

Cleaning Flame Ionization Detectors: When and How

Noisy chromatograms, random spikes, and poor detector sensitivity are symptoms of a dirty FID — a common problem in gas chromatography. You will consistently obtain better chromatograms and reduce instrument down-time if you keep the FID clean. This bulletin describes methods of troubleshooting noise to confirm whether the source is a dirty FID, methods of cleaning FIDs, and ways to reduce contamination in the future.

Key Words:

- gas chromatograph maintenance ● flame ionization
- detector maintenance

The most common source of contamination in a flame ionization detector (FID) is bleed from silicone stationary phases and silylating reagents, which combust in the FID and produce silica. When deposited on surfaces within the detector, this white powder causes noisy chromatograms, random spikes, and poor detector sensitivity (Figure A). The use of carbon disulfide or hydrocarbons as solvents creates other forms of detector contamination.

Is the Detector Really Your Problem?

Before you shut down your instrument and clean your detector, it is wise to confirm that the problem is detector-related, rather than related to some other component of your system. The few, simple procedures described here can eliminate other possibilities as the source of the problem.

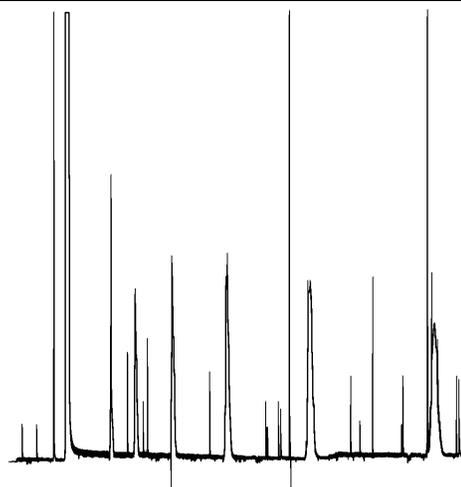
Carrier Gas and Stationary Phase

Seal the detector inlet in the oven with a Swagelok® plug and ignite the detector. If the chromatogram noise disappears, then the source of the problem is contaminants in the carrier gas or bleed from the chromatography column, not a dirty FID. To prevent this problem, you should always use a carrier gas purification system and columns prepared from chromatography-quality stationary phases.

Hydrogen and Air Systems

A problem in the hydrogen or compressed air delivery system can be a source of noise. Measure the flow rates for both gases (refer to your instrument manual). An incorrect flow rate in either source can cause noise, lack of sensitivity, and/or difficulty when igniting the flame. Also check the connections on both systems. An electronic gas leak detector, such as one of the GOW-MAC® detectors from Supelco, will quickly and easily pinpoint gas leaks via differences in thermal conductivity. If you do not find a major leak, check the gas pressure at the cylinders. If the pressure is less than 500psig, replace the cylinder.

Figure A. A Noisy Chromatogram, Caused by a Dirty FID



796-0593

A contaminated cylinder of gas could be the source of the problem, especially if the noise appeared several hours after you changed a cylinder. Check each cylinder for contaminants and replace if necessary. To eliminate the problem of contaminated air, we recommend using a zero air generator (see page 3).

Electrical System

Electrical noise can cause symptoms similar to a dirty FID. A poor connection due to oxidized contacts will act as a small capacitor and cause spikes and/or loss of sensitivity. If your FID has clip contacts to the collector or flame jet, we recommend you clean them periodically with emery cloth.

Electrical devices located near your chromatograph can interfere with the instrument's power supply and cause intermittent or random spikes. To isolate this source of noise, disconnect the electrometer cable(s) from the FID. If noise persists, it is coming from the electrical system.

In a two-detector system, electrical noise will appear on both detectors. If noise is present on only one channel, the electrometer or electrometer cable(s) may be the problem. Switch the cables at the electrometer and FID. If the noise now appears on the channel that was previously noise-free, replace the cable supplying the noise. If the noise does not change channels, check the electrometer on the malfunctioning channel by removing the electrometer cable at the FID, resting the cable connector on a surface to prevent contact with the metal frame of the GC. If the noise continues, the electrometer should be serviced. If the noise is eliminated, examine the hydrogen and oxidant systems.

Before Cleaning

Before you disassemble the detector, we suggest you take the following precautions:

- *Think Safety!* Disconnect the power to the detector, and be sure the collector assembly is cool before you begin.
- *Be Prepared!* Have spare detector parts available, as recommended in your instrument manual. Some detectors contain ceramic parts which break easily; and spare parts can minimize downtime should breakage occur.
- *Pay Attention!* Carefully note the distance from the collector assembly to the flame jet. On some detectors this distance is not fixed, and if you inadvertently alter the distance when you reassemble the detector, you can drastically change the FID response. The flame may be difficult to ignite and noise can occur.

How to Clean an FID

To properly clean an FID, you must clean the collector assembly, the jets, the Teflon® or ceramic insulators, and the housing. If the detector has been cleaned recently, a light coat of silica or a single silica flake could be causing the trouble, and only a light cleaning is required. If the detector has not been cleaned recently, a thorough cleaning probably is necessary.

Light Cleaning

There are two ways of removing light coatings of dirt. Both methods are simple and should be attempted before costly instrument downtime is incurred.

Freon® TF Injection: A light coat of silica can be removed by injecting several microliters of Freon TF (Detector Cleaner No. 1, see Ordering Information) into the column while the detector is lit. The Freon is combusted by the flame, producing hydrofluoric acid (HF). The HF converts the silica to a volatile fluoride, cleaning the detector in the process. Freon TF acts best when used on a regular basis, to prevent the buildup of silica, rather than as a cure for a very dirty detector.

Scrubbing: Disassemble the detector and scrub the contamination from the components. The brass wire brushes in our detector cleaning kits (see Ordering Information) will not scratch metal or ceramic parts; use a nylon brush on Teflon parts.

1. Disconnect the power to the detector, and be sure the collector assembly is cool.
2. Remove the collector assembly and brush the collector to remove the deposits.
3. Clean the jets, including the bore, using a brass toothbrush and a fine wire, such as a syringe needle cleaning wire.
4. Clean the electrical contacts, using a fine emery cloth. (Be careful not to bend the contacts.)

Thorough Cleaning

If the light cleaning methods do not adequately clean your FID, a more thorough cleaning is required. Disconnect the power to the detector, allow the detector to cool, then disassemble it.

1. Fill the basin in an ultrasonic cleaning device with a detergent that will effectively remove silica and other contamination from the FID (e.g., a 10:1 water solution of Detector Cleaner No. 2, a surfactant which is especially effective in removing heavy deposits of silica). Immerse the FID parts, except the electrical contacts, and sonicate for 2 hours. You can brush

the collector assembly and jets (with nylon or brass brushes) with the cleaning solution during the ultrasonic treatment. After the treatment, rinse the parts with distilled water to remove the detergent, then rinse with acetone to remove the water. Use a fine emery cloth to clean the electrical contacts.

2. Ceramic parts of an FID are best cleaned with aqua regia, a 1:~3 mixture of concentrated nitric and hydrochloric acids, at ambient or mildly elevated temperature. Before treatment, remove all metal and rubber from the ceramic parts – aqua regia will attack these. Place the ceramic parts in a beaker half-filled with aqua regia for one hour, then rinse with water and acetone as in step 1.

Exercise extreme caution – aqua regia is extremely corrosive.

3. After you have cleaned all parts of the detector, check all O-rings and replace them if necessary. Worn-out O-rings will cause gas leaks, which can produce detector noise or an increase in detector contamination.

Reassemble the FID, light the flame, and allow the detector temperature to equilibrate at 10°C–50°C higher than the column will reach during typical operation. This will reduce the amount of phase condensing onto the detector parts. Do not exceed the maximum temperature limit of the stationary phase – many columns fit far enough into the detector to expose the phase to these elevated temperatures. Set the proper flow rates for hydrogen and compressed air (refer to the instrument manual), and ignite the flame. Turn on the electrometer and allow a few minutes for warmup. The flame should now be stable and noise-free.

Reducing Detector Noise and Contamination

Conditioning

Most detector noise and contamination is the result of column bleed. The amount of bleed is greatest when the column is initially conditioned. Your detector will remain clean longer if you condition a new column *before* connecting it to the detector. By-products eluted during conditioning, potentially harmful to the FID, are voided into the oven.

Connect the column inlet to the injector as usual. Place a restrictor at the column exit to prevent back diffusion of air into the column (exposure of a heated column to air can destroy the liquid phase). Purge the column with carrier gas at room temperature for a few hours before you begin the temperature program. *Do not* allow a combustible carrier gas such as hydrogen, methane, etc. to exit the column into the oven. Pipe these materials out of the oven and into a hood. (Be sure to attach a restrictor to the outlet of the pipeline in the hood.) Consult the column manufacturer for conditioning details, i.e., duration and temperature of conditioning. *Do not* routinely condition new columns at the maximum temperature limit of the stationary phase – this will reduce column life.

Connecting a well-conditioned column to a clean FID should produce good sensitivity. If detector stability quickly degenerates, you should evaluate the quality of your stationary phase and carrier gas.

Stationary Phase

Use GC quality stationary phases whenever possible — they are purified to remove lower molecular weight components. Technical grade materials will bleed more than GC quality materials.

Carrier Gas

Moisture and oxygen in the carrier gas will cause stationary phase to deteriorate and bleed. Use chromatography-quality gases, and periodically monitor the gas system for leaks, which might allow atmospheric oxygen and water to enter the column. For details on carrier gas purification, ask for **Bulletin 848** and **Bulletin 918**.

Septa

Frequently check the septum for leaks. A leaking septum can allow oxygen and water to enter the carrier gas and cause the stationary phase to deteriorate and bleed. To check for leaks without contaminating the septum (and subsequent samples) with liquid leak detectors, use an electronic GOW-MAC leak detector (see Ordering Information). If you run your instrument frequently, we recommend you change the septum daily. If you change the septum at the end of the work day you can condition the new septum overnight. For septa-related information and troubleshooting hints, request **Application Note 82**.

Ordering Information:

Packard Zero Air Generators



994-0071

Reduce total hydrocarbons to less than 0.1ppm, stabilizing baselines and improving detection

CE approved – UL and CSA listed – IEC 1010 certified

These Packard zero air generators produce ultra-high purity (UHP) air from a standard compressed air supply – at continuous flow rates up to 3500cc/min, at pressures up to 125psig. We recommend using a zero air generator with flame ionization detectors – the resulting noise reduction and improved baseline stability allow lower detection limits, increasing the sensitivity of your analyses.

The Packard system consists of three stages: a 0.5µm coalescing inlet filter removes particles, oil, and water, a heated catalyst removes hydrocarbons, and a 0.01µm cellulose fiber outlet filter removes residual particulate material from the product air stream. Maintenance is minimal – just clean the inlet and outlet filters every 6 months and change them every 2 years. For replacement filters, refer to our catalog.

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GOW-MAC – GOW-MAC Instrument Co.
Snoop – Nupro Company
Supelco – Sigma-Aldrich Co.
Swagelok – Crawford Fitting Co.

Specifications

Product Purity:	< 0.1ppm total hydrocarbons (as methane)
Inlet Air Pressure:	2-125psig
Delivery Pressure:	125psig
Outlet:	1/8" Swagelok
Pressure Drop at Maximum Flow Rate:	5psig
Flow Rate Pressure Stabilization Time:	<2 minutes
Maximum Air Flow Rate:	<i>Models 1000, 1001</i> 1000cc/min (serves 2-3 FIDs) <i>Models 3500, 3501</i> 3500cc/min (serves 10 FIDs)
Dimensions:	<i>Models 1000, 1001</i> 25 x 14.7 x 30.8cm (H x W x D) <i>Models 3500, 3501</i> 29.2 x 17.8 x 39.4cm
Weight:	<i>Models 1000, 1001</i> 11lbs/5kg <i>Models 3500, 3501</i> 20lbs/9.1kg

Electrical Requirements

<i>Models 1000, 3500 (110 VAC)</i>
<i>100-125VAC, 60Hz, 100W, 1.0A (1.5A fuse)</i>
<i>Models 1001, 3500 (220VAC)</i>
<i>200-250VAC, 50 Hz, 100W, 0.5A (0.8A fuse)</i>

One-Year Parts and Labor Warranty from Packard.

Description	Cat. No.
Packard Zero Air Generators	
Model 1000 (110VAC)	22824
Model 3500 (110VAC)	27625-U
Model 1001 (220VAC)	22830-U
Model 3501 (220VAC)	27626-U

Activated Charcoal Trap

If halocarbons or sulfur-containing compounds might be present in the source air, we recommend using a Supelpure-HC trap to avoid contaminating the catalyst in the zero air generator.

Supelpure-HC Trap

1/8" fittings	22445-U
1/4" fittings	22446
Replacement Charcoal, 400cc	22451

Description	Cat. No.
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Detector Cleaner No. 1

Detector Cleaner No. 1, a halocarbon liquid (Freon TF), cleans your FID in place. Just inject a few microliters into a column that is connected to the lighted detector. The combustion products of the cleaner remove silica deposits from the detector electrodes. Recommended for preventive cleaning. In liquid form, Detector Cleaner No. 1 is useful for removing greases and oils from glassware, syringes, etc.

Detector Cleaner No. 1, 100mL **33000-U**

Detector Cleaner No. 2

For removing heavy silica deposits. Immerse silica-coated detector parts in a 1:10 mixture of this surfactant cleaning solution in water, preferably in an ultrasonic bath. The concentrate is an innocuous aqueous solution.

Detector Cleaner No. 2, 100mL **22662**

Detector Cleaning Kit

Consists of two brass wire brushes, a brass tube brush for your specific injection port, a brass toothbrush, and a piece of fine emery cloth.



910-0030

For Hewlett-Packard Models (collector \cong 0.145" ID) **22403**

For Perkin Elmer Sigma Series Models (collector \cong 0.187" ID) **22405**

For Varian Models (collector \cong 0.235" ID) **22404**

Needle and Jet Cleaning Kit

10 wires, each in 5 diameters, plus syringe cleaning solution.



910-0031

Needle Cleaning Kit **21578**

Description	Cat. No.
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Molecular Sieve 5A Water Vapor Traps

These traps efficiently remove water and heavy hydrocarbons from compressed air, electrolytically produced hydrogen, house nitrogen, or other gases with high moisture or hydrocarbon content. 200cc traps are 2' x 1" (61 x 2.5cm), 750cc traps are 18" x 2 3/8" (45.7 x 6cm). The extended bed length ensures prolonged contact between the gas and the adsorbent. Use the smaller tubes with up to 5 GCs, the larger traps with 6-20 instruments.



E000008

Molecular Sieve 5A Water Vapor Traps

200cc, 1/8" Fittings	20619
200cc, 1/4" Fittings	20618
750cc, 1/4" Fittings	23991
750cc, 1/2" Fittings	23992
S-Trap, 1/8" Fittings	503118
Molecular Sieve 5A Refill, 1/2lb./0.22kg	20298
Mounting Clip for 200cc Traps	503231
Mounting Clip for 750cc Traps	24983
Mounting Clip for S-Trap	502901

GOW-MAC Gas Leak Detectors



995-0110

GOW-MAC Gas Leak Detectors pinpoint leaks by detecting gases that have a thermal conductivity value different from that of air. This clean, efficient method of leak detection completely eliminates the risk of system contamination that can result from using soap solution. These easy-to-use units feature probes designed to reach difficult and confined locations. In the deluxe model, an audible tone alerts you to a leak. An LED bar graph on the handheld mini model visually alerts you to leaks.

Deluxe Model 21-250	22409
Mini Model 21-050	22807
110VAC/60Hz	22808
220VAC/50Hz	22809
Carrying Case for Mini Model	22809

BULLETIN 783

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