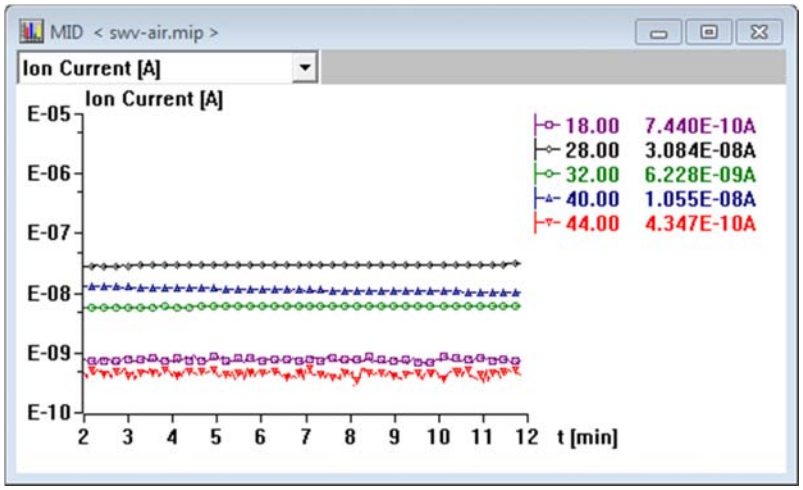
Two simple tests will help you decide if the MS transfer line is clogged, requiring maintenance. First, start the Aeolos Turbo Pump Control program. Turn on the Pressure gauge. The reading should be in the mid-to-high E-6 range. It will differ slightly depending upon the purge gas. If the reading is in the low E-6 range then there is a partial clog in the MS transfer line, and sensitivity will be reduced. If the reading is in the mid-low E-7 range, then the transfer line is severely clogged and you will not get good MS results.



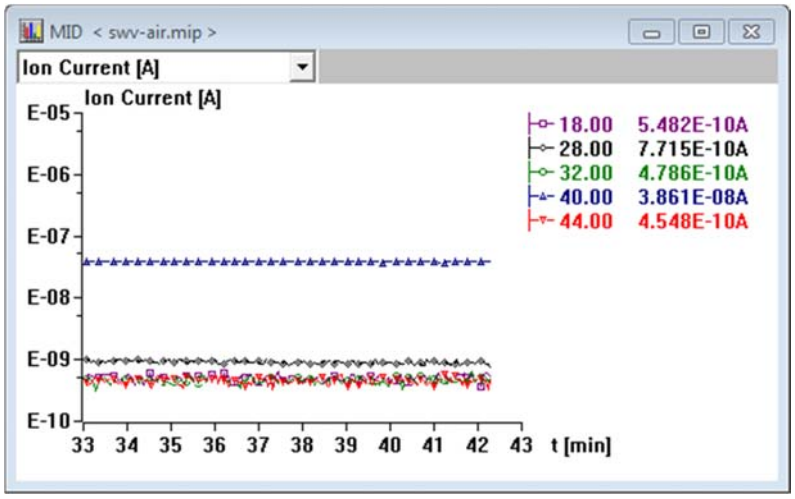
Second, start the Aeolos Measure program (not Measure+Sequencer!!). Choose the "MID Versus Time" measurement, and use the parameter set "swv-air.mip". This monitors ions representative of water (18), nitrogen (28), oxygen (32), argon (40), and carbon dioxide (44).



The results will depend upon the purge gas, and whether you do the test with the furnace raised or lowered. Two examples are shown below. In the first, argon is the purge gas, but the furnace had been in the raised position for quite a while. Consequently, strong MS signals are seen for m/z 28, 32, and 40. Note that the y-axis is a log scale. The most intense ion (m/z 28 in this case) has an ion current in the mid E-07 ampere range, and the signal for oxygen is about 1/5th, in the E-08 range. The argon signal at m/z 40 is decaying as more air enters the furnace, displacing the argon. The signals for water (18) and CO2 (44) are low, in the E-09 range.



In the second case, the furnace was closed and an "evac and fill" cycle was executed. See **"Using the Gas Delivery Manifold"** training guide. After the evac and fill cycle completes, only m/z 40 (argon) is large. All other "air" signals are about two orders of magnitude lower.



Testing IR Performance

First, determine if the majority of the gas flow is going through the light pipe. To do this, follow the exit tubing from the light pipe to the location of the filter assembly on the top-rear of the DSC/TGA unit. Detach the filter housing; it is a simple press fit through an o-ring, so just lift it out. Then attach it to the Gasmet gas flow meter. Be sure the connection on the back of the Gasmet is to the "nitrogen" port. The flow is measured on the upper scale and should be approximately the same as the sum of the P1, P2, and Pr flows. It will differ slightly if the purge gas is something other than nitrogen. In the example below, the total flow of 40 ml/min of argon measured as 60 ml/min on the nitrogen scale. If the flow is near zero then you know there is a gas leak or obstruction in the transfer line and/or light pipe path. This will likely result in reduced IR sensitivity.

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The second test is to start OPUS -> MEASURE, load the TGA parameter set, and run Check Signal. With pristine light pipe windows we see centerburst signals greater than 20,000 counts, but the windows begin to get contaminated quickly. As long as the signal is greater than 10,000 you should have no concerns regarding IR sensitivity. The system is still perfectly usable at 5,000 counts, but perhaps not for the most demanding measurements.