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Troubleshooting FID Noise and High Background

6890/6850 Gas Chromatograph

The purpose of this document is to outline a logical procedure for troubleshooting FID baseline problems, including noise, high background, drift or cycling.

OVERVIEW

The 6890/6850 FID uses a hydrogen flame to burn organic samples, resulting in increased ionization that is measured as a current in the 5 pa to 10^6 pa range. Normal FID background levels will be in the 5-20 pa range, with no sample present and the GC oven cool (<100 degrees C). For a FID noise specification, refer to section 600 of the 6890 Service Manual. The FID background level is due to presence of organic material in the carrier, makeup, FID air and H2 gases, and the amount of stationary phase that is coming off of the GC column. For successful GC work using FID, it is important to observe the requirements for gas purity, ***MINIMUM GAS PURITY*** should be ***99.9995%*** or greater. Also the FID temperature should be maintained at least 20 degrees C > the final oven temp of the GC analysis. The FID should never be operated less than 300 degrees C.

The gas delivery system must meet the following requirements:

-Stainless steel diaphragm tank regulators capable of sufficient supply pressure.

-All metal plumbing manifold comprised of clean 1/8" copper (Agilent Technologies P/N 5180-4196) or stainless steel tubing and swagelok fittings. (Many times "clean" tubing from other vendors has caused high FID background).

-The gas delivery system must be leak free.

-Gas traps are recommended for carrier and makeup gas supplies—minimum trap configuration should be:

-All metal, conditioned moisture trap, closest to the supply tank. (P/N 5060-9084)

-All glass, indicating O2 trap closest to the GC. (P/N 3150-0528)

Never use traps made of plastic or that use o-ring seals.

FID noise can be caused by many contributing factors, including:

1) Contamination in the gas supplies to the GC—(gas contamination usually yields a high background of ->20 pa).
2) Electrical current leakage in the FID interconnect or across the Teflon insulators that isolate the FID collector from ground.
3) Poor flame stability due to a partially plugged or contaminated FID jet assembly.
4) Mechanical noise due to loose or vibrating FID components—interconnect/spring, collector etc.
5) Electronic noise due to faulty or out of spec components on the FID electrometer or signal board.

NOTE: If the FID baseline has a periodic cycling component, it could be due to poor regulation and filtering of a house air compressor system, a defective tank regulator or a faulty detector EPC module.

To Troubleshoot high noise or high baseline:

1) Confirm the integrity of the gas supply, check gas purities and leak check the plumbing manifolds.

   Carrier/Makeup—80-100 psi

   Air 80 psi minimum

   H2 60 psi minimum

2) Evaluate the level of leakage current in the FID. This is the amount of current flowing in the FID electrometer with the flame extinguished. The FID should be at operating temperature (300 degrees C minimum). To perform the test, turn the FID off from the GC front panel or hand held controller. Allow the FID background to stabilize—it should immediately drop to 2-3 pa and slowly be moving towards 0 pa.

The output on the display should also be very stable at this point—not jumping more than +/- 0.1 pa at a time.

If the background stays above 5 pa or is very unstable, suspect the following:

1) The interconnect or spring—loose, contaminated, deformed. (the spring should never be touched with bare hands as this will cause leakage current.)

2) The Teflon insulators that electronically isolate the collector or the collector itself could be contaminated.

3) One of the electronic components.

If the leakage current test passes, proceed with the next test.

3) Eliminate the column/carrier as the source of noise/background. Remove the column from the FID and cap the fitting. (using a blank no hole ferrule or a paper clip inserted in the ferrule) Re-light the flame, allow it to stabilize and reevaluate the noise /background. If the FID background/noise becomes acceptable, the problem could be due to contaminated carrier gas or excessive column bleed. If the problem persists, continue the procedure—otherwise reevaluate with a known, good, well conditioned, thin film column (an uncoated retention gap is a good troubleshooting tool to isolate column bleed problems.)

4) Measure the FID flows with an independent measurement device.

Bubble meter or electronic flow meter. Use the FID flow adapter P/N 19301-60660.

The recommended flows for the FID are:

Column or Column + Makeup—30 ml/min

H2—Equal or > Column or Column+Makeup if using capillary column

(Optimum FID signal to noise performance is achieved at about a 1:1 ratio of H2 to inert gas)

Air—400 ml/min

There will no carrier flow, because the column is disconnected and the detector fitting plugged. Measure the H2, Air and Makeup gas flows independently by turning them on one at a time from the GC front panel or hand held controller. They should be within +/- 10% of the setpoint. If the flows are significantly off , the jet could be partially plugged, there could be a leak anywhere in the FID pneumatic system, or the detector EPC module could be defective. Remember that flow measurement from a soap film bubble meter is affected by ambient pressure. Flow modules are calibrated at Standard Temperature and Pressure. For accurate readings, use a true mass flow measurement device. Resolve any flow control problems and retest the FID noise.

5) Perform FID maintenance per the GC operator's manual.
   - Clean or replace the FID jet
   - Clean or replace the FID collector and Teflon insulators.
   - Inspect the underside of the brass castle assembly for rust or corrosion—replace if dirty.
   - When reassembling the FID make sure that all mechanical assemblies are tight and that the interconnect spring does not get deformed—it should be oriented into the channel on the outer perimeter of the FID collector.
   - Plug the FID column fitting to continue with the troubleshooting procedure.

6) Re-light the flame and bakeout the detector at 350 C for an hour.
7) Reevaluate the FID noise and background at normal FID and GC oven operating temperature.

If the noise and or background is still too high, the Air, H2 and Makeup gas purity is suspected. The FID can be operated with just Air and H2. Turn off the makeup gas and reevaluate the FID noise/background. If there is a significant drop in noise or baseline (>5pa drop in background) then the makeup gas purity is suspect. It is recommended at this point to replace traps in the Makeup Gas supply line. If the problem persists replace the moisture traps in the H2 and Air supply lines.

If the problem persists after gas purity issues are addressed, there could be a possible problem with the electronics. Call Agilent