GC TROUBLESHOOTING GUIDE

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Australia
t: 02-9428-6444
f: 02-9428-6445
auinfo@phenomenex.com

Austria
t: 01-319-1301
f: 01-319-1300
anfrage@phenomenex.com

Belgium
t: +31 (0)30-2418700
f: +31 (0)30-2383749
beinfo@phenomenex.com

Canada
t: (800) 543-3681
f: (310) 328-7768
info@phenomenex.com

Denmark
t: 4824 8048
f: 4810 6265
nordicinfo@phenomenex.com

Finland
t: (09)4789 0063
f: +45 4810 6265
nordicinfo@phenomenex.com

France
01 30 09 21 10
01 30 09 21 11
franceinfo@phenomenex.com

Germany
06021-58830-0
06021-58830-11
anfrage@phenomenex.com

Ireland
01 247 5405
+44 1625-501796
eireinfo@phenomenex.com

Italy
051 6327511
051 6327555
italiainfo@phenomenex.com

Luxembourg
+31 (0)30-2418700
+31 (0)30-2383749
nlinfo@phenomenex.com

Mexico
(55) 5018 3791
(310) 328-7768
tecnicomx@phenomenex.com

The Netherlands
030-2418700
030-2383749
nlinfo@phenomenex.com

New Zealand
09-4780951
09-4780952
nzinfo@phenomenex.com

Norway
810 02 005
+45 4810 6265
nordicinfo@phenomenex.com

Puerto Rico
(800) 541-HPLC
(310) 328-7768
info@phenomenex.com

United Kingdom
01625-501367
01625-501796
ukinfo@phenomenex.com

All other countries:
Corporate Office USA
(310) 212-0555
(310) 328-7768
info@phenomenex.com

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GC Troubleshooting Guide

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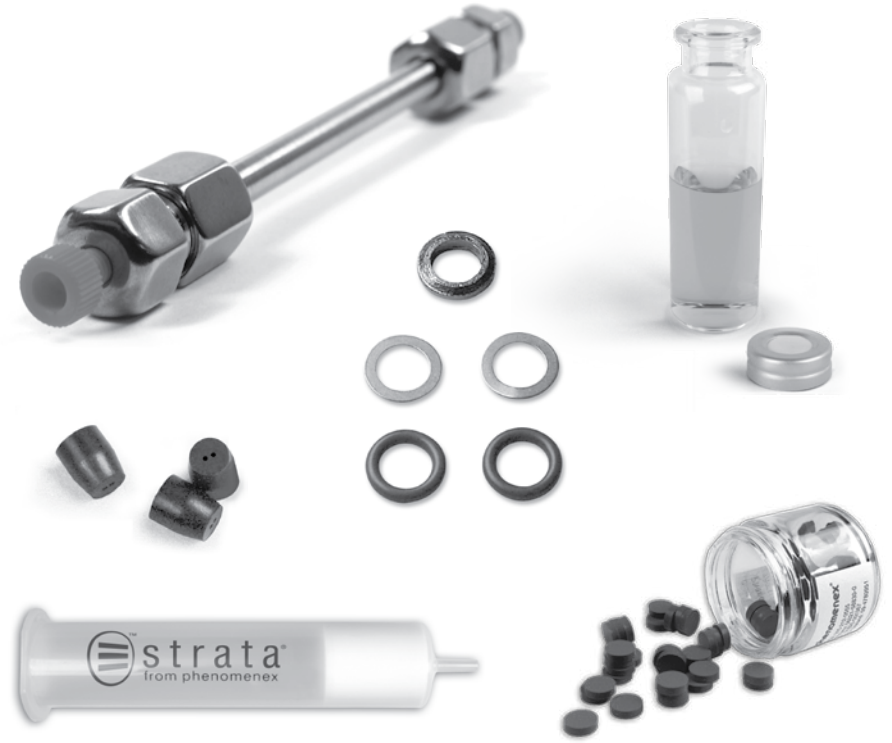

I. INTRODUCTION

LOCATING AND CORRECTING THE PROBLEM
Identifying the problem is the first step in troubleshooting. This guide is organized by eight major categories of symptoms:

- Baseline problems, pp. 6-8
- Peak shape, pp. 9-16
- Ghost peaks, pp. 10-11
- Irreproducible peak heights or areas, p. 11
- Retention time shifts, p. 14
- Resolution, p. 15
- Broad solvent fronts, p. 15
- Rapid column performance degradation, p. 16

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, injector liners
- Electronic leak detector
- Reference sample
- Reference column with known performance

When you have corrected the problem, record the incident in the system record book to help avoid future problems.

PREVENTION
Many GC problems can be prevented if the column is properly installed and maintained routinely. For example, replacing septa at regular intervals and keeping the injector and detector clean and well-maintained should solve many problems. Section VII lists the most common problem areas for each GC module and preventive maintenance practices that will reduce their frequency. These suggestions should be modified to fit your particular model of GC, and then made a regular part of your laboratory routine.

WHERE TO GET 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, faxes or emails.

- There are a number of reference sources that can give you guidance in problem solving:

M. McMaster, ***GC/MS: A Practical User's Guide,*** 1998.

Robert L. Grob*,* ***Modern Practice of Gas Chromatography,*** 1995.

Dolan, J.W., "Troubleshooting", *LC/GC Magazine.* This is a monthly column.

McNair, Harold M. and James M. Miller, ***Basic Gas Chromatography***, J. Wiley and Sons, 1997.

II. BASELINE PROBLEMS

Baseline symptoms are divided into the following categories:

Drift.....The rather slow movement of the baseline in one direction (either up or down), p. 6
Noise.....The rapid and random movement of the signal amplitude, p. 6
Offset.....The sudden unexplained change of the baseline position, p. 7
Spiking..... Peaks with no width, either positive or negative, p. 8
Wander ....Low frequency noise, p. 8

Downward drift

POSSIBLE CAUSE	SUGGESTED REMEDY
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, p. 17
Unequilibrated detector or oven	Allow sufficient time for (temperature) equilibration of the detector or oven.
Downward drift is frequently due to the “bake-out” of contaminants from the detector or other parts of the GC	Clean out contamination. See pp. 23-26

Upward drift

POSSIBLE CAUSE	SUGGESTED REMEDY
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 p. 17
Drift in gas flow rates	Clean or replace flow or pressure regulator(s). Adjust pressure. See pp. 23-26

Noise

POSSIBLE CAUSE	SUGGESTED REMEDY
The column may be inserted too far into the flame of an FID, NPD or FPD detector	Reinstall the column. See p. 17. Be sure to insert the column into the detector exactly the correct distance specified in the instrument manual.
An air leak can result in noise in ECD and TCD detectors	Eliminate the leak

II. BASELINE PROBLEMS (continued)

Noise (continued)

POSSIBLE CAUSE	SUGGESTED REMEDY
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. See p. 25. Replace inlet liner. Replace glass wool.
Contaminated column	Bake out the column. Cut off first 4 inches of column. Solvent rinse or replace column. See pp.17, 22
Defective detector	Clean and/or replace parts as necessary. See pp. 23-25
Defective detector board	Consult GC manufacturer.

Offset

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. See p. 25. Replace inlet liner. Replace glass wool.
Contaminated column	Bake out the column. Cut off first 4 inches of column. Solvent rinse or replace column. See pp.17, 22
Column inserted too far into the flame of FID, NPD, or FPD detectors	Reinstall the column. See p.17. Be sure to insert the column into the detector exactly the correct distance specified in the instrument manual.
Contaminated detector	Clean the detector. See pp. 23-25
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.

II. BASELINE PROBLEMS (continued)

Spiking

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. See pp. 23- 25. A clean H_2 flame is invisible. Most organic matter generates a yellow flame.
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 flame ionization detector parts.

Wander

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. See p. 25. Replace inlet liner. Replace glass wool.
Contaminated column	Bake out the column. Cut off first 4 inches of column. Solvent rinse or replace column. See pp.17, 22
Poor control of gas flow rates	Clean or change flow controller(s).

III. DISTORTED PEAK SHAPES

All peaks reduced in size

POSSIBLE CAUSE	SUGGESTED REMEDY
Defective 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. See p. 17
Purge flow or split ratio too high	Adjust gas flow rates.
ıjector and/or column temperature too low for high molecular weight or low volatility samples	Increase injector and/or column temperature(s). For a temperature programmed column, the upper temperature is important. Consult column manufacturer.
NPD detector may be coated with silicon dioxide. This coating may be the result of bleed from a silicone column or from residual silanizing regents used in derivatization.	Replace active element. Avoid exposure to silicon containing compounds. Many beads have short lifetimes of only 6 months.
NPD detector may be damaged by loss of rubidium salt as a result of exposure to overheating, heating in the absence of clean gas flow or humidity during storage	Replace the active element. Turn the detector off whenever the gas flow is interrupted. Avoid over-heating. 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 column temperature is below the boiling point of the solvent.
Detector-sample mismatch	Make sure that the detector will respond to the compounds being analyzed.
Inadequate signal amplification	Check output signal levels.

III. DISTORTED PEAK SHAPES (continued)

All peaks reduced in size (continued)

POSSIBLE CAUSE	SUGGESTED REMEDY
Sample validity	Check the concentration and stability of the sample.

Flattened top peaks

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

Fronting peaks

Fronting peaks are usually the result of column overloading. In this case, the effect should be a function of injection volume and split ratio. Solutions include reducing the injection volume, adding or increasing a split flow or using a column with greater capacity. Columns with larger diameter or thicker stationary phase coatings generally have larger sample capacities; however, resolution may be reduced. In some cases, fronting peaks can be due to a polarity mis-match. For example, polar compounds will have lower concentration capacity on a non-polar phase.

Ghost peaks

Ghost peaks are peaks observed when no sample has been introduced into the system (i.e., discrete peak(s) in a blank run).

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. See p. 25. Condition the column at a temperature higher than has been used but lower than the maximum continuous operating temperature for the column. Cut 4 inches off the inlet end of the column and/or reverse it (end-for-end) before reconditioning it. Solvent rinse, or replace the column. See pp. 17, 22

III. DISTORTED PEAK SHAPES (continued)

Ghost peaks (continued)

POSSIBLE CAUSE	SUGGESTED REMEDY
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".	Backflash can be minimized by using: • septum purge, see p. 20 • small injection volumes • large injector liners • optimal injector temperatures • pulsed pressure programming • 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. See pp. 25-26
Syringe contamination	Replace the syringe.

Irreproducible peak heights or areas

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. See Peak shape, pp. 9-16.
Baseline disturbances	See Baseline problems, pp. 6-8.
Variations in GC operating parameters	Standardize operating parameters.

Negative peaks

POSSIBLE CAUSE	SUGGESTED REMEDY
Incorrect polarity of the recorder connections results in nearly all peaks being negative	Reverse polarity of recorder connections.
Sample compound has greater thermal conductivity than the carrier gas and you are using a TCD detector	No solution required or suggested.
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.

III. DISTORTED PEAK SHAPES (continued)

Negative peaks (continued)

POSSIBLE CAUSE	SUGGESTED REMEDY
Dirty ECD detector can give negative peak after a positive one	Clean or replace the ECD detector. See p. 23

No peaks at all

POSSIBLE CAUSE	SUGGESTED REMEDY
Defective 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	Replace or reinstall the column. See p. 17
The detector not functioning or 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.

Selective sensitivity loss

POSSIBLE CAUSE	SUGGESTED REMEDY
Contamination of column and/or liner can lead to loss of sensitivity for active compounds	Clean liner. See pp. 25-26 Bake out the column. Solvent rinse or replace the column. See pp. 17, 22
Injector leaks reduce the peak height of the most volatile components of a sample more than the less volatile	Find and fix any leaks
Initial column temperature too high for splitless injection which can prevent refocusing of sample. This affects the more volatile components most.	Initial column temperature should be below the boiling point of the solvent. Decrease the initial column temperature, or use less volatile solvent.
Inlet descrimination—Injector temperature is too low. Later eluting and less volatile compounds have low response.	Increase injection temperature or use on-column injection of direct connect liner

Split peaks

POSSIBLE CAUSE	SUGGESTED REMEDY
Poor (jerky or erratic) injection	Use smooth, steady plunger depression. Use autosampler.
Bad column installation	Reinstall column, p. 17.
Solvent mismatch–The polarity of the stationary phase does not match the polarity of the solvent	Change solvent, use very large split ratio, install retention gap, or change 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.

III. DISTORTED PEAK SHAPES (continued)

Split peaks (continued)

POSSIBLE CAUSE	SUGGESTED REMEDY
Fluctuations in column temperature	Repair temperature control system.
Mixed sample solvent for splitless or on-column injections	Use single solvent.
When using injection techniques that require "solvent effect" refocusing such as splitless injection, the solvent must form a compact, continuous flooded zone in the column. If the solvent does not wet the stationary phase (column lining) sufficiently as might be the case for methanol used with a nonpolar stationary phase, the solvent flooded zone may be several meters long and not of uniform thickness. This will result in broad and distorted peaks because the solutes will not be refocused into a narrow band near the beginning of the column.	• Installing a retention gap (5 meters require "solvent effect" refocusing of uncoated but deactivated column) ahead of the chromatographic column may reduce or eliminate this problem. • Change solvent or GC column phase • Use a very high split ratio

Tailing peaks

POSSIBLE CAUSE	SUGGESTED REMEDY
Contaminated or active injector liner or column	Clean or replace injector liner. See pp. 25-26 Don't use glass wool in the liner. Solvent rinse or replace the column. See pp. 17, 22
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 pp. 17, 25-26
Ragged column end	Score the tubing lightly with a sapphire scribe or 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 p. 17

III. DISTORTED PEAK SHAPES (continued)

Tailing peaks (continued)

POSSIBLE CAUSE	SUGGESTED REMEDY
A bad match between the polarities of the stationary phase and the solvent	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.
Debris in the liner or column	Clean or replace the liner. Cut 4 inches off the end of the column and reinstall it. See pp. 17, 25-26
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 sample.
Some types of compounds such as alcoholic amines, primary and secondary amines, and carboxylic acids tend to tail	Try a more polar column. Make a derivative of the sample.

Retention time shifts

POSSIBLE CAUSE	SUGGESTED REMEDY
Change in column temperature	Check GC oven temperature.
Change 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.
Leak in the injector	Check the septum first. Change, if necessary. Find the leak and fix it.
Change of solvent	Use the same solvent for standards and samples.
Contaminated column	Bake out the column Cut 4 inches off the end of the column Solvent rinse or replace the column See pp. 17, 22

III. DISTORTED PEAK SHAPES (continued)

Loss of resolution

POSSIBLE CAUSE	SUGGESTED REMEDY
Damage to stationary phase of column	Replace the column. See p. 17 This is usually indicated by excessive column bleeding
Injector problems	Check for: • leaks • inappropriate temperature • split ratio • purge time • dirty liner • glass wool in liner
Large increases in sample concentration	Dilute sample Inject less Use higher split ratio

Broad solvent fronts

POSSIBLE CAUSE	SUGGESTED REMEDY
Bad column installation	Reinstall column. See p. 17
Injector leak	Find and fix leak
Injection volume too large dilute	Decrease sample injection volume or 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. 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 (splitless injection)	Use a shorter purge valve closed time.

III. DISTORTED PEAK SHAPES (continued)

Rapid column performance degradation

POSSIBLE CAUSE	SUGGESTED REMEDY
Broken column	Replace column. See p. 17. 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 possibly break spontaneously later.
Column too hot for too long	Replace column. See p. 17. Stay below limits specified for the column.
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	Prevent nonvolatile materials from getting into column. For example, use a guard column or a Guardian™ integrated column.

IV. CAPILLARY COLUMN INSTALLATION

In order to obtain optimal performance from your gas chromatograph, the column must be properly installed. The column must be inserted into the injector and the detector exactly the distance prescribed by the GC instrument manual. Carefully follow the procedure presented below to obtain the best performance from your chromatograph. **Phenomenex offers a comprehensive Users Guide to Fused Silica GC Capillary Columns. Call to reserve your copy.**

General Precautions

Standard Fused silica capillary columns become brittle if the polyimide coating applied during manufacture is damaged. 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 abrade 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.

The stationary phase that coats the inside of the column must also be protected. All foreign material, including debris from the septa or ferrules, must be kept out of the column. The column should be installed in a chromatograph as soon as practical after the ends have been opened and a flow of dry, oxygen-free carrier gas maintained until the column is removed and resealed.

The ends of the column must be clean and square. In order to insure this, the ends must be trimmed and examined. To cut the column, first use a sapphire scribe or a ceramic scoring wafer to lightly score the outer surface. Very little force is required, since only the thin polyimide coating needs to be cut. Grasp the column on each side of this scribe and pull along the tubing length while bending away from the scribe. A clean cut should result. 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 falling into the column. Cut column after the nut and ferrule.

To install a column

Warning! ***It is advisable to wear safety glasses.***

1. Turn off inlet and detector and allow them to cool.
2. Clean and replace injector liner.
3. Replace seals and septum.
4. Inspect the column for damage.
5. Install a nut and ferrule. Cut a centimeter or two off an end of the column after the nut and ferrule to avoid pieces in column. Be sure the ferrule is the right size and pointed in the correct direction.

IV. COLUMN INSTALLATION (continued)

6. Mount the column in the GC oven without damaging the column coating.
7. Insert the column into the injector exactly the correct distance specified in the instrument manual. Tighten the ferrule nut until the column resists movement. One-quarter turn past finger tight is about right.
8. Adjust the head pressure to obtain the flow rate listed on the test chromatogram.
9. Check the inlet connections for leaks.
10. Confirm gas flow through the column by observing bubbles when the column outlet end is immersed in a nontoxic solvent such as acetone.
11. Purge the column with carrier gas for approximately 15 minutes. Repeat step 5. Then insert the outlet end of the column into the detector exactly the distance prescribed in the instrument manual.
12. Set gas-flow rates to instrument specifications.
13. Check the system for leaks. It is preferable to use a thermal-conductivity-type leak detector.
14. Set detector temperature. ***Warning!*** *Some detectors can be damaged by heating without proper gas flow.* Turn the detector on when steady-state temperatures are achieved.
15. Increase the oven temperature to the maximum continuous operating temperature for the column. Maintain maximum temperature until a flat baseline is observed. If the detector signal does not drop in 10 minutes, immediately cool the column and check for leaks.
16. Inject a detectable unretained sample such as methane to determine dead volume time and linear gas velocity. Adjust gas pressure to obtain proper values for your analytical method.
17. Set oven to starting temperature. Inject another sample of a detectable unretained substance. Reset the carrier gas velocity to desired value.
18. Check the performance of the GC and the column by injecting a known sample. If all peaks tail, it could indicate loose fittings, improper column installation, or broken liner.
19. Calibrate the instrument.

Tip:

For fast and accurate GC installation, use the Cool-Lock™ Nut.

V. SAMPLE INJECTION

Much of the trouble of getting quantitative results with good peak shapes is due to the selection of an inappropriate sample injection mode. There is no single sample injection mode that accommodates the great variety of capillary columns, as well as the enormous diversity of samples, that can be analyzed by gas chromatography. As a result, the operator must select an appropriate injection mode for each type of sample and GC column.

The objectives are:

- to introduce the sample into the column so that it retains its original composition (i.e. there should be no sample degradation or selective losses during injection), and
- the sample should initially occupy the shortest possible length of column. The shorter the initial sample band, the sharper the peaks on the chromatogram. Sharper peaks result in greater sensitivity and better resolution.

Several injection modes have been invented in attempts to reach these objectives for specific types of samples. The most popular modes are:

- Split injection
- Splitless injection
- On-column injection
- Direct injection
- Programmed temperature vaporizing injection

Split injection

The split injection mode is among the most common. A sample introduced into a split injector is rapidly vaporized and mixed with carrier gas. Most of the sample is simply 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".) The total gas flow through the injector during injection is high and the sample is swept rapidly onto the column. This rapid sample introduction provides the basis for sharp peaks and good resolution. However, the rapid sample handling also leads to discrimination against the less volatile compounds which do not fully vaporize before they are discarded through the split vent, so this technique may be inappropriate if sample components vary widely in their boiling points.

Injector temperatures

The temperature of an injector should be hot enough to ensure rapid vaporization of the entire sample, but not too hot to degrade any of the components. Some experimentation may be required to determine a temperature which will minimize backflash and reduce discrimination against the less volatile compounds which do not fully vaporize before they are discarded through the split vent. For many samples, a satisfactory injector temperature is 250 °C.

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 injector, the less the discrimination but the broader the peaks in the chromatogram.

Backflash
The term "backflash" refers to vapors from the vaporization of the sample that expand to exceed the volume of the injector liner. When this occurs, the gases may come in contact with cold spots, like the septum and gas inlets to the injector. Some of the less volatile components may condense. These condensates may vaporize later and interfere with subsequent analyses, sometimes producing “ghost peaks.”

Expansion outside the injector liner may also expose the sample to metal surfaces which are not inert, and active components of the sample may be lost.

Backflash can be minimized by using septum purge, small injection volumes, large volume injector liners, and optimal injector temperatures.

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

Sample size and 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 diluent used. If the sample volume is too large, backflash may occur.

Splitless injection

In splitless injection, the entire flow through the injector passes into the column for the first 15 to 90 seconds. This long injection would lead to very broad peaks if the sample was not refocused before the chromatographic process started.

Refocusing (Solvent effects and cold trapping)
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 diluent. Then, when the solvent vapor leaves the injector and enters the cooler column, the solvent condenses at the front of the column as a liquid band. Solute vapors will condense in this band and be trapped and refocused. This process is called the “solvent effect.”

Peaks that elute before the solvent will not be refocused by the solvent effect and may be malformed. Using a lower boiling solvent may obviate this difficulty. If the solvent does not wet the stationary phase (column lining) sufficiently, as might be the case for methanol used with a nonpolar stationary phase, the solvent flooded zone may be several meters long and not of uniform thickness. This will result in broad and distorted peaks because the solutes will not be refocused to a narrow band near the beginning of the column. Installing a retention gap (a length of uncoated but deactivated column) ahead of the chromatographic column may reduce or eliminate this problem.

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 size
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.

On-column injection

Properly carried out, on-column injection can provide extremely accurate results. Syringe discrimination and inlet-related discrimination are eliminated. However, if polar solvents are used with non-polar column linings, a retention gap is recommended.

If the solvent wets the stationary phase and the injection is carried out at an initial column temperature below the boiling point of the solvent, the sample is initially distributed over a flooded zone at the beginning of the column. The less volatile components are distributed in the stationary phase of the column. Under the influence of the carrier gas, the solvent evaporates beginning at the inlet end of the column. As the solvent evaporates, the more volatile components become concentrated in the shrinking solvent at the upstream edge of the flooded zone where they are refocused by solvent trapping.

Clearly, the distribution of solutes in the area of the flooded zone is not homogeneous and this leads to broadening of the peaks. This peak broadening can be neglected for many applications and good quantitative results can be obtained using on-column injection.

If the boiling points of the solutes differ greatly from that of the solvent, ballistic heating to high temperature can be employed. If the solutes’ boiling points are close to that of the solvent, temperature programming can be used to fully exploit the solvent effect.

V. SAMPLE INJECTION (continued)

The use of wide-bore columns will make on-column injection easier, and if the need for high resolution precludes their use, a deactivated but uncoated wide-bore precolumn 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.

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.

Programmed temperature vaporizing injection

In programmed temperature vaporizing 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.

VI. SOLVENT RINSING

Solvent rinsing will remove most soluble contaminants and often restore column performance. This should be used as a last resort. In most cases, it is better to simply replace the GC column.

Warning! *The column must have cross-linked stationary phase lining that is bonded to the capillary walls to withstand solvent rinsing.*

Solvents can be forced through the column by pressurizing a vial of the solvent into which the capillary column is sealed with 10 to 15 psi pressure. Kits for rinsing columns are available from a number of GC manufacturers and column suppliers.

A series of solvents should be used in rinsing the column. Start with the most polar solvent and finish with the least polar. Include the injection solvent, if practical. Each successive solvent must be miscible with the one previously used. Begin with water followed by methanol if water-based samples (or samples extracted from aqueous solution) have been injected.

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. However, after the last solvent has been removed the column should be purged with pure carrier gas for about 10 minutes before reinstalling in the chromatograph. Program the oven temperature at 2 °C/min until the normal condition temperature is reached; then condition the column as usual.

VII. PREVENTIVE MAINTENANCE

CLEANING THE DETECTOR

Procedures for cleaning a detector depend on its type. Choose from the following list:

- ECD
- FID
- FPD
- NPD
- TCD

Cleaning ECD detectors

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.

Cleaning FID detectors

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

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.

Cleaning FPD detectors

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

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.

VII. PREVENTIVE MAINTENANCE (continued)

Cleaning FPD detectors (continued)

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.

Cleaning NPD detectors

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.

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

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.

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 the detector and use clean compressed gas to remove loose material from the inside of the collector. Remember to wear eye protection when using compressed gas.
5. Do not disturb the active element. Do not use brushes or wires to clean this type of detector. Do not touch the lower end of the collector (the end nearest the jet), because fingerprints may cause baseline drift or noise.
6. Wash the collector with non-polar solvents such as hexane or isooctane. Avoid polar solvents, especially water, because they may dissolve the rubidium salt coating on the active element.
7. Wash the internal bore and the exterior of the jet with an equal mixture of methanol and acetone. Blow off excess solvent with clean dry gas and dry the jet in an oven at about 70 °C for more than half an hour.
8. Clean the detector base cavity with a swab and organic solvents. Blow dry with compressed gas.
9. Reinstall the jet.

Cleaning TCD detectors

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

Cleaning a TCD detector is limited to baking it out after verifying there are no leaks and the carrier gas is clean and dry.

VII. PREVENTIVE MAINTENANCE (continued)

Cleaning TCD detectors (continued)

Cleaning procedure:

1. Turn off detector.
2. Remove the column from the detector and cap the detector column fitting.
3. Establish proper reference gas flow (about 25 mL/min) through the detector.
4. Set oven temperature to about 250 °C.
5. Heat the detector to 400 °C for several hours.

Cleaning the inlet

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.

* 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.

VIII. PHENOMENEX ZEBRON™ CAPILLARY GC COLUMNS

Engineered Self Cross-Linking™ (ESC) polymer technology gives Phenomenex Zebron™ Capillary GC columns significant advantages over competitive columns:

Standard phase chemistry

- Similar selectivity to other manufacturer's columns
- Fast method transfer from standard columns

Low bleed

- Increased sensitivity for trace analysis
- Reduced detector contamination
- Low noise contribution to MS signals
- Fast baseline equilibration
- Flat baselines during temperature programming

Extended upper temperature limits

- Reduced analysis times
- Fast purge of high molecular weight fractions
- Extended column lifetime

Unique phases chemistry

- Unique phases designed for specific applications
- Provides enhanced separation of compounds

Phenomenex Zebron GC columns are certified for mass spec detection

Phenomenex offers a polymer technology designed to exceed the thermal and bleed barriers with minimal changes to the chromatographic selectivity. Our enhanced columns have great durability and rinseability with extremely low bleed levels.

SELECTING THE RIGHT STATIONARY PHASE

APPLICATIONS	ZEBRON PHASE	COMPOSITION	POLARITY	TEMPERATURE RANGE*	MS CERTIFIED
Essential oils, gases (refinery), hydrocarbons, MTBE, natural gas odorants, oxygenates and GROs, semi-volatiles, solvent impurities, sulfur compounds (light)	**ZB-1**	100% dimethyl-polysiloxane	Nonpolar	-60 to 360/370 °C	✔
Amines, acids, diesel fuel, drugs of abuse, flavors & fragrances, pesticides, Polychlorinated Biphenyls (EPA 1668)	**ZB-1ms**	100% dimethyl-polysiloxane	Nonpolar	-60 to 360/370 °C	✔
High boiling petroleum products, simulated distillation methods, hydrocarbons, high molecular weight waxes, polymers/plastics, motor oils, diesel fuel	**ZB-1HT Inferno™**	100% dimethyl-polysiloxane	Nonpolar	-60 to 400/430 °C	✔
Petroleum analysis, simulated distillation, hydrocarbons	**ZB-1XT SimDist**	100% dimethyl-polysiloxane	Nonpolar	-60 to 450 °C	✔
Alkaloids, dioxins, drugs, essential oils/flavors, FAMEs, halo-hydrocarbons, PCBs/aroclors, pesticides/herbicides, phenols, residual solvents, semi-volatiles, solvent Impurities	**ZB-5**	5%-phenyl-95%-dimethyl-polysiloxane	Nonpolar	-60 to 360/370 °C	✔
Drugs of abuse, FAMEs, Nitrosamines, phenols, pesticides, EPA methods	**ZB-5MSi**	5%-phenyl-95%-dimethyl-polysiloxane	Nonpolar	-60 to 360/370 °C	✔
Acids, alkaloids, amines, dioxins, drugs,essential oils/flavors, EPA Methods, FAMEs, halo-hydrocarbons, PCBs/aroclors, pesticides/herbicides, phenols, Polyaromatic Hydrocarbons (PAH), residual solvents, semi-volatiles, solvent impurities	**ZB-5ms**	5%-phenyl Arylene-95%-dimethyl-polysiloxane	Nonpolar	-60 to 325/350 °C	✔
High boiling petroleum products, triglycerides, simulated distillation methods, diesel fuel, long-chained hydrocarbons, motor oils, polymers/plastics, surfactants, high molecular weight waxes	**ZB-5HT Inferno™**	5 %-phenyl-95 % dimethyl-polysiloxane	Nonpolar	-60 to 400/430 °C	✔
Aroclors, amines, nitrogen containing pesticides, organochlorine pesticides, organophosphorous pesticides, pharmaceuticals, semi-volatiles, EPA Method 508, 608, 8081, 8141, 8151	**ZB-35**	35%-phenyl-65%-dimethyl-polysiloxane	Low-Mid Polar	40 to 340/360 °C	✔
Aroclors, semi-volatiles, amines, EPA Methods 508, 608, 8081, 8141, 8151, pesticides, drugs of abuse, pharmaceuticals, steroids, chemicals	**ZB-35HT Inferno**	35%-phenyl-65%-dimethyl-polysiloxane	Low-Mid Polar	40 to 400 °C	✔
Antidepressants, aroclors, cholesterols, drugs of abuse (especially basic), glycols, nitrogen containing pesticides, organochlorine pesticides, organophosphorous pesticides, steroids, triglycerides, EPA Method 508, 608, 8081, 8141, 8151	**ZB-50**	50%-phenyl-50%-dimethyl-polysiloxane	Inter-mediate	40 to 320/340 °C	✔
Volatile organic compounds (VOCs). Widely used phase to separate residual solvents in industrial or pharmaceutical products (OVIs). Excellent for US EPA Methods 501.3, 502.2, 503.1, 524.2, 601, 602, 624, 8010, 8015, 8020, 8240, 8260, 8021	**ZB-624**	6%-cyanopropyl-phenyl-94%-dimethyl-polysiloxane	Inter-mediate	-20 to 260 °C	
Alcohols, amines, aromatic hydrocarbons, nitrogen containing pesticides, organophosphorous pesticides, PAHs, PCBs/aroclors, pesticides/herbicides, phenols, steroids, tranquilizers	**ZB-1701**	14%-cyanopropyl-phenyl-86%-dimethyl-polysiloxane	Polar	-20 to 280/300 °C	
Organochlorine pesticides	**ZB-1701P**	14%-cyanopropyl-phenyl-86%-dimethyl-polysiloxane	Polar	-20 to 280/300 °C	
Alcohols, aldehydes, aromatics, essential oils, flavors & fragrances, glycols, OVIs, pharmaceuticals, solvents, styrene, xylenes isomers	**ZB-WAX**	polyethylene glycol	Polar	40 to 250/260 °C	✔
Alcoholic beverages, alcohols, OVIs, aldehydes, aromatics, acids (free), essential oils, flavors / fragrances, styrene, glycols, pharmaceuticals, solvents, xylene isomers	**ZB-WAX-*PLUS*™**	polyethylene glycol	Polar	20 to 250/260 °C	
Acrylates, alcohols, aldehydes, free fatty acids, glycols, ketones, organic acids, phenols, volatile free acids	**ZB-FFAP**	nitroterephthalic acid modified polyethylene glycol	Polar	40 to 250/260°C	
Organochlorine pesticides, insecticides, organophosphorous, pesticides, aroclors/ PCBs, nitrogen containing pesticides, haloacetic acids, herbicides, multi-pesticide residue methods	**ZB-Multi-Residue™-1/2**	Proprietary	Low-Mid Polar	-60 to 320/340 °C	✔
Polychlorinated Biphenyls (PCBs), pesticides, herbicides, EPA methods	**ZB-XLB**	Proprietary	Low-Mid Polar	30 to 340/360 °C	✔
Polychlorinated Biphenyls (PCBs), pesticides, herbicides, EPA methods	**ZB-XLB-HT Inferno**	Proprietary	Low-Mid Polar	30 to 360/400 °C	✔
Blood alcohol analysis, glycols, abused inhalant anesthetics, industrial solvents	**ZB-BAC1/2**	Proprietary	Low-Mid Polar	-20 to 260/280 °C	✔
Bioethanol, alcohol, fusel alcohols	**ZB-Bioethanol**	Proprietary	Low-Mid Polar	-60 to 340/360 °C	✔
Drugs of abuse	**ZB-Drug-1**	Proprietary	Low-Mid Polar	-60 to 320/340 °C	✔

*** See individual phases for detailed specifications and limitations.**

guarantee

If Zebron does not provide at least equivalent separations as compared to a competing column of the same phase and dimension, return the column with comparative data within 45 days for a FULL REFUND.

IX. GC COLUMN SELECTION GUIDE

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.

Composition	ZEBRON	Restek	J&W	Supelco	Agilent Technologies (HP)	Alltech	SGE	Varian (Chrompack)
100 % dimethyl-polysiloxane	ZB-1	Rtx-1, Rtx-1PONA, Rtx-1 F&F	DB-1, DB-2887, DB-1 EVDX	SPB-1, SPB-1 TG, SE-30, MET-1, SPB-1 Sulfur, SPB-HAP	HP-1, HP-101, HP-PONA, Ultra 1	AT-1, AT-Sulfur, EC-1	BP1, BP1-PONA, BPX1-SimD	CP-Sil 5 CB
100 % dimethyl-polysiloxane	ZB-1ms	Rtx-1ms	DB-1ms	MDN-1, Equity-1	HP-1ms	AT-1ms	SolGel-1ms	CP-Sil 5 CB MS, VF-1ms
100 % dimethyl-polysiloxane	ZB-1HT Inferno	MXT-1 SimDist	DB-HT Sim Dis					CP-SimDist Ultimetal
100 % dimethylpolysi-loxane	ZB-1XT SimDist	MXT-1HT Sim Dist	DB-1ht	Petrocol 2887				CP-SimDist
5 %-phenyl-95 %-dimethylpolysiloxane	ZB-5	Rtx-5	DB-5	MDN-5, SPB-5, PTE-5, SE-54, PTA-5, Equity-5, Sac-5	HP-5, Ultra 2, HP-PAS-5	AT-5, EC-5	BP5, BPX5	CP-Sil 8 CB
5 %-phenyl-95 %-dimethylpolysiloxane	ZB-5 MSi	Rtx-5ms, Rtx-5Amine, Rxi-5ms	DB-5	MDN-5S	HP-5ms, HP-5msi			
5 %-phenyl-95 %-dimethylpolysiloxane	ZB-5HT Inferno	Stx-5HT, XTI-5HT	DB-5ht	HT-5				VF-5ht
5 %-phenyl-Arylene-95 %-dimethylpolysiloxane	ZB-5ms	Rtx-5SilMS	DB-5ms, DB-5.625, DB-5ms EVDX					VF-5ms, CP-Sil 8 CB MS
35 %-phenyl-65 %-dimethylpolysiloxane	ZB-35	Rtx-35, Rtx-35ms	DB-35, DB-35ms	MDN-35, SPB-35, SPB-608	HP-35, HP-35ms	AT-35	BPX35, BPX608	
Proprietary ZB-35HT Inferno (no equivalent)	ZB-35HT Inferno							
50 %-phenyl-50 %-dimethylpolysiloxane	ZB-50	Rtx-50	DB-17, DB-17HT, DB-17ms, DB-17 EVDX	SP-2250, SPB-17, SPB-50	HP-50+	AT-50	BPX50	CP-Sil 24 CB
6 %-cyanopropylphenyl-94 %-dimethylpolysiloxane	ZB-624	Rtx-1301, Rtx-624	DB-1301, DB-624, DB-VRX	SPB-1301, SPB-624	HP-VOC	AT-624, AT-1301	BP624	CP-1301, CP-Select-624 CB
14 %-cyanopropylphenyl-86 %-dimethylpolysiloxane	ZB-1701	Rtx-1701	DB-1701	SPB-1701, Equity-1701		AT-1701	BP10	CP-Sil 19 CB
14 %-cyanopropylphenyl-86 %-dimethylpolysiloxane	ZB-1701P		DB-1701P					
Polyethylene glycol	ZB-WAX	Rtx-WAX, Famewax, Stabilwax-DB	DB-WAXetr	Met-Wax, Omegawax	HP-INNOWax	EC-Wax	SolGel-WAX	CP-Wax 57 CB
Polyethylene glycol	ZB-WAX*PLUS*	Stabilwax	DB-WAX, CAM	Supelcowax 10	HP-20M, Carbowax-20M	AT-Wax, AT-AquaWax	BP20	CP-Wax 52 CB
Nitroterephthalic acid modified polyethylene glycol	ZB-FFAP	Stabilwax-DA	DB-FFAP	Nukol, SPB-1000	HP-FFAP	AT-1000, EC-1000	BP21	CP-Wax 58 CB
Proprietary	MultiResidue-1 (MR-1)	Rtx-CLPesticides, Stx-CLPesticides						
Proprietary	MultiResidue-2 (MR-2)	Rtx-CLPesti-cides2, Stx-CLPesticides2						
Proprietary	ZB-XLB	Rtx-XLB	DB-XLB	MDN-12				VF-Xms
Proprietary	ZB-XLB-HT Inferno							
Proprietary	ZB-BAC1	Rtx-BAC1	DB-ALC1					
Proprietary	ZB-BAC2	Rtx-BAC2	DB-ALC2					
Proprietary	ZB-Bioethanol							
Proprietary	ZB-Drug-1							

X. INDEX

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