How to Increase Your Gas Chromatography System Uptime

Troubleshooting your GC and Detectors with the Agilent ADM Flow Meter

Technical Overview

Introduction

Flow meters are an integral part of gas chromatography. It is a tool that can be used to verify the proper function of a GC as well as troubleshoot a problem. As with previous flow meters, the Agilent ADM Flow Meter has been developed to measure the volumetric flow rate of any noncorrosive gas, including mixtures of gases. Unlike those flow meters, which have to be sent off-site to be calibrated annually, the ADM Flow Meter has an easily swappable calibration cartridge. All you have to do is order a new NIST-certified cartridge, and you can replace the old one yourself, increasing uptime while saving money and time spent on shipping and paperwork.

Agilent Technologies

A built-in calibration timer automatically warns you when the calibration cartridge needs replacing. This minimizes the risk of volumetric measurement drift, and allows you to plan when you need to order a new calibration cartridge.

Easily Power Your ADM Flow Meter

The ADM Flow Meter can be powered by three AA batteries, or a USB connector, ensuring that your flow meter is ready to use when you need it.

Update the Firmware in Your Own Laboratory

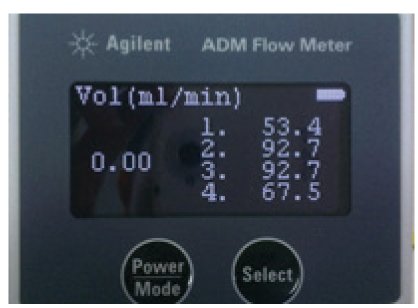
The USB connector also enables you to download new features and capabilities directly to your flow meter, and upgrade the firmware when it is needed, allowing your flow meter to always be up to date.

Large OLED Screen

The OLED screen on the ADM Flow Meter is larger than previous versions, making it easier to read no matter what lighting you have in your laboratory. Additionally, the kickstand allows you to use your flow meter hands-free.

Record up to Four Flows

When verifying flows while troubleshooting your GC, you no longer have to worry about finding a piece of paper to record your values. The ADM Flow Meter can record up to four flows in volumetric and mass flow mode, making it easier and faster for you to troubleshoot your GC.



New Adaptors for the ADM Flow Meter

In addition to the traditional flow meter adapters, there is a new option, an 1/8" Swagelok adaptor. You do not have to create homemade adapters from parts found around the lab. You can have confidence that your gas flow measurement has been accurately measured.

The streamlined look of the brass adapters has been made to connect perfectly to anywhere you want to measure flow. You no longer need to waste time stretching tubing to make an adaptor fit. These are designed to fit, ensuring that the flow you measure is correct every time.

Adapter	Part number	Adapter image
FID	5190-9576	
TCD	5190-9578	
Split Vent	5190-9580	
NPD	5190-9577	
ECD	5190-9579	

Determining the Location of a Blockage (Troubleshooting your GC)

Issues with gas flow can appear in a few ways, and it can be difficult to determine the location of the problem without the help of a flow meter. The appearance of shifting peaks or a noisy baseline are both indications of a disturbance in carrier gas flow, but by using the ADM Flow Meter you can locate the location of the problem faster, increasing your instrument uptime, and consequently your productivity.

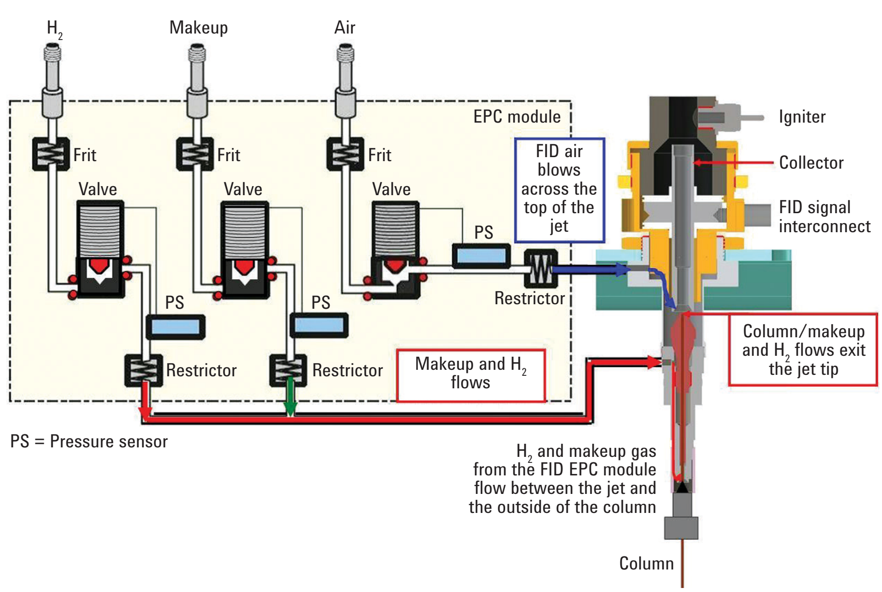
Flow not ready

One of the most frustrating errors an analyst deals with is when they are ready to run an analysis, but the GC is not. A GC reading *Flow not Ready* is one of those error messages, and can be caused by multiple factors. The most likely cause of this error is due to a clog or restriction in the line somewhere around the inlet. This can be caused by a buildup of residue in the split vent line over a long period of time, or an injection of a condensed sample.

To verify a restriction in the split vent, connect the 1/8″ swagelok adaptor to the ADM Flow Meter, and measure the flow on the split vent and the septum purge vent. If the flow meter differs by ±10 %, there is a restriction in the line, and it is necessary to replace the split vent trap. If the split vent was clogged due to a condensed sample, then it is important to verify that the gold seal is also free of restriction. Additionally, if it is important to the analysis to continue injecting condensed samples, it is important to ensure that you are using the right liner.

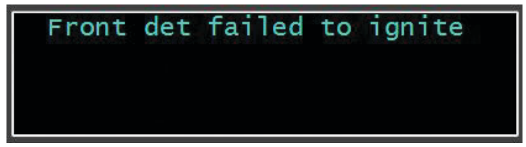
Flame ionization detectors (FIDs)

Effluent from the column mixes with hydrogen and air, and are pyrolyzed by a flame. Upon combustion, most organic compounds produce ions that have the ability to conduct electric current through a flame. It is this conduction of electricity that produces an increased signal output when an organic compound passes through the flame, and creates a peak. FID is very sensitive for hydrocarbons, and can detect quantities as low as 10^{-13} g/sec.



Detector Ignition Problems

One of the most common problems people encounter with FIDs related to gas flow, is when the FID fails to ignite, or blows out on injection.



What happens during an ignition sequence

During a normal ignition sequence, the detector will wait for the temperature of the detector to stabilize. Once the temperature has stabilized at the set point, the hydrogen flow will turn on, and the ignitor glow plug will turn on. Next, the air flow will turn on and slowly ramp up to the set point. You will hear a noticeable popping noise as the flame ignites. The output of this signal is compared to the Lit Offset, and if the value of the detector is greater than the offset (Lit-offset), the makeup gas will be turned on. If the output remains above the Lit Offset value with makeup gas flowing, it declares the flame ON (lit). If the output is less than the Lit Offset, it declares the flame OFF (not lit), and will try to ignite two more times before giving the error message, *Front or Back Detector Failed to Ignite*.

To determine the cause of the *Failure to Ignite* error, it is necessary to verify gas flows on the detector.

1. Decrease the oven to ambient temperatures (25 to 30 °C).
2. Turn off the flame.
3. Turn off the carrier gas.
4. Turn off all flows to the detector.
5. Connect your ADM Flow Meter to the top of the FID using the FID adaptor, and verify all flows by turning them on, one at a time. If the readout on the flow meter differs by ±10 %, there is most likely a plug in the FID jet, causing a restriction in the line.

Solution: Clean or replace the jet.

If all flows read correctly, check out the Lit Offset value. The default value for the Lit Offset is 2.0 Pa, and if the output is at or below this value the GC will declare the flame Off. In some very clean systems, with very low FID background, the Lit Offset may need to be set below 2.0 Pa to keep the FID from trying to re-ignite.

Sensitivity Problems

It can be difficult to determine the location of a problem, when there are issues with sensitivity. Low sensitivity could be due to a dirty jet or a restricted split vent. To verify the source of the decreased sensitivity, use the ADM Flow Meter to check the flows on the detector as well as split vent flow. Checking the split vent flow is easier with the 1/8″ Swagelok adaptor. If the test pressure on the split vent exceeds the method column head pressure, there may be a clog in the split vent trap.

Micro Electron Capture Detector (μECD)

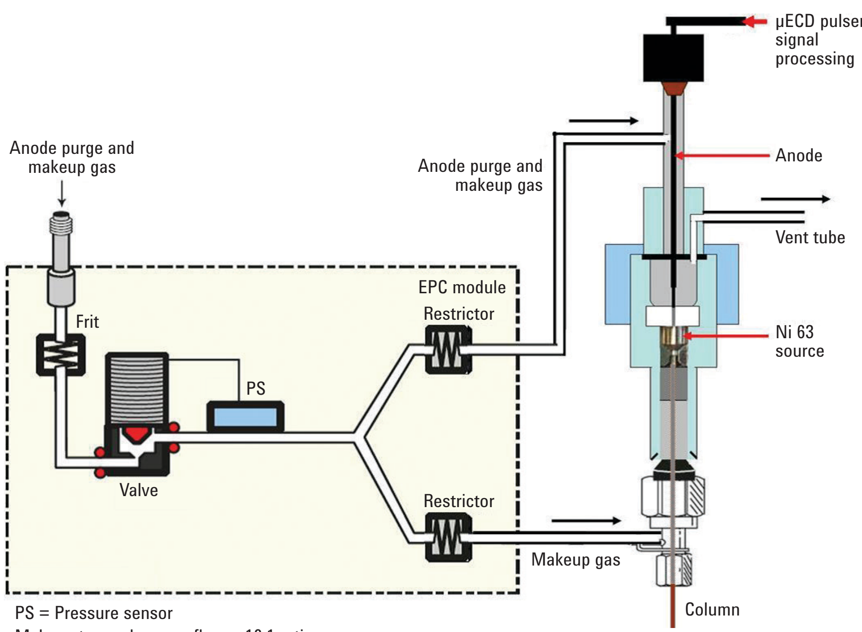
A μECD is commonly used in environmental sample testing due to its selectivity and sensitivity for halogenated compounds.

How it works

1. Effluent passes over a radioactive source (in most cases a nickel β emitter).

2. Electrons from the emitter ionize the carrier gas and makeup gas (usually nitrogen), and creates a detectable current that serves as a continuous background.

3. When electronegative organics and halogenated compounds pass over the emitter, they will grab the free electrons, causing the current to decrease. The magnitude of the decrease in current is proportional to the size of the peak.



PS = Pressure sensor
Makeup to anode purge flow = 10:1 ratio

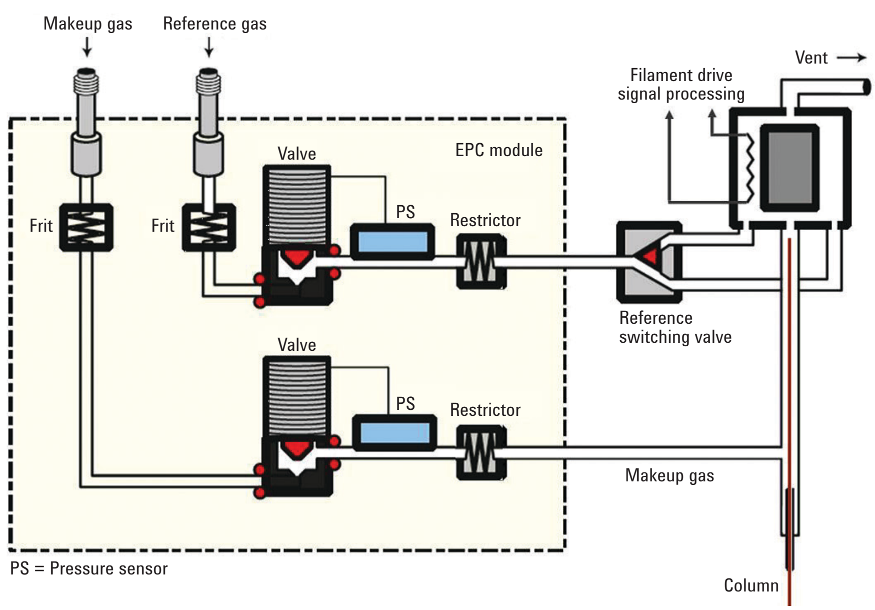
CAUTION: Detector disassembly or cleaning procedures other than thermal bakeout should be performed only by personnel trained and licensed to handle radioactive materials. Trace amounts of radioactive ^{63}Ni may be removed during these other procedures, causing possible hazardous exposure to radiation.

Due to the radioactive nature of the β emitter, it is important to check all other possible causes of a decrease in sensitivity, such as a clogged split vent or dirty liner, prior to suspecting the detector. ECDs are very stable over time, as long as they have a clean carrier gas.

Because only one makeup gas is used is to create a detectable current, any fluctuations in the flow will affect the sensitivity of the detector. To measure the flow of the makeup gas, connect the ADM Flow Meter to the ECD using the µECD adapter. If the readout on the flow meter differs by ±10 %, there may be an issue with your makeup gas line. Verify when the air and moisture traps were changed, and ensure that you are using clean nitrogen as a makeup gas. The ECD requires ≥99.9995 % pure carrier gas, so even the smallest bit of contamination can cause a decrease in sensitivity.

Thermal Conductivity Detector (TCD)

A TCD (also known as a Katharometer) uses a nondestructive detection technique. It is ideal for all compounds, making it an excellent general purpose detector. Inside the detector, a filament-thin platinum or gold wire is heated electrically. The detector contains a switching valve that rapidly switches between two gas flows: a reference gas, which serves as a background; and a makeup gas that combines with the column effluent. The thermal conductivity of each gas is measured and compared, and analytes within the makeup gas stream generate differences in thermal conductivity, which are measured and quantified.

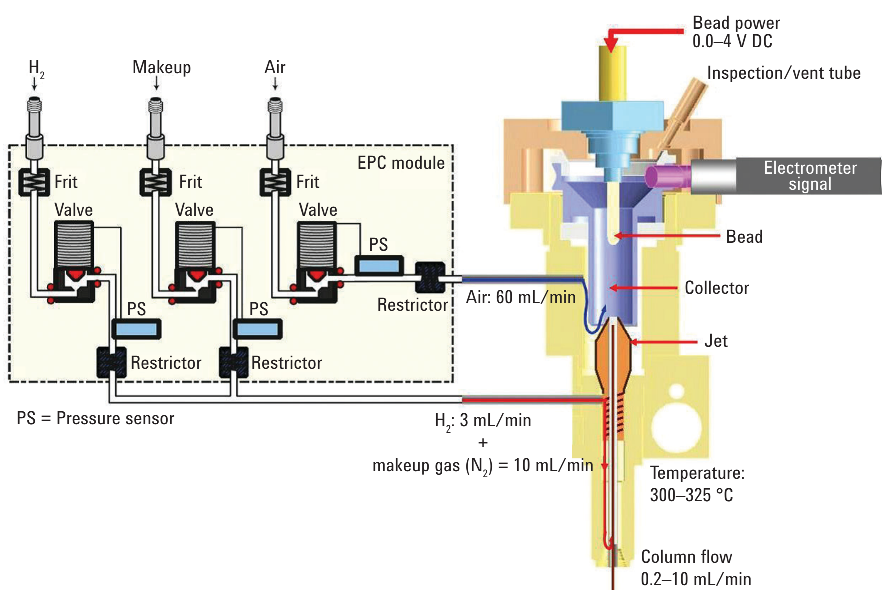


Due to the nature of the TCD, comparing the resistance of a reference gas to the resistance of a makeup gas in combination with an effluent, fluctuations in gas flow rate or leaks will affect the sensitivity as well as the chromatogram baseline. Air leaks in the TCD system can manifest chromatographically as a negative dip on the tail of a peak or an unstable baseline. To verify the flow, connect the AMD Flow Meter to the TCD using the TCD adapter, and measure each flow individually. If there is greater than 10 % difference on the readout, there may be a leak in the gas line.

Nitrogen Phosphorous Detector (NPD)

An NPD is a type of thermionic-specific detector, in which analytes are ionized by thermal energy, and is selective for nitrogen- and phosphorous-containing compounds. A hydrogen/air mixture is heated by a rubidium or cesium bead to form a cold plasma. This plasma can range from 600 °C to 800 °C. Electrons are ejected from this plasma, and flow between an anode and a cathode. Similar to an FID, the effluent mixes with hydrogen, and is exposed to plasma. A heated ceramic bead coated with an alkali metal facilitates ionization. When nitrogen- or phosphorous-containing analytes are present, they interact with the heated surface, causing a measured increase in current.

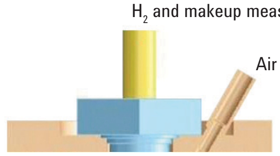
This detector is 50 times more sensitive for nitrogen-containing compounds, and 500 times more sensitive for phosphorous-containing compounds than FID.



Measuring your flow on an NPD

A plugged NPD jet will cause the NPD to have no output. To determine if the jet is clogged, it is important to measure all flow rates on the NPD. When measuring your flow on an NPD, follow the proper procedure, due to potential gas leakage around the ceramic insulators that could cause errors in measurement.

1. Set the bead voltage to zero.
2. Cool the detector to ambient temperature (25 to 30 °C).
3. Remove the bead.
4. Turn of all flows, and measure them one at a time using the NPD adapter.
   - **Measuring air**
     Air can only be measured from the inspection/vent tube.
   - **Measuring H_2 and makeup gas**
     Insert the NPD adapter directly into the collector.



If the readout on the flow meter differs by ±10 %, most likely there is a plug in the NPD jet, causing a restriction in the line.

For more Information

For more information on the new ADM Flow Meter, visit our website at www.agilent.com/chem/admflowmeter

www.agilent.com/chem

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