# GC

## TROUBLESHOOTING GUIDE

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GC Troubleshooting Guide

# INDEX

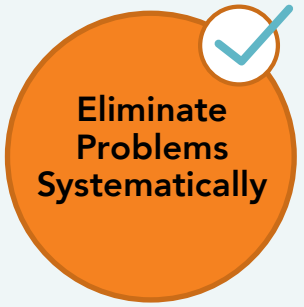
## Goals & Approaches

An important first step in troubleshooting is understanding the problem. This is best approached systematically; once you have a good understanding of what is causing the problem, it will be easier to implement a logical solution. Understanding the problem can also allow you to alter analysis or maintenance habits to avoid the problem in the future. Prevention is usually the most cost effective solution!

### Common Troubleshooting Approaches


Changing everything relating to the problem may allow a remedy, but won't allow you to fully understand the solution. In the long run, this may be more costly than other approaches.


Narrowing down or eliminating parts of the process may give a good starting point to determine the cause of the problem, but does not necessarily allow you to solve or prevent the problem in the future.




A systematic approach includes checking the chromatographic system from one end to the other, isolating the problem, learning what went wrong, solving the problem, and then preventing it. This is the recommended approach!

### Problem Prevention

Many GC problems can be prevented if the column and system are maintained routinely. *Problem Prevention (see p. 32)* outlines maintenance practices that will reduce the frequency of common issues. These suggestions should be modified to fit your GC column and instrument, and then made a regular part of your laboratory routine.

## Troubleshooting Tools

### What To Have On Hand

Have your instrument manual and these diagnostic tools at hand:

- Flow meter with a range of 10 to 500 mL/minute
- New syringes
- Non-retained, detectable compound such as methane or propane
- Septa, ferrules, inlet liners, and other consumables
- Electronic leak detector
- Reference sample
- Reference column with known performance





## Tips For Effective Troubleshooting

- When troubleshooting, remember to look at recent changes in the system, especially if the system was working previously. Was there something that changed and may be causing the problems now? Can you undo the changes and go back to the original performance?
- Try to isolate the problem to one specific cause to minimize the changes that have to be made to the system that may result in other malfunctions. This will also make it easier to prevent the problem in the future and shorten troubleshooting times if a similar problem does occur in the future.
- Remember to check every part of the process. Don't overlook the obvious. If you are not getting peaks for example, the makeup flow to the detector may be off or the syringe may be clogged. Has the sample preparation method been altered? Verify your samples on another instrument if possible.
- Keep good records of the troubleshooting process – closely observe and note operating parameters (temperatures, flow rates, columns used, etc.). Reliable system maintenance records (inlet liner changes, detector cleanings, etc.) are also important.

### For Additional Help

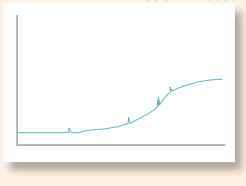
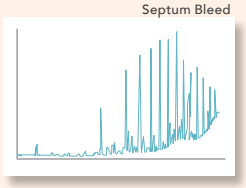
- The operator's and service manuals for the instrument should be consulted. These contain exploded diagrams, troubleshooting procedures for specific models, and part numbers to help you order replacement parts.
- Other people in the lab may have had experience solving a problem that is giving you trouble; they can be a helpful resource.
- The manufacturer of your instrument can help you. Most GC manufacturers offer free technical support to their customers. Phenomenex has experienced technical consultants who can assist you with almost any problem. We welcome your phone calls or emails!

# BASELINE PROBLEMS

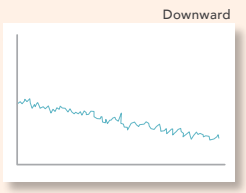
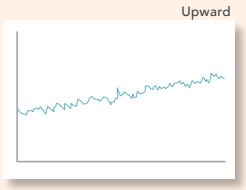


# BASELINE PROBLEMS

## Bleed

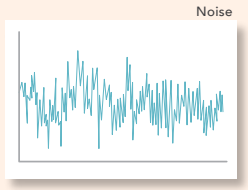
| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  Column Bleed | Improper column conditioning. | Properly condition the column. *See column installation.* |
| | Contaminated column. | There are several options:<br>– Trim the column<br>– Bake out the column<br>– Solvent rinse the column<br>– Replace the column |
| | Contaminated injector. | Perform inlet maintenance – clean the injector, replace the inlet liner, replace glass wool. *See inlet maintenance.* |
| | Leak in system causing column oxidation. | Check for leaks in the system. Tighten or replace connections; replace seals and filters. If column is severely damaged, replace. *See column installation.* |
|  Septum Bleed | Septum is not conditioned. | Condition septum prior to analysis or use pre-conditioned septum. Check septa temperature rating – should be sufficient to run at method temperatures. |
| | Septum core is present in the flow path. | Remove septum core from the inlet. Check septum nut and make sure it is not over tightened. Inspect injector syringe for bent or blunt tip and replace as necessary. |

## Drift Slow movement of the baseline in one direction (either up or down).

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  Downward | Downward drift for a few minutes is normal after installing a new column. | Increase the oven temperature to the maximum continuous operating temperature for the column. Maintain that temperature until a flat baseline is observed. If the detector signal continues to raise or does not drop in 10 minutes, immediately cool the column and check for leaks. *See column installation.* |
| | Unequilibrated detector or oven. | Allow sufficient time for (temperature) equilibration of the detector or oven. |
| | Downward drift is frequently due to the "bakeout" of contaminants from the detector or other parts of the GC. | Clean out contamination. *See detector maintenance.* |
|  Upward | Excessive damage to the stationary phase of the GC column. | Determine the cause of the damage. It may be due to impurities in the carrier gas or to excessive temperatures. Replace column. *See column installation.* |
| | Drift in gas flow rates. | Clean or replace flow or pressure regulator(s). Adjust pressure. *See column installation.* |

# BASELINE PROBLEMS

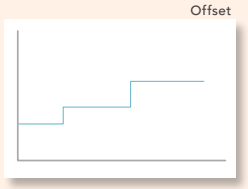
## Noise Rapid, random movement of the signal amplitude.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|   | The column may be inserted too far into the flame of an FID, NPD or FPD detector. | Reinstall the column. Be sure to insert the column into the detector exactly the correct distance specified in the instrument manual. *See column installation.* |
| | An air leak can result in noise in ECD and TCD detectors. | Eliminate the leak. |
| | Incorrect combustion gases or flow rates can generate noise in FID, NPD, or FPD detectors. | Be sure your gases are the proper grade, as well as clean and dry. Reset the flow rates of the gases to their proper values. |
| | Contaminated injector. | Clean injector. Replace inlet liner. Replace glass wool. *See injector maintenance.* |
| | Contaminated column. | Bake out the column.Cut off first 4 inches of column. Solvent rinse or replace column. *See column installation.* |
| | Drift in gas flow rates. | Clean and/or replace parts as necessary. *See detector maintenance.* |
| | Defective detector board. | Consult GC instrument manufacturer. |

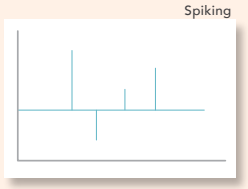



# BASELINE PROBLEMS

## Offset Sudden unexplained changes in baseline position.

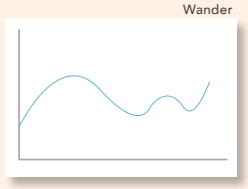
| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|   | Line voltage changes. | Monitor line voltage for correlation with offset. If correlation is found, install voltage regulator. |
| | Poor electrical connections. | Check electrical connections. Tighten any loose connections. Clean any dirty or corroded connections. |
| | Contaminated injector. | Clean injector. Replace inlet liner. Replace glass wool. *See injector maintenance.* |
| | Contaminated column. | Bake out the column. Cut off the first 4 inches of column. Solvent rinse or replace the column. *See column installation.* |
| | Column inserted too far into the flame of FID, NPD, or FPD detectors. | Reinstall the column. Be sure to insert the column into the detector exactly the correct distance specified in the instrument manual. *See column installation.* |
| | Contaminated detector. | Clean the detector. *See detector maintenance.* |
| | Gas generator cycle. | Baseline fluctuations can occur as the generator turns on and off. Add a tank with the appropriate volume after the generator to buffer any pressure changes. |

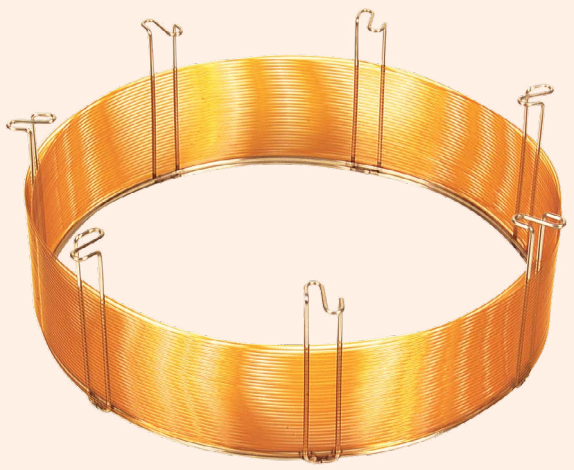
## Spiking Peaks with no width, either positive or negative.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|   | Electrical disturbances entering the chromatogram through power cables, even shielded cables. | Try to correlate spikes with events in equipment near the chromatogram. Periodicity is often a clue. Turn off equipment or move it. If necessary, install a voltage regulator. |
| | Particulate matter passing through the detector. | Clean the detector and eliminate the source of particles. A clean hydrogen flame is invisible. Most organic matter generates a yellow flame. *See detector maintenance.* |
| | Pressure may build up and gas may escape through a seal and thus reduce the pressure below the point where the escape occurs. If this is the cause, the frequency of spikes will be pressure-dependent. | Fix leaking seal. |
| | Loose, dirty, or corroded electrical connections in the detector or at connections along the signal path can cause spiking. | Check electrical connections. Tighten any loose connections. Clean any dirty or corroded connections. Replace badly corroded FID parts. |

# BASELINE PROBLEMS

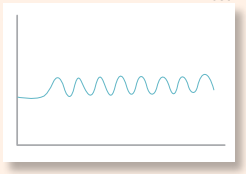
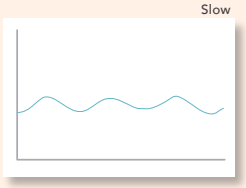
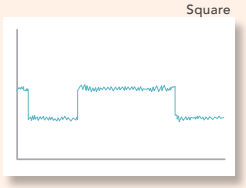
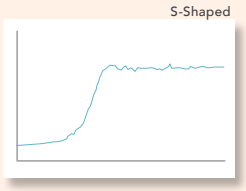
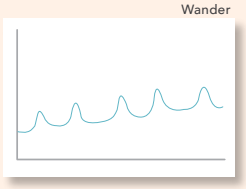
## Wander Low frequency noise.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|   | Baseline wandering may be caused by changes in environmental conditions such as temperature or line voltage. | Try to correlate the wandering with environmental parameters. If a correlation is observed, you will know what to do. Good luck. |
| | Inadequate temperature control. Check if variations can be correlated with changes in the baseline position. | Measure detector temperature. Check detector, if TCD is used. |
| | Wandering while using isothermal conditions may be due to contaminated carrier gas. | Change the carrier gas or the gas purification traps. |
| | Contaminated injector. | Clean injector. Replace inlet liner. Replace glass wool. *See injecor maintenance.* |
| | Contaminated column. | Bake out the column. Cut off first 4 inches of column. Solvent rinse or replace column. *See column installation.* |
| | Poor control of gas flow rates. | Clean or change flow controller(s). |



# BASELINE PROBLEMS

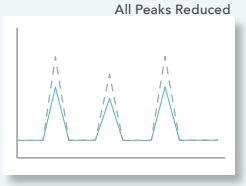
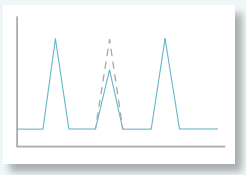
## Waves Baseline oscillations different from typical noise.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|   | Detector related problem. | Baking the detector at the maximum temperature (450 °C for FID) for 30 min to 1 hour may provide temporary relief. For a lasting solution, physically clean the detector. |
|   | Gas pressure fluctuations. | Storage tank pressure varies, causing dips in flow. Adding a dual stage regulator can minimize pressure fluctuations and help to alleviate the problem. |
|   | Unbalanced column-switching or gas-sampling valves (for TCD detectors). | Measure, check, and set flows accurately. |
| | AC power fluctuations; interference from other equipment lines. | Use a dedicated AC source with sufficient power. |
|   | Excessive column bleed. | Check max column temperature, re-condition column. If column is damaged beyond repair, replace column. |
| | Oxygen contamination is degrading the column phase. | Install or check oxygen traps. Check system for oxygen leaks. |
|   | Contamination during isothermal parts of a run. | The column is separating the contaminants (commonly siloxanes or hydrocarbons) as peaks. Changing samples, wash solvents, liner, gold seal, and sometimes the syringe may be required to eliminate the contamination. |

# PEAK SHAPE PROBLEMS

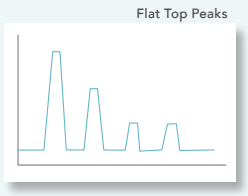
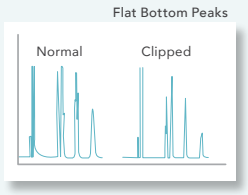
# PEAK SHAPE PROBLEMS

## Reduced Size Some or all peaks are reduced in size.

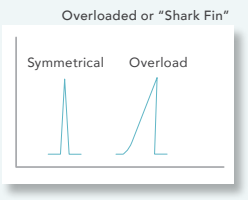
| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
| All Peaks Reduced  | Defective or plugged syringe. | Try a new or proven syringe. |
| | "Blown" septum or other massive leaks at the inlet or with carrier gas flow. Poor peak shapes usually result from bad leaks. | Find and fix leaks and adjust gas flow. *See column installation.* |
| | Purge flow or split ratio too high. | Adjust gas flow rates. |
| | Injector and/or column temperature too low for high molecular weight or low volatility samples. | Increase injector and/or column temperature(s). |
| | NPD detector may be coated with silicon dioxide due to column bleed or residual derivatization reagents. | Replace the active element. Avoid exposure to silicon containing compounds. |
| | NPD damage by loss of rubidium salt as a result of exposure to overheating, heating in the absence of clean gas flow, or humidity. | Replace the active element. Turn off detector whenever the gas flow is interrupted. Avoid overheating. Keep element warm (150 °C) when not in use. Use a desiccator for extended storage. |
| | For splitless injection, if the split vent is closed for too short a period of time or if the initial column temperature is too high, this may hinder refocusing of the sample. | Increase the time the split vent is closed. Decrease the initial column temperature or use a less volatile solvent so that the initial temperature is below the boiling point of the solvent. |
| | Detector-sample mismatch. | Ensure that the detector will respond to the analytes. |
| | Inadequate signal amplification. | Check output signal levels. |
| | Sample invalidity. | Check sample concentration and stability. |
| Some Peaks Reduced  | Activity in the inlet liner or column if the reduced peak is an active compound. | Clean or replace the inlet liner. Ensure an inert column is used. If necessary, replace the column. *See column installation.* |
| | Leak in the injector if the reduced peak is a more volatile compound. | Find or repair the leaks and adjust gas flow. |
| | Initial temperature too high for splitless or on-column injections. | Lower the initial column temperature. |
| | | Use a higher boiling solvent. |
| | Analytes are decomposing or breaking down for active or thermally labile compounds. | Check the integrity of the sample. |
| | | For thermal lability, lower the temperature and use on-column injection, a column with thinner stationary phase, a shorter column lengths, or a higher carrier gas flow rate. For active compounds, ensure an inert column is used. If necessary, replace the column. *See column installation.* |

# PEAK SHAPE PROBLEMS

## Clipped/Flat
Peaks are clipped and flat at either the top or the bottom.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Detector overload. The broad peaks may have a rounded top or even valleys in the top. | Reduce sample volume, dilute with solvent, or increase or add a split flow. |
| | Overload of the signal processing electronics. The peaks are clipped with flat tops. | Attenuate detector output reduce sample volume, or add a split flow |
|  | Detector, recorder, or integrator set too low; detector drifted below zero. | Correctly set zero. Reconnect leads from recorder and set zero of recorder baseline to ~5% of full scale. Check the integrator threshold and adjust accordingly.<br><br>Use an auto-zero function. |

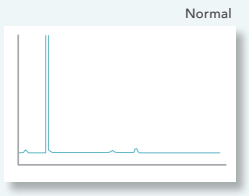
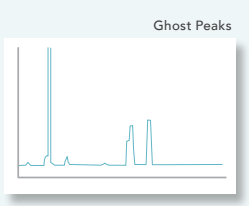
## Fronting
Moderate to severe asymmetry towards the front or left side of the peak.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Improper column installation. | Reinstall the column.<br>*See column installation.* |
| | Sample is condensing in the injector or column. | Check injector and oven temperature with an accurate thermometer. If accurate, increase temperature as necessary, but do not exceed the maximum temperature limit of the column. |
|  | Column is overloaded as a result of injection volume and split ratio. | Reduce the injection volume; add or increase split flow.<br><br>Use a column with greater capacity. Columns with larger diameter or thicker stationary phase coatings generally have larger sample capacities; however, resolution may be reduced. |
| | Polarity mismatch. | Polar compounds will have lower concentration capacity on a non-polar phase and vice-versa. Choose a phase with the appropriate polarity and selectivity for your sample. |

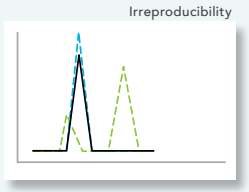


# PEAK SHAPE PROBLEMS

## Ghost Peaks
Peaks observed when no sample has been introduced into the system.

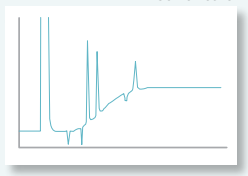
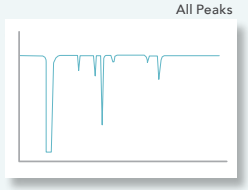
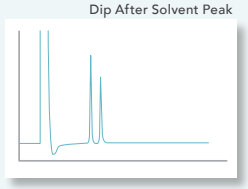
| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Remnants of previous samples in the inlet or column are most likely to occur when increasing inlet or column temperature(s). | Increase the final temperature and lengthen the run time to allow for the complete elution of previous samples. If ghost peaks continue to occur, clean the inlet.<br>*See injector maintenance.*<br><br>Condition the column at a higher temperature that is still lower than the maximum isothermal limit for the column. Cut 4 inches off the inlet end of the column and/or reverse it (end-for-end) before reconditioning. Solvent rinse or replace the column.<br>*See column installation.* |
|  | Sample expanded to exceed the volume of the injector liner. These vapors may come in contact with colder spots, such as the septum and gas inlets to the injector. Less volatile components may condense. These condensates may vaporize later and interfere with subsequent analyses, sometimes producing "ghost peaks". | Minimize backflash by using:<br>• a septum purge<br>• small injection volumes<br>• large inlet liners<br>• optimal injector temperatures<br>• pulsed pressure programming<br>• increased split flow |
| | Bleed from the septum or fragments of the septum lodged in the inlet or liner | Clean the inlet. Replace the inlet liner or glass wool, and septum.<br>*See injector maintenance.* |
| | Syringe contamination. | Replace the syringe. |

## Irreproducibility
Peak heights, areas, or retention times are inconsistent from injection to injection

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Inconsistent injection. | Develop a reproducible injection technique. Use autosampler or replace injection needle. |
| | Distorted peak shapes can adversely affect quantitative determinations. | Correct any problems that result in the distortion of peak shape. |
| | Baseline disturbances. | Disturbances in baseline are affecting peaks.<br>*See baseline problems.* |
| | Variations in GC operating parameters. | Standardize parameters. |

# PEAK SHAPE PROBLEMS

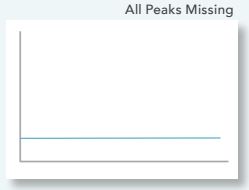
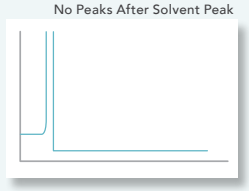
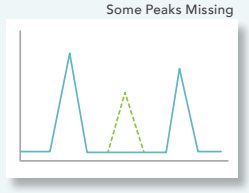
## Negative Peaks Some or all peaks dip below the baseline.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
| Some Peaks  | Detector overload in element-specific detectors such as ECD, NPD, FPD, etc. can produce both positive and negative peaks. | Have the compounds of interest arrive at the detector at a different time from the solvent or other compounds in high concentration. $H_2$ produces negative peaks with a TCD and helium carrier gas. |
| | Dirty ECD detector can give a negative peak after a positive one. | Clean or replace the ECD detector. *See detector maintenance.* |
| | Sample contaminants (hydrocarbons or other non-responders) are present when using ECD, PID, or NPD (thermoionic specific) detectors. | Improve sample preparation and cleanup methods prior to injection. |
| All Peaks  | Incorrect polarity of the recorder connections results in nearly all peaks being negative. | Reverse polarity of recorder connections. |
| | Recorder-integrator wires reversed. | Correct connections. |
| | Sample injected onto the wrong column for dual-column setups. | Reinject the sample onto the correct column. |
| Dip After Solvent Peak  | Detector contamination. | Clean or bake out the detector. *See detector maintenance.* |
| | Sample contamination. | For PID detectors, check that the sample has not been contaminated with methanol or water. If necessary, prepare a fresh sample. |
| | Often normal for NPD (thermoionic specific) detectors. | No correction necessary. |



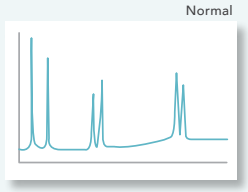
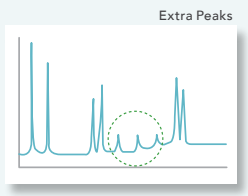
# PEAK SHAPE PROBLEMS

## No Peaks Some or all peaks are missing from the run.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
| All Peaks Missing  | Defective or clogged syringe. | Try a new or proven syringe. |
| | "Blown" septum or massive leaks at the inlet. | Find and fix leaks. |
| | Problems with carrier gas flow. | Adjust gas flow. |
| | Column may be broken or installed in the wrong detector or inlet. | If breakage is close to the beginning or end, cut off the short piece. Breakage in the middle can be repaired with a press-fit connector. For multiple breakages, replace or reinstall the column. *See column installation.* |
| | The detector is not functioning or is not connected to the recorder or integrator. | Ensure detector is working properly. (e.g. is the flame in a FID lit?) Check connection to the output device. |
| | Incorrect injector temperature:<br>• Injector too cold: sample is not vaporized.<br>• Injector too hot: thermally labile sample is decomposing. | Cold injector: check injector and oven temperature with an accurate thermometer. If accurate, increase temperature as necessary, but do not exceed the maximum temperature limit of the column. Inject the sample directly onto the column.<br>Hot injector: check injector and oven temperature with an accurate thermometer. If accurate, reduce temperature as necessary, ensuring compatibility with sample and column minimum limit. |
| No Peaks After Solvent Peak  | Sample volume is too high. | Inject less sample or use a higher split ratio. |
| | For FID detectors, the flame is blown out by the solvent peak. | Check the detector temperature. |
| | Carrier gas flow is too high. | Reduce the flow rate. |
| | Incorrect column temperature; column is too hot and sample is eluting in solvent peak. | Check oven temperature with an accurate thermometer. If accurate, reduce temperature as necessary, ensuring compatibility with sample and column minimum limit. |
| | Column cannot separate components from solvent. | Change solvent or column. |
| Some Peaks Missing  | Activity in the inlet liner or column if the missing peak is an active compound. | Clean or replace the inlet liner. Ensure an inert column is used. If necessary, replace the column. *See column installation.* |

# PEAK SHAPE PROBLEMS

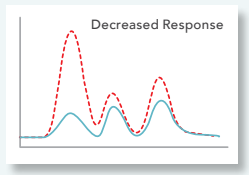
## Peaks Added There are more peaks than normal in the run.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Septum bleed (especially for runs with oven ramp). | Turn off the injector heater. If extra peaks disappear, choose a higher temperature rated septum or use a lower injection temperature. |
| | Carryover of sample or contaminants from previous runs. | Increase the analysis time prior to the next run or bake out the column between runs. |
|  | Contaminants in current sample or solvent. | Inject solvent by itself using a clean syringe. Switch to a higher quality solvent if extra peaks appear. If only solvent appears, run the solvent through any sample preparation methods, analyzing the solvent at each step of the process to identify the source of extra peaks. If only the solvent peak appears, the extra peaks are part of the sample. |
| | Impurities in carrier gas are eluting. | Install or check gas purifiers. Replace if necessary. Ensure only high-quality gases are used. |
| | Analytes are decomposing or breaking down for active or thermally labile compounds. | If compounds are thermally labile, lower the temperature and use on-column injection, a column with thinner stationary phase, a shorter column lengths, or a higher carrier gas flow rate. |
| | | If compounds are active, ensure an inert column is used. If necessary, replace the column. *See column installation.* |

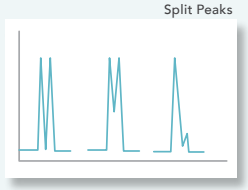


# PEAK SHAPE PROBLEMS

## Sensitivity Loss Some or all peaks are displaying decreased response.

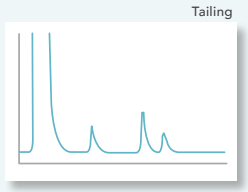
| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Contamination of column and/or liner can lead to loss of sensitivity for active compounds. | Clean liner. *See injector maintenance.* |
| | | Bake out the column. Solvent rinse or replace the column. *See column installation.* |
| | Injector leaks reduce the peak height of the most volatile components of a sample. | Find and fix any leaks. |
| | Initial column temperature is too high for splitless injection prevents refocusing of sample. This affects the more volatile components most. | Lower temperature below the boiling point of the solvent. Decrease the initial column temperature, or use a less volatile solvent. |
| | Inlet discrimination: injector temperature is too low. Later eluting and less volatile compounds have low response. | Increase the injection temperature or use on-column injection with direct connect liner. |
| | Issues with the sample. | Check the sample concentration, any sample preparation procedures, and shelf life. Prepare a fresh sample ensuring the proper concentration. |

## Split Peaks Peaks are duplicated or separated.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Poor (jerky or erratic) injection for manual injection. | Use smooth, steady plunger depression. Use autosampler. |
| | Bad column installation. | Reinstall column. *See column installation.* |
| | Solvent mismatch: polarity of the stationary phase does not match the polarity of the solvent. | Change solvents, use a very large split ratio, install a retention gap, or change the stationary phase. |
| | Wrong inlet liner is not vaporizing samples in one location. | Use a liner with glass wool in the middle of the liner if possible. |
| | Fluctuations in column temperature. | Repair temperature control system. |
| | Mixed sample solvent for splitless or on-column injections. | Use a single solvent. |
| | Improper use of "solvent effect" refocusing techniques result in broad, distorted peaks because solutes are not refocused into a narrow band near the beginning of the column. The solvent must form a compact, continuous flooded zone in the column. If the solvent does not wet the stationary phase sufficiently (as might be the case for methanol used with a nonpolar phase), the flood zone may be several meters long and not of uniform thickness. | Install a retention gap (5 meters of uncoated but deactivated column) ahead of the column to reduce or eliminate the problem. |
| | | Change solvent or GC column phase. |
| | | Use a very high split ratio. |

# PEAK SHAPE PROBLEMS

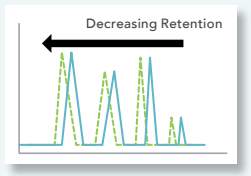
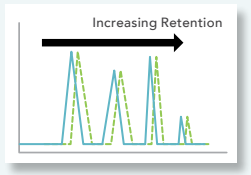
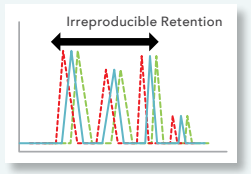
## Tailing Moderate to severe asymmetry towards the back or right side of the peak.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Contaminated inlet liner or column. | Clean or replace inlet liner. Bake out or replace the column. *See column installation.* |
| | Activity in the inlet liner or column if the missing peak is an active compound. | Clean or replace the inlet liner. Ensure an inert column is used. If necessary, replace the column. *See column installation.* |
| | Dead volume due to poorly installed liner or column. | Confirm by injecting inert peak methane; if it tails, column is not properly installed. Reinstall liner and column as necessary. *See column installation.* |
| | Ragged column end. | Score the tubing lightly with a ceramic scoring wafer before breaking it. Examine the end (a 20-power magnifying glass is recommended). If the break is not clean and the end square, cut the column again. Point the end down while breaking it, and while installing a nut and ferrule, to prevent fragments from entering the column. Reinstall the column. *See column installation.* |
| | Solvent-phase mismatch. | Change the stationary phase. Usually polar analytes tail on non-polar columns, or dirty columns. |
| | A cold region in the sample flow path. | Remove any cold zones in the flow path or check the MS transfer line trap. |
| | Column or inlet liner temperature is too low for tailing hydrocarbons. | Check injector and oven temperature with an accurate thermometer. If accurate, increase temperature as necessary, but do not exceed the maximum temperature limit of the column. |
| | Debris in the liner or column. | Clean or replace the liner. Cut 4 inches off the end of the column and reinstall it. *See column installation.* |
| | Injection takes too long. | Improve injection technique. |
| | Split ratio is too low. | Increase split ratio to at least 20:1. |
| | Overloading the inlet. | Decrease the sample volume or dilute the sample. |
| | Some types of compounds such as alcoholic amines, primary and secondary amines, and carboxylic acids tend to tail. | Try a more polar column. Derivatize the sample. |



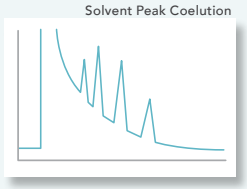
# PEAK SHAPE PROBLEMS

## Retention Time Shifts Peak retention times drift or move.

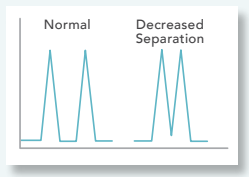
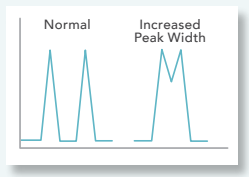
| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|  | Increase in column temperature. | Check GC oven temperature and adjust as necessary. Ensure run conditions do not exceed the minimum temperature limits of the column. |
| | Increase in gas flow rate (linear velocity). | Inject a detectable unretained sample such as methane to determine the linear gas velocity. Adjust gas pressure to obtain proper values for your analytical method. |
| | Change of solvent. | Use the same solvent for standards and samples. |
| | Significant loss of stationary phase due to column bleed. | Reduce oven temperature. Ensure run conditions do not exceed the maximum temperature limit of the column.<br>Replace the column if necessary. *See column installation.* |
|  | Leak in the injector. | Find the leak and fix it. Check the septum first. Change if necessary. |
| | Decrease in column temperature. | Check GC oven temperature and adjust as necessary. Ensure run conditions do not exceed the maximum temperature limits of the column. |
| | Decrease in gas flow rate (linear velocity). | Inject a detectable unretained sample such as methane to determine the linear gas velocity. Adjust gas pressure to obtain proper values for your analytical method. |
|  | Poor (jerky or erratic) injection for manual injection. | Use smooth, steady plunger depression. Use autosampler. |
| | Contaminated column. | Bake out the column. Cut 4 inches off the end of the column. Solvent rinse or replace the column. *See column installation.* |
| | Leak in the injector. | Find the leak and fix it. Check the septum first. Change if necessary. |
| | Near-empty carrier gas tank. | Check and replace the tank if necessary. |

# PEAK SHAPE PROBLEMS

## Solvent Peak Broad
The solvent peak is wide and coeluting with analyte peaks.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|   | Bad column installation. | Reinstall column. *See column installation.* |
| | Injector leak. | Find and fix leak. |
| | Injection volume too large. | Decrease sample injection volume or dilute to 1:10. |
| | Injection temperature too low. | Increase injection temperature so the entire sample is vaporized "instantly." An injection temperature higher than the temperature limit of the column will not damage the column. |
| | Split ratio is too low. | Increase split ratio. |
| | Column temperature too low. | Increase column temperature. Use a lower boiling solvent. |
| | Initial column temperature too high for splitless injection. | Decrease the initial column temperature.<br>Use a less volatile solvent so the initial column temperature is below the boiling point of the solvent. |
| | Purge time (splitless hold time) too long for splitless injection. | Use a shorter purge valve closed time. |

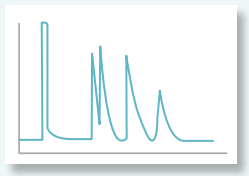
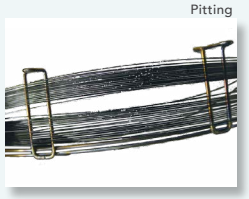
## Resolution Loss
Peaks begin to coelute or overlap.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|   | Change in column dimensions or stationary phase; excessive column trimming. | Differences in retention time or peak shape of other compounds will be apparent. Check the column phase and dimensions switch the column if necessary. |
| | Damage to column stationary phase. | This is usually indicated by excessive column bleed. Replace the column. *See column installation.* |
|   | Damage to column stationary phase. | This is usually indicated by excessive column bleed. Replace the column. *See column installation.* |
| | Injector problems. | Check for:<br>• leaks<br>• inappropriate temperature<br>• split ratio<br>• purge time<br>• dirty liner<br>• glass wool in liner |



# PEAK SHAPE PROBLEMS

## Performance Loss (Column)
The column deteriorates too quickly after installation.

| Symptom | Possible Cause | Suggested Remedy |
|---|---|---|
|   | Column too hot for too long. | Stay below limits specified for the column. Replace column. *See column installation.* |
| | Exposure to oxygen, particularly at elevated temperatures. | Find and fix any leaks. Be sure carrier gas is sufficiently pure. |
| | Chemical damage due to inorganic acids or bases. | Keep inorganic acids or bases out of column. Neutralize samples. |
| | Contamination of the column with nonvolatile materials. | Find and fix any leaks. Be sure carrier gas is sufficiently pure.<br>Keep inorganic acids or bases out of column. Neutralize samples.<br>Prevent nonvolatile materials from getting into column. For example, use a guard column or a Zebron™ column with Guardian™ integrated guard. |
|     | Broken column. | Replace column. *See column installation.*<br>Avoid damaging the polyimide coating on the column. Except when using Zebron Inferno™ GC columns, avoid temperatures above 370 °C; abrasion of columns (for example, do not install a column so that it touches the side of the oven, because vibration may then damage the polyimide coating); or excessive bending or twisting, which will damage this protective coating. Remember, even if the column does not break immediately, when the protective coating is damaged the column may break spontaneously later. |

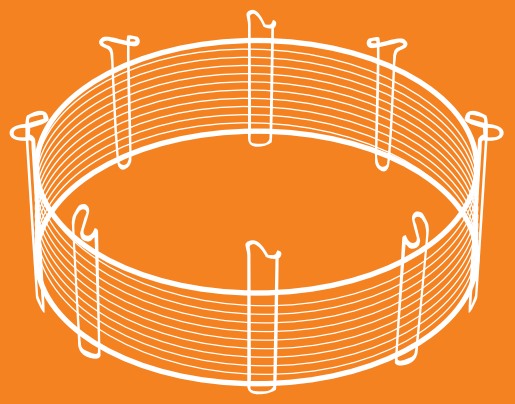
### A Note On Solvent Rinsing

Solvent rinsing (where a pressurized vial of solvent is forced through the column with 10 to 15 psi pressure) may remove most soluble contaminants and restore column performance as a last resort. In most cases, it is better to replace the column.

Use a series of solvents, starting with the most polar and finishing with the least polar. Include the injection solvent if practical. Successive solvents must be miscible with their predecessors. Begin with water followed by methanol for water-based samples (or aqueous extracts). Avoid halogenated solvents as a final rinse if you are using an ECD. Avoid acetonitrile as a final rinse if you are using an NPD. Methanol, followed by methylene chloride and then hexane, is a useful combination.

Each solvent should remain in the column for at least 10 minutes. There is no need to remove the previous solvent before introducing the next. After the last solvent has been removed, the column should be purged with pure carrier gas for 10 minutes before reinstallation. Program the oven temperature at 2°C/min until the normal conditioning temperature is reached, then condition the column as usual.

# COLUMN SELECTION PROBLEMS





COLUMN SELECTION PROBLEMS

## The Impact of Selectivity

Resolution between two analytes is mainly determined by the selectivity of the stationary phase. By increasing the resolution between two compounds, the total analysis time can often be reduced significantly!

**The Master Resolution Equation**

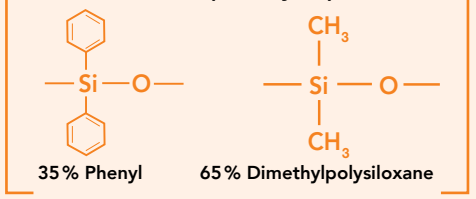
$$R_s = \left[\frac{\sqrt{N}}{4}\right] \times \left[\frac{\alpha - 1}{\alpha}\right] \times \left[\frac{k}{k+1}\right]$$

**Efficiency Term** **Selectivity Term** **Retention Term**

## Selectivity vs. Polarity

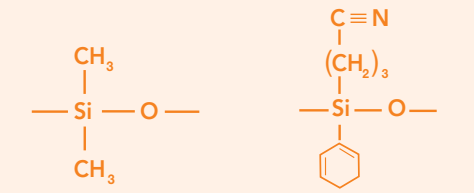
Column polarity and selectivity are often confused – polarity gives a general guideline for sample capacity and separation, which can affect peak shape and resolution. However, two columns may have similar polarity but show very different separation profiles because of differences in phase chemistry. Example: The ZB-1701 cyanopropyl group makes it very different from ZB-35 in terms of selectivity though polarity is similar.

**ZB-35 (Polarity: 18)**




35 % Phenyl 65 % Dimethylpolysiloxane

**ZB-1701 (Polarity: 19)**




86 % Dimethylpolysiloxane 14 % Cyanopropylphenyl

## The 3 Most Prevalent GC Interactions

### Dispersive Forces (Van der Waals Interactions)

- Weakest of all intermolecular forces and occurs between non-polar compounds
- Separation is based on boiling point (classic example – hydrocarbon separation in simulated distillation analysis)

### Dipole-Dipole Interactions

- Either permanently present or induced by analyte-stationary phase interactions
- Higher dipole-dipole interaction can help separate compounds with similar boiling points, but different chemical structures

### Hydrogen Bonding (Acid-Base Interactions)

- Can cause poor peak shape or irreversible binding to the inlet liner or to the column itself
- Zebron™ columns are specially deactivated to minimize these interactions

# COLUMN SELECTION PROBLEMS

## Dimension Selection

### Short
(15 m or less)

**Applications**
- High boilers
- GC-MS applications

**Advantages**
- Faster run times
- Higher temp. limits
- Lower bleed
- Higher efficiency

**Disadvantages**
- Less inert
- Limited retention

LENGTH
**30m**

### Long
(60 m or more)

**Applications**
- Complex samples with closely eluting peaks
- Low boilers
- Less active samples
- Complex temperature ramps

**Advantages**
- Better resolution

**Disadvantages**
- Slow run times

### Narrow
(0.10, 0.18, or 0.20 mm)

**Applications**
- Complex samples

**Advantages**
- Faster run times
- Better resolution

**Disadvantages**
- Lower sample capacity
- Easily overloaded

INTERNAL DIAMETER
**0.25mm**

### Wide
(0.32 or 0.53 mm)

**Applications**
- Dirty samples
- Highly concentrated samples

**Advantages**
- Increased sample capacity
- Good for routine on-column injection

**Disadvantages**
- Decreased efficiency
- May need higher flow rates
- Not compatible with most GC-MS

### Thin
(0.10 or 0.18 µm)

**Applications**
- High boilers
- GC-MS applications

**Advantages**
- Faster run times
- Higher temp. limits
- Lower bleed
- Higher efficiency

**Disadvantages**
- Less inert
- Limited retention

FILM THICKNESS
**0.25µm**

### Thick
(0.50 µm or more)

**Applications**
- Low boilers
- Gases, solvents, purgeables, volatiles
- Purity testing

**Advantages**
- Better inertness
- Higher capacity

**Disadvantages**
- Slower run times
- Lower temp. limits
- Higher bleed

# COLUMN SELECTION PROBLEMS

## Phase Selection Chart

| Polarity | Phase | Composition | Temperature Limits (Isothermal/TPGC) | GC-MS Certified | Recommended Applications |
|---|---|---|---|---|---|
| **Essentials** | | | | | |
| Non-Polar | ZB-1 | 100% Dimethylpolysiloxane | -60 to 360/370°C* | ✓ | Amines, Drugs, Essential Oils, Ethanol, Gases (Refinery), Hydrocarbons, Mercaptans, MTBE, Natural Gas Odorants, Oxygenates, Simulated Distillation, Solvent Impurities, Sulfur Compounds (Light) |
| Non-Polar | ZB-5 | 95% Dimethylpolysiloxane 5% Phenyl | -60 to 360/370°C* | ✓ | Alkaloids, Dioxins, Drugs, Essential Oils/Flavors, Halo-hydrocarbons, PCBs/Aroclors, Pesticides/Herbicides, Phenols, Residual Solvents, Semi-volatiles |
| Non-Polar | ZB-5ms | 95% Dimethylpolysiloxane 5% Phenyl-Arylene | -60 to 325/350°C | ✓ | Acids, Alkaloids, Amines, Dioxins, Drugs, EPA Methods (525, 610, 625, 8100), Essential Oils/Flavors, FAMEs, Halo-hydrocarbons, PCBs/Aroclors, Pesticides/Herbicides, Phenols, Residual Solvents, Semi-volatiles, Solvent Impurities |
| Intermediate | ZB-35 | 65% Dimethylpolysiloxane 35% Phenyl | 40 to 340/360°C | ✓ | Amines, Aroclors, Drugs, EPA Methods (508, 608, 8081, 8141, 8151), Pesticides, Pharmaceuticals, Semi-volatiles, Steroids |
| Intermediate | ZB-624 | 94% Dimethylpolysiloxane 6% Cyanopropylphenyl | -20 to 260°C | | EPA Methods (501.3, 502.2, 503.1, 524.2, 601, 602, 624, 8010, 8015, 8020, 8021, 8240, 8260), Pharmaceuticals, Residual Solvents, Volatile Organic Compounds (VOCs) |
| Intermediate | ZB-XLB | Proprietary | 30 to 340/360°C* | ✓ | PCBs, Pesticides/Herbicides |
| Intermediate | ZB-1701 | 86% Dimethylpolysiloxane 14% Cyanopropylphenyl | -20 to 280/300°C* | | Alcohols, Amines, Drugs, Esters, PAHs, PCBs, Pharmaceutical Intermediates, Phenols, Solvents, Steroids, TMS Sugars, Tranquilizers |
| Polar | ZB-50 | 50% Dimethylpolysiloxane 50% Phenyl | 40 to 320/340°C* | ✓ | Antidepressants, Aroclors, Aromatic Hydrocarbons, Cholesterols, Drugs of Abuse, EPA Methods (508, 608, 8081, 8141, 8151), Glycols, Pesticides/Herbicides, Steroids, Triglycerides |
| Polar | ZB-WAX | 100% Polyethylene Glycol | 20 to 250/260°C | ✓ | Alcohols, Aldehydes, Aromatics, Basic Compounds, Essential Oils, Flavors & Fragrances, Glycols, Pharmaceuticals, Solvents, Styrene, Xylene Isomers |
| Polar | ZB-FFAP | Nitroterephthalic Acid Modified Polyethylene Glycol | 40 to 250/260°C | | Acrylates, Alcohols, Aldehydes, Free Fatty Acids, Ketones, Organic Acids, Phenols, Volatile Free Acids |
| **Inferno™** | | | | | |
| Non-Polar | ZB-1HT | 100% Dimethylpolysiloxane | -60 to 400/430°C* | ✓ | GROs, High Boiling Petroleum Products, High Molecular Weight Waxes, Long-chained Hydrocarbons, Motor Oils, Polymers/Plastics, Simulated Distillation |
| Non-Polar | ZB-5HT | 95% Dimethylpolysiloxane 5% Phenyl | -60 to 400/430°C* | ✓ | Diesel Fuels, High Boiling Petroleum Products, High Molecular Weight Waxes, Long-chained Hydrocarbons, Motor Oils, Polymers/Plastics, Simulated Distillation, Surfactants, Triglycerides |
| Intermediate | ZB-35HT | 65% Dimethylpolysiloxane 35% Phenyl | 40 to 400°C | ✓ | Amines, Aroclors, Chemicals, Drugs, EPA Methods (508, 608, 8081, 8141, 8151), Pesticides, Pharmaceuticals, Semi-volatiles, Steroids |
| Intermediate | ZB-XLB-HT | Proprietary | 30 to 400°C | ✓ | EPA Methods, PCBs, Pesticides/Herbicides |
| ***PLUS*** | | | | | |
| Non-Polar | ZB-1*PLUS*™ | 100% Dimethylpolysiloxane | -60 to 360/370°C* | ✓ | Acids, Amines, Diesel Fuel, Drugs, Flavors & Fragrances, PCBs (EPA Method 1668), Pesticides |
| Non-Polar | ZB-5*PLUS*™ | 95% Dimethylpolysiloxane 5% Phenyl | -60 to 360/370°C | ✓ | Drugs, EPA Methods, Nitrosamines, Pesticides, Phenols |
| Non-Polar | ZB-5MS*PLUS*™ | 95% Dimethylpolysiloxane 5% Phenyl-Arylene | -60 to 325/350°C | ✓ | Acids, Alkaloids, Amines, Drugs, Ethanolamines, Essential Oils/Flavors, Halo-hydrocarbons, Pesticides/Herbicides, Phenols, Solvent Impurities |
| Intermediate | ZB-624*PLUS*™ | Proprietary | -20 to 300/320°C | ✓ | Cannabis, Terpenes, Residual Solvents, Volatile Amines, EPA Methods (524, 624, 8260), Food, Flavors, Fragrances, Solvent Purity, Alcohols |
| Polar | ZB-WAX*PLUS*™ | 100% Polyethylene Glycol | 20 to 250/260°C* | | Alcohols, Aldehydes, Aromatics, Essential Oils, Flavors & Fragrances, Glycols, OVIs, Pharmaceuticals, Solvents / Residual Solvents, Styrene, Xylene Isomers |
| **Unlimited** | | | | | |
| Non-Polar | ZB-1XT SimDist | 100% Dimethylpolysiloxane | -60 to 450°C* | ✓ | ASTM Methods (D2887, D2887X, D3710, D6352, D7169), Crude Oil, Gasoline Fractions, Petroleum Distillates, Petroleum Fractions, Simulated Distillation, Vacuum Distillates |
| Non-Polar | ZB-DHA-PONA | 100% Dimethylsiloxane | -60 to 360/370°C | ✓ | DHA, PONA, PIONA, PIANO, ASTM Methods (D5134, D5441, D5501, D6729, D6730, D6733) |
| Non-Polar | ZB-SemiVolatiles | 95% Dimethylpolysiloxane 5% Phenyl-Arylene | -60 to 325/350°C | ✓ | Semi-volatiles (SVOCs), PAHs, EPA Methods (525, 610, 625, 8100, 8270D) |
| Intermediate | ZB-MultiResidue™-1 & -2 | Proprietary | -60 to 320/340°C | ✓ | Aroclors/PCBs, Haloacetic Acids, Herbicides, Insecticides, Multi-Pesticide Screening, Nitrogen Containing Pesticides, Organochlorine Pesticides, Organophosphorous Pesticides |
| Intermediate | ZB-CLPesticides-1 & -2 | Proprietary | 40 to 320/340°C | ✓ | Dual-Column Chlorinated Pesticides by GC-ECD, EPA Methods (8081, 8082, 8151, 504, 505, 508, and 552) |
| Polar | ZB-FAME | High Cyanopropyl | -20 to 280°C | ✓ | Fatty acid methyl esters (FAMEs), Cis/trans FAME isomers |
| Proprietary | ZB-BAC-1 & -2 | Proprietary | -20 to 260/280°C | ✓ | Abused Inhalant Anesthetics, Blood Alcohol Analysis |
| Proprietary | ZB-Drug-1 | Proprietary | 40 to 320/340°C | ✓ | Drug Screening (6-MAM, Amphetamines, Barbiturates, Benzodiazepines, Opiates, PCP, THC) |
| Proprietary | ZB-Bioethanol | Proprietary | -60 to 340/360°C | ✓ | Alcohol, Bioethanol, Fusel Alcohols |
| Proprietary | ZB-Dioxin | Proprietary | 40 to 320/340°C | ✓ | Dioxin and PCB in Food and Environmental Samples, POPs in Food |
| Proprietary | ZB-PAH-EU | Proprietary | 40 to 340/360°C | ✓ | Analysis of 15+1 EU-Regulated and EPA-Regulated PAHs in Food Testing, Rubber, Plastic, Coal |
| Proprietary | ZB-PAH-CT | Proprietary | 40 to 320/340°C | ✓ | Analysis of 15+1 EU-Regulated and EPA- Regulated PAHs in Food Testing, Rubber, Plastic, Coal |

*Difference in film thickness determines the upper temperature limit.

# GC Column Cross-Reference Chart

| Zebron™ Phase | Restek® | Agilent® Technologies | Supelco® | Alltech® | SGE® | OV® |
|---|---|---|---|---|---|---|
| ZB-1 | Rtx®-1, Rtx-1PONA, Rtx-1 F&F | DB®-1, DB-2887, DB-1 EVDX, HP-1, HP-101, HP-PONA, Ultra 1, CP-Sil 5 CB | SPB®-1, SPB-1 TG, SE-30, MET-1, SPB-1 Sulfur, SPB-HAP | AT™-1, AT-Sulfur, EC-1 | BP1, BP1-PONA, BPX1-SimD | OV-1 |
| ZB-DHA-PONA | Rtx-DHA | HP-PONA, DB-PETRO, CP-Sil PONA CB | Petrocol®-DH | | | |
| ZB-1*PLUS*™ | Rtx-1ms, Rxi®-1ms | DB-1ms, DB-1ms Ultra Inert, HP-1ms, HP-1ms Ultra Inert, CP-Sil 5 CB MS, VF-1ms | MDN™-1, Equity®-1 | AT-1ms | SolGEL-1ms™ | |
| ZB-1HT Inferno™ | Rxi®-1HT | DB-1ht, CP-SimDist | Petrocol® 2887 | | | |
| ZB-1XT SimDist | MXT®-1HT SimDist MXT-1, MXT-1 SimDist, MXT-2887 | CP-SimDist UltiMetal, CP-Sil 8 CB UltiMetal, BPX1-SimD, DB-PS1, DB-HT SimDis, DB-PS2887 | | | | |
| ZB-5 | Rtx-5 | DB-5, HP-5, Ultra 2, HP-PAS-5, CP-Sil 8 CB, HP-5ms Ultra Inert | MDN-5, SPB-5, PTE-5, SE-54, PTA-5, Equity-5, Sac-5 | AT-5, EC-5 | BP5, BPX5 | OV-5 |
| ZB-5HT Inferno | Stx®-5HT, Rtx-5HT, Rxi-5HT, XTI®-5HT | DB-5ht, VF-5ht | HT-5 | | | |
| ZB-5ms | Rtx-5Sil MS, Rxi-5Sil MS | DB-5ms, DB-5.625, DB-5ms EVDX, VF-5ms, CP-Sil 8 CB MS | | | | |
| ZB-5*PLUS*™ | Rtx-5ms, Rtx-5Amine, Rxi-5ms | DB-5, HP-5ms, HP-5msi | MDN-5S | | | |
| ZB-5MS*PLUS*™ | Rxi-5Sil MS | DB-5ms Ultra Inert, HP-5ms Ultra Inert, DB-5ms, VF-5ms | SLB®-5ms | | | |
| ZB-SemiVolatiles | Rxi-5Sil MS Rxi-5ms | DB-5ms Ultra Inert | SLB®-5ms | | | |
| ZB-35 | Rtx-35, Rtx-35ms | DB-35, DB-35ms, HP-35, HP-35ms | MDN-35, SPB-35, SPB-608 | AT-35 | BPX35, BPX608 | OV-11 |
| ZB-35HT Inferno | | | Phenomenex Exclusive | | | |
| ZB-50 | Rtx-50 | DB-17, DB-17HT, DB-17ms, DB-17 EVDX, HP-50+, CP-Sil 24 CB | SP-2250, SPB-17, SPB-50 | AT-50 | BPX50 | OV-17 |
| ZB-624 | Rtx-1301, Rtx-624 | DB-1301, DB-624, DB-VRX, HP-VOC, CP-1301, CP-Select 624 CB | SPB-1301, SPB-624 | AT-624, AT-1301 | BP624 | OV- 624 |
| ZB-624*PLUS*™ | Rxi-624Sil MS | CP-Select 624 CB, DB-624UI Ultra Inert | | | | |
| ZB-1701 | Rtx-1701 | DB-1701 , CP-Sil 19 CB | SPB-1701, Equity-1701 | AT-1701 | BP10 | OV -1701 |
| ZB-1701P | | DB-1701P | | | | |
| ZB-WAX | Rtx-WAX, Famewax™, Stabilwax®-DB | DB-WAXetr, HP-INNOWax, CP-Wax 57 CB | Met-Wax, Omegawax® | EC-Wax | SolGEL-WAX™ | |
| ZB-WAX*PLUS*™ | Stabilwax® | DB-WAX, CAM, HP-20M, Carbowax 20M, CP-Wax 52 CB | SUPELCOWAX® 10 | AT-Wax, AT-AquaWax | BP20 | Carbowax 20M |
| ZB-FAME | | CP-Sil 88, HP-88, DB-23 | SP-2560, SP-2330 | | | |
| ZB-FFAP | Stabilwax-DA | DB-FFAP, HP-FFAP, CP-Wax 58 (FFAP) CB, CP-FFAP CB | Nukol™, SPB-1000 | AT-1000, EC-1000 | BP21 | OV-351 |
| ZB-MultiResidue™-1 | Rtx-CLPesticides, Stx-CLPesticides | | | | | |
| ZB-MultiResidue-2 | Rtx-CLPesticides2, Stx-CLPesticides2 | | | | | |
| ZB-CLPesticides-1 | Rtx-CLPesticides, Stx-CLPesticides | | | | | |
| ZB-CLPesticides-2 | Rtx-CLPesticides2, Stx-CLPesticides2 | | | | | |
| ZB-XLB | Rtx-XLB, Rxi-XLB | DB-XLB, VF-XMS | MDN-12 | | | |
| ZB-XLB-HT Inferno | | | Phenomenex Exclusive | | | |
| ZB-Drug-1 | | | Phenomenex Exclusive | | | |
| ZB-BAC1 | Rtx-BAC-1 | DB-ALC1 | | | | |
| ZB-BAC2 | Rtx-BAC-2 | DB-ALC2 | | | | |
| ZB-Bioethanol | | | Phenomenex Exclusive | | | |
| ZB-Dioxin | | Rtx-Dioxin-2 | SP-2330 | | | |
| ZB-PAH-EU | Rxi-PAH | DB-PAH-EU | | | | |
| ZB-PAH-CT | | PAH-Select | | | | |



This section is, neither in terms of manufacturers nor in terms of their products, a complete list, and the accuracy of the data is not guaranteed. Small differences in dimensions or performance might be possible and slight adjustments to your application may be necessary.

# PROBLEM PREVENTION





## The Inlet: Sample Injection Techniques

One size does not fit all – there is no single injection mode that accommodates all samples and all columns. Instead, an appropriate injection mode introduces the sample so that it:

- Retains its original composition (i.e. there should be no sample degradation or selective losses during injection)
- Occupies the shortest possible length of column (the shorter the initial sample band, the sharper the peaks, the better the sensitivity, and the better the resolution)

### Injection Modes: Split

In split injection, the sample is rapidly vaporized and mixed with carrier gas. Most of the sample is vented through the split vent, while a small amount enters the column. The flow through the split vent divided by the flow through the column is called the "split ratio". This rapid sample introduction provides the basis for sharp peaks and good resolution; it may however be inappropriate if sample components vary widely in their boiling points.

**Inlet Discrimination** | The less volatile components of a sample will not vaporize as rapidly, so immediately after injection the vaporized sample has a greater proportion of the more volatile compounds than the original sample. This effect is called "discrimination". The longer the sample spends in the heated inlet, the less the discrimination – but the broader the peaks.

**Backflash** | Backflash occurs when the vaporized sample expands and exceeds inlet liner volume. Vapors may come in contact with cold spots (e.g. the septum or inlets to the injector) and less volatile components may condense. These condensates may vaporize later and interfere with subsequent analyses, sometimes producing "ghost peaks." Expansion outside the liner may also expose the sample to active metal surfaces and reactive components of the sample may be lost. Minimized backflash by using a septum purge, small injection volumes, large volume inlet liners, and optimal inlet temperatures.

**Inlet Temperature** | The temperature should be hot enough to ensure rapid vaporization of the entire sample, but not too hot to degrade any analytes. Experimentation may be required to mitigate inlet discrimination and backflash. A good starting point is 250 °C.

**Septum Purge** | Gas sweeping the bottom face of the septum and through a purge vent carries contaminants out. Higher than optimum purge flows may result in the loss of more volatile sample components. Septum purge flow rates are usually between 0.5 and 5 mL/min.

**Sample Size & Concentration** | Split injection is used for highly concentrated samples. Typical concentrations are from 0.1-10 µg/µL. Injection volumes of 1 to 2 µL are common, and up to 5 µL can be used without great problems, depending on the solvent used. If the sample volume is too large, backflash may occur.

### Injection Modes: Splitless

In splitless injection, the entire flow through the injector passes into the column for the first 15 to 90 seconds, and is then refocused.

**The Solvent Effect** | To avoid the broad peaks that would otherwise result from slow split injections, samples are refocused before starting the chromatographic process following splitless injection. Refocusing can be accomplished by adjusting the initial column temperature to 10 °C or more below the boiling point of the sample solvent. When the vapor leaves the injector and enters the cooler column, the solvent condenses at the front of the column as a liquid band; vapors will condense in this band and be trapped and refocused. This process is called the "solvent effect." Improper use of solvent effect techniques result in broad, distorted peaks because solutes are not refocused into a narrow band near the beginning of the column. The solvent must form a compact, continuous flooded zone in the column. If the solvent does not wet the stationary phase sufficiently (as may be the case for methanol with a nonpolar phase), the flood zone may be several meters long and not of uniform thickness.

# PROBLEM PREVENTION

## The Inlet: Sample Injection Techniques

**Cold Trapping** | Solutes that boil at 150 °C or more above the initial column temperature do not require the solvent effect in order to refocus. These high boiling compounds will condense at the beginning of the column in a short band without the aid of the solvent. This process is called "cold trapping." Both the solvent effect and cold trapping can be achieved by operating in a temperature programmed mode.

**Sample Volume** | Samples are usually limited to 2 µL or less to avoid overloading the inlet liner and the column. Sample injection volumes must be reproducible in order to obtain reproducible retention times or quantitative data.

### Injection Modes: On-Column

On-column injection can eliminate syringe and inlet-related discrimination. If polar solvents are used with non-polar column linings, a retention gap is recommended. If the solvent is below boiling point at injection, the sample is distributed over a flooded zone at the front of the column and less volatile compounds are distributed in the phase. As the carrier gas evaporates the solvent at the front end, volatile components are concentrated and refocused.

**Sample Focusing** | The distribution of solutes in the area of the flooded zone is not homogeneous and this leads to peak broadening; this can be neglected for many applications and good quantitative results can still be obtained. If the compound boiling points are vastly different from the solvent, ballistic heating to high temperature can be employed. If compound boiling points are close to the solvent, temperature programming can be used. Wide-bore columns make on-column injection easier; alternatively a deactivated but uncoated wide-bore retention gap may be connected to a narrow-bore column.

**Sample Size** | Samples between 1 and 2 µL can be injected rapidly into a column below the boiling point of the solvent. To keep the flooded zone short, sample size should be limited to 1 µL.

### Injection Modes: Direct Injection

Direct injection should not be confused with on-column injection. It is a flash vaporizing method in which the inlet system is heated independently from the column oven. Sample evaporation occurs in the inlet.

### Injection Modes: Programmed Temperature Vaporizing (PTV)

In PTV injection, the liquid sample is injected into a cold glass liner. After withdrawal of the syringe needle, the vaporizing tube is heated in a controlled manner (usually rapidly) to vaporize the sample. This injection method permits special handling of the sample to vent the solvent, or to avoid thermal decomposition of thermally labile compounds, etc.



# PROBLEM PREVENTION

## The Inlet: Setting A Maintenance Schedule

Many GC troubleshooting issues arise because system parts need to be replaced. It is not always obvious which part needs replacing. Therefore, a fair amount of time can be spent locating the problem part before it can be fixed.

Instead of waiting for a problem to occur, it is best to be proactive about your GC maintenance. For instance, the majority of GC issues are inlet related. By replacing inlet parts such as liners and septa on a regular basis, problems will occur far less frequently. Since problems will be occurring less often, there will be fewer instrument downtimes, resulting in greater productivity.

Below is a list of inlet parts which should be replaced regularly to prevent instrument downtime. Depending on how dirty the samples are, some parts will need to be replaced more or less often. In such instances, adjust the length of time or number of injections that is appropriate for the samples.

| | Item | Replacement Frequency |
|---|---|---|
|  | Septa | 100 Injections<br>(depends on needle style) |
|  | Inlet Liner | Sample & Matrix Dependent<br>*Common Replacement Frequencies*<br>• Dirty/Soil Samples: < 2 weeks<br>• Water Extracts: ~ 4 weeks<br>• Headspace Extracts: ~ 6 months |
|  | O-Ring | 6 months<br>(or with each liner change) |
|  | Inlet Seal | Sample Dependent<br>(no more than 6 months) |

**For a much more in-depth discussion of setting your GC inlet maintenance schedule, please contact your GC Specialist at GCSpecialist@Phenomenex.com.**

# PROBLEM PREVENTION

## The Inlet: Maintenance & Cleaning

**Warning! This procedure involves the use of compressed gas and therefore eye protection should be worn.**

**Note:** It best to have clean replacement liners or inserts available for quick exchange.

Full maintenance cleaning procedure:

1. Turn off inlet heat and allow inlet to cool.
2. Remove septum.
3. Remove liner or insert.
4. Remove base seal if applicable.
5. Use dry air or nitrogen to blow out any loose particles.
6. Use swab and solvent to clean interior walls if required.
7. Replace septum, liner or insert, and base seal.
8. Vent lines may also require replacement or cleaning.
9. Reassemble inlet and purge with clean, dry gas to remove solvent.

**Note:** Light maintenance may not require changing of septum or base seal. Avoid touching any parts that go inside the inlet with fingers as fingerprints will cause contamination.




# PROBLEM PREVENTION

## The Detector: Maintenance & Cleaning

**Warning! Wear eye protection when working with fused silica tubing or compressed gas.**

### Electron Capture Detectors (ECD)

Because of the use of radioactive nickel in this type of detector, it should not be disassembled by those without specialized training and an appropriate license. Cleaning is limited to baking it out at 350 °C from 3 hours to overnight. Verify there are no leaks and the carrier gas is clean and dry before baking.

### Flame Ionization Detectors (FID)

The collector bore and the jet require occasional cleaning to remove deposits. The deposits, which usually consist of white silica from column bleed or black carbonaceous soot, cause noise and spikes.

**Cleaning procedure:**

1. Turn off detector and its heater.
2. Turn off gases to the detector.
3. Allow time for the detector to cool.
4. Open up the detector and use mechanical means (brush, wire, etc., and compressed gas) to remove contamination.
5. Wash out the collector with distilled water and organic solvents as required.
6. Dry in an oven at about 70 °C for more than half an hour.

### Flame Photometric Detectors (FPD)

**Cleaning procedure:**

1. Set instrument temperatures to cool to safe temperatures.
2. Turn off gasses to the detector.
3. Turn off power to the gas chromatograph and unplug main power cord.
4. Remove detector covers, disconnect, and remove the detector.
5. Remove and inspect jet assembly. Remove any deposits mechanically, for example, by using a wire.
6. Inspect and clean, if necessary, the glow plug and the quartz windows.
7. Blow loose particles away with compressed gas.
8. Replace the jet if it is damaged or difficult to clean with a wire.

### Nitrogen Phosphorous Detectors (NPD)

**Caution:** If the hydrogen gas used to fuel the NPD detector is left on after the detector is disconnected from the column, this gas can accumulate in the oven and create an explosion hazard.

The collector bore and the jet require occasional cleaning to remove deposits. The deposits, which usually consists of white silica from column bleed or black carbonaceous soot, cause noise and spikes.

# COLUMN CARE & USE GUIDELINES



# COLUMN CARE & USE GUIDELINES

## Column Installation

### Pre-Installation Checklist

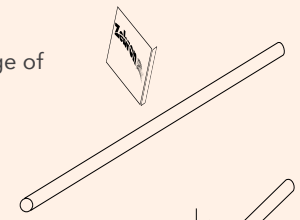
- Replace oxygen, moisture and hydrocarbon traps as necessary.
- Ensure that the injection port is clean and free of sample residues, septum, or capillary debris.
- Check and replace as necessary critical injector components such as seals, liners, and septa.
- Check and replace detector seals as necessary.
- Carefully inspect your column for damage or breakage.
- Check gas cylinder pressures to ensure that an adequate supply of carrier, make-up and fuel gases are available. Carrier gases should be of the highest purity. **Note:** It is critical that oxygen and water be removed from the carrier gas by the appropriate use of filters and adsorbents.

### Designate A Flow Direction

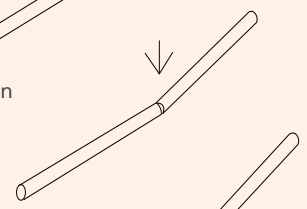
**Note:** GC columns do not have a specific directional flow when received from the manufacturer. Upon initial use of your new Zebron™ column, Phenomenex recommends the practice of dedicating one specific end of the column for injector installation only. This is particularly important when dealing with active/caustic or contaminating compounds. If these compounds are routinely injected onto the column, degradation of the phase will occur—leading to higher bleed. A typical first step to remedying (removing) this bleed would be to trim 10 cm from the front (injector) end of the column and keep trimming this inlet end of the column as necessary. Trying to remedy any bleed issues by trimming the column may not work if both ends have been interchangeably installed into the inlet.

### Ensure A Proper Column Cut

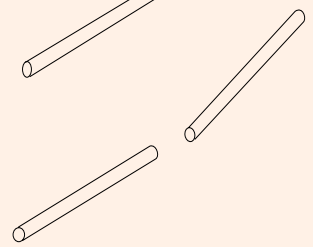
1. Score capillary with smooth edge of wafer at a 45° angle



2. Apply force in a downward direction



3. Tubing should break cleanly



4. Inspect cut with a magnifying glass - the cut should be smooth, not jagged

**Proper & Improperly Cut Capillary**

Correct Incorrect

# COLUMN CARE & USE GUIDELINES

## Column Installation

### Installation Into the Injector

1. Place a capillary nut and ferrule on the injector end of the GC column, allowing a section of column to protrude. Trim one to two centimeters from the protruding end to remove ferrule contamination that may have entered the column. Inspect the cut with a magnifier to ensure that a smooth, clean, square-cut edge has been made - recut if necessary.
2. Carefully hang the column in the GC oven, being cautious not to scratch or damage the polyimide coating on the capillary tubing. Rotate the column to avoid sharp bends of the capillary column and any contact of the column with oven surfaces.
3. Insert the column into the injector exactly the correct distance specified in the instrument manual. Tighten the ferrule nut finger-tight then 1/2 turn with a wrench. If the column can still be moved, tighten another 1/4 turn until the column is secure.
4. Adjust the carrier gas to obtain the flow rate listed on the test chromatogram.

### Installation Into the Detector

**Note:** For users with sensitive detectors such as MS and ECD, column conditioning steps should be performed before installing the column to prevent contamination and frequent maintenance of the detector.

1. Place the column nut and ferrule past the end of the column and cut a centimeter or two off the end of the column. Be sure that the ferrule is the right size and pointing in the correct direction. Inspect the cut with a magnifier and ensure that the cut is square and smooth. Recut if needed.

2. Insert the outlet end of the column into the detector exactly the distance prescribed in the instrument manual. Distances will vary between detectors. Tighten the ferrule nut finger-tight then 1/2 turn with a wrench. If the column can still be moved, tighten another 1/4 turn until the column is secure.

3. Inspect the column connections for leaks using an electronic leak detector. Leaks at the inlet end may introduce oxygen to the column that will result in increased column bleed and damage to the column phase.



# COLUMN CARE & USE GUIDELINES

## Conditioning Basics: The Column

### Column Conditioning Steps

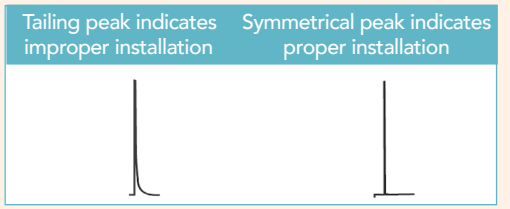
1. Allow sufficient time for the carrier gas to flow through the column to purge any oxygen that may be in the system.
2. Raise the temperature of the column to the maximum isothermal operating temperature that is listed on the individual Zebron™ GC Column Test Report. Maintain this temperature until a constant baseline is achieved. Conditioning times will depend on the phase identity and thickness, with thicker films taking longer to stabilize. In order to minimize the downtime of the instrument, columns can be conditioned overnight at the maximum isothermal temperature.

### Installation Testing

1. Inject a detectable unretained sample, such as methane for an FID, to determine dead volume time and linear gas velocity at the desired column temperature. Adjust gas pressure for optimal flow depending on carrier gas selection.
2. The non-retained peak must have ideal peak shape or installation is faulty and needs to be redone.

**If the peak is broad and/or tailing, check the following:**

- Improper column positioning/insertion into inlet or detector
- Gross contamination of the splitter sleeve
- Chipped or cracked splitter sleeve
- Improper sweeping of sample at column end by makeup gas
- Damaged or crushed column end




**Unretained Peak Times and Markers**
**Methane with FID/TCD:** Calculate linear velocity by injecting 25-100 µL of 1% methane in $N_2$ gas blend. Measure the retention time of the methane peak and calculate the following:
**Linear Velocity (u) = L/to**

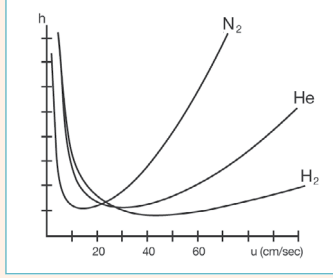
| Detector Type | Marker Compound |
|---|---|
| ECD | Methylene chloride[2, 3], Dichlorodifluoromethane |
| FID | Methane, Butane[1] |
| NPD | Acetonitrile[2, 4] |
| PID ELCDVinyl chloride | |
| TCD, MS Methane, Butane[1], air | |

1. From a disposable lighter
2. Place 1-2 drops in an autosampler vial and tightly cap. Shake and inject 1-2 µL from the headspace of the vial. Do not inject any liquid.
3. Use a column temperature above 55 °C.
4. Use a column temperature above 95 °C.

**Recommended Non-Retained Retention Times**

| Length (m) | $H_2$ (sec) | He (sec) | $N_2$ (sec) |
|---|---|---|---|
| 15 | 38 | 75 | 150 |
| 30 | 75 | 150 | 300 |
| 60 | 150 | 300 | 600 |

**h (u) Curves for Different Gases**



## Conditioning Basics: The System

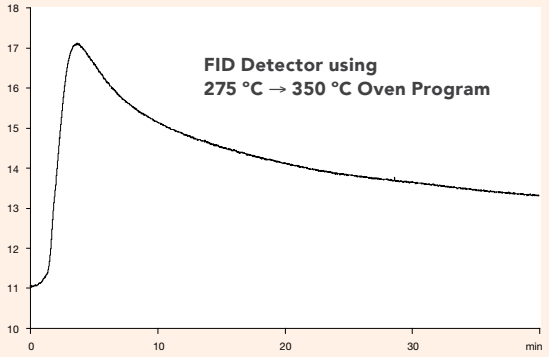
Increased signal is common and expected during the conditioning step when new components are installed. After a new column installation, detectors show increased signal response that decrease slowly over time with constant increased temperature. Misconception is that the baseline rise is solely due to column bleed. It may be a combination of many causes that collectively can be called system bleed. In fact, at temperatures below 200 °C, almost all background signal is the result of system noise.

### Detector Effects

During new column installation, detectors are sometimes allowed to cool for convenience. If the column is connected to the detector during conditioning, the detector can become contaminated. When the detector is heated following column installation an increased signal will be observed that can be interpreted as column bleed as shown below. An increase in detector temperature from a constant temperature of 275 °C to 350 °C caused a signal increase of 6 pA!

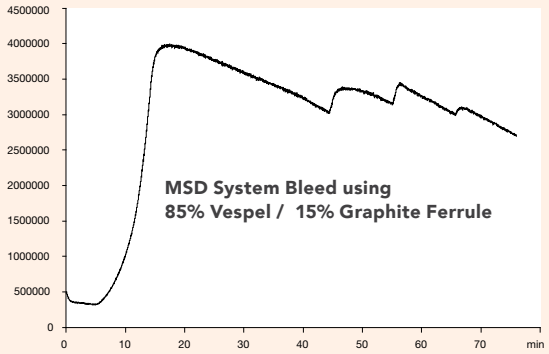
The effect would be greater if the detector had been constant at room temperature for an extended period of time. It is not uncommon for more sensitive detectors such as Electron Capture Detectors (ECD) to show very high signals that may persist for days after prolonged dormancy.

Repetitive heating and cooling of detectors can cause seal and ferrule distortion, allowing leaks to form. This might introduce oxygen into the system resulting in increased signal response.




### Accessory Effects

If the detector temperature remains constant, other causes for baseline increases are still possible. Ferrules absorb gases and other substances that offgas when heated. Figure shows a spectrum that was obtained when an uncoated capillary column, which does not contain stationary phase, was installed using new ferrules. The signal is due to system noise only and not column bleed. Notice that the intensity of the signal is very high and would be greater than most analyte peaks. This would decrease the signal to noise ratio making detection limits much worse than if the system was conditioned and the baseline minimized.



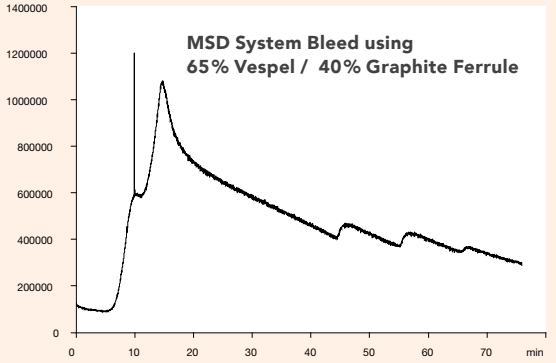

**Oven Program:**
40 °C for 5 min to 320 °C at 30 °C/min for 30 min to 340 °C at 30 °C/min for 10 min to 360 °C at 30 °C/min for 10 min to 370 °C at 30 °C/min for 10 min.





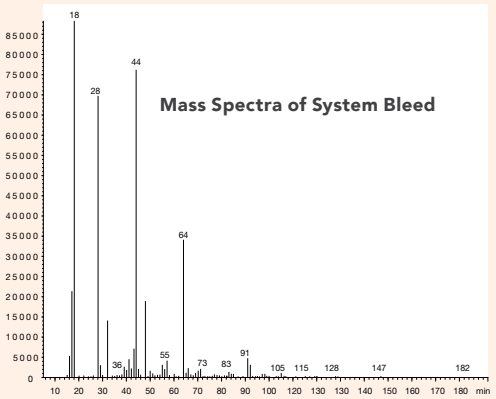
## Conditioning Basics: The System

Ferrule composition also effects system bleed. After the completion of the same oven program using a ferrule with higher graphite content, the signal is 90% lower.

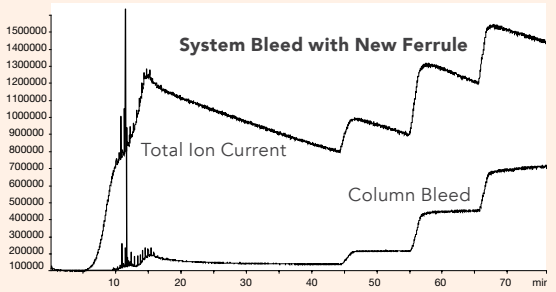



**Oven Program:**
40 °C for 5 min to 320 °C at 30 °C/min for 30 min to 340 °C at 30 °C/min for 10 min to 360 °C at 30 °C/min for 10 min to 370 °C at 30 °C/min for 10 min.

The major ions seen are 17, 18, 28, 32, and 44, and 64. Most masses can be easily explained by common gases adsorbing on the ferrule (such as water, carbon monoxide, nitrogen, oxygen, and carbon dioxide.) Since these same ions are indicators of a gas leak or contaminated vacuum chamber, an air and water check was run and passed before analysis. Subsequent runs after initial conditioning showed drastically reduced signals for these ions. An air leak would likely remain constant.




New 60/40% Vespel-Graphite ferrules were installed with a Zebron™ ZB-5 column. Subtracting the ions associated with system bleed shows bleed from column conditioning only. At normal operating conditions, over 90% of the ion intensity is completely due to system bleed derived from ferrule offgassing.




Installation of any column should be followed by a heating cycle to condition the system. During this conditioning cycle, the entire system is being conditioned, not only the column. To prevent detector contamination, it is recommended that you do not connect the column to the detector. Ferrule composition can also determine the extent of conditioning needed, as it is the key component responsible for most offgassing. Injector contributions should also be considered when determining system bleed; they may appear as peaks.

# GC

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