Bulletin 853B

Capillary GC Troubleshooting Guide: How to Locate Problems and Solve Them

The real task in correcting a problem with your capillary GC system is identifying the cause of the problem without wasting time. The systematic approach to troubleshooting described in this guide will enable you to solve many problems yourself. The guide also contains suggestions for maintaining your system, including the column, at optimal performance levels. By following these recommendations, you can reduce repair costs and instrument down time.

Contents

Troubleshooting Suggestions

Make troubleshooting faster and easier by closely observing and keeping complete records of instrument operation (temperatures, linear gas velocities, chart speeds, column dimensions, stationary phase type and film thickness, etc.). Reliable records on system maintenance (septum changes, inlet liner changes, etc.) are equally important.

Troubleshooting will also progress more smoothly if you have on hand:

- *An electronic leak detector* – A leak-free system is mandatory for good performance. Use the detector to test all fittings for leaks. NEVER use liquid leak detectors with a capillary GC system. They can severely contaminate your column or other system components.
- *A flow meter* – There are many gas flows to monitor in a capillary GC system. Use a flow meter to check gas flows from the splitter, septum purge vent, and detector.
- *An accurate thermometer* – Routinely monitor the oven temperature to be sure settings are correct and temperature control is effective.
- *A reliable analytical column* – Ideally, this should be identical to the column you are using when you discover the problem, but it can be any reliable column. If the replacement column performs well, you will have isolated the problem to the original column.
- *New syringes* – Repeat the analysis with a new, clean syringe. If the trouble (e.g., ghost peaks) disappears, examine and clean the original syringe according to the procedures in the *Tips to Help Prevent Problems* section of this guide.
- *Spare septa and high temperature septa* – Replace the septum in your instrument to determine if the problem (e.g., extra peaks, poor reproducibility) is caused by a leaking or bleeding septum.
- *Spare ferrules* – Use these, when necessary, to eliminate leaks.
- *Detector cleaning solutions* – A dirty detector produces noisy baselines. Routine cleaning is excellent preventive maintenance.
- *Spare recorder and electrometer cables* – Use these to test the recording system as a trouble source.
- *Instrument manuals* – Refer to these for flow rates, insertion distances, and other instrument-specific conditions.

Also valuable, when you can obtain them, are reference standards containing the components in your sample – without extraneous or unknown components. You can waste many hours looking for problems in your instrument or column when the problem might very well be in the sample you are analyzing (e.g., incomplete clean-up, wrong solvent, etc.) If your system separates a reference standard well and reproducibly, the problem is probably sample related.

Isolating the Problem Source

Establish a Systematic Approach

Carefully note the symptoms you are encountering (e.g., broad peaks, long retention times, etc.), then find these in the index at the front of the Troubleshooting Table (page 9). Consult the table for possible causes for each symptom, then eliminate the possibilities one at a time. If one cause is common to all symptoms, this is most likely the problem.

The troubleshooting table contains most of the problems you will encounter, but not every situation can be anticipated. If your problem is not covered in the table, you can still systematically isolate the cause. There are five sources of problems in gas chromatography: the operator, the sample, the column, the instrument electrical systems, and the gas flow system. Eliminate these one by one to isolate the source of your problem.

1. Rule out *operator error* by double checking all operating parameters (temperatures, gas flow rates, column identity, etc.)
2. Identify *sample-related problems* by injecting a reference standard. If the chromatogram is good, the problem is most likely sample related. If the chromatogram is poor, the problem is probably column or instrument related.
3. To check for a *column problem*, replace the column with one you know will provide good results under proper conditions. If you do not have a duplicate column, use any good column that will provide an acceptable analysis of your sample. (If you must change the sample for compatibility with the replacement column, however, you cannot rule out the sample as the problem source.) If you obtain good results, the problem is related to the original column. If the symptom persists, the problem is instrument-related.
4. Isolate *electrical system-related problems* by listing the systems that could cause the symptoms (e.g., for broad peaks with long retention times: column heating system, injection port heating system), then evaluating the performance of each system. Remember, in some instruments it is easier to check the gas supply system (step 5, next page) before checking the electrical systems.

Check the electronic system (detector, electrometer, recorder, and associated wiring) for malfunctions. Use the following procedure. If your instrument is equipped with dual channels (two detectors, electrometers, recorders, etc.) refer to paragraph (c).

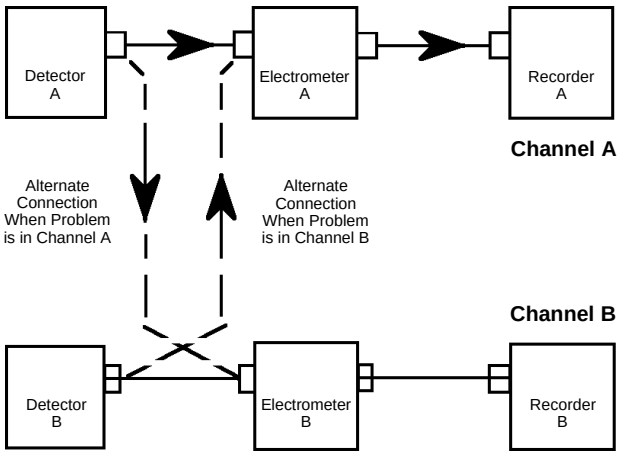
(a) Set the chromatograph attenuation to infinity. The recorder pen should go to electronic zero. If the symptom (baseline drift, etc.) continues, refer to the recorder instruction manual. If the symptom disappears, the problem is not in the recorder.

(b) Turn off the detector and disconnect the detector-electrometer cable at the detector end. (To prevent introducing extraneous noise onto the cable, it may be necessary to install a coaxial cap on the free end, or wrap it with aluminum foil.) Turn the power on. If the symptom disappears, the detector is causing

the problem. If the symptom continues, turn off the power, reconnect the cable to the detector, then disconnect it at the electrometer end. Turn the power on. If the symptom *now* disappears, the cable is defective and must be replaced. If the symptom continues, the electrometer is causing the problem. If you have traced the problem to the detector or electrometer, refer to the appropriate instrument manual for help.

(c) If your chromatograph is equipped with dual detector channels, you have a simple but effective alternate means of identifying problems in the electronic system (Figure A). Disconnect the detector-electrometer cables at the detectors. If the symptom occurs in channel A, connect the electrometer B cable to the outlet of detector A. The signal from detector A will be applied to electrometer and recorder B. If the symptom does not appear on recorder B, the problem is in the channel A electrometer, recorder, or cables. If the symptom is not eliminated, the problem is in the channel A detector. If the symptom occurs in channel B, connect the electrometer A cable to the outlet of detector B. The signal from detector B will be applied to electrometer and recorder A. If the symptom does not appear on recorder A, the problem is in the channel B electrometer, recorder, or cables. If the symptom is not eliminated, the problem is in the channel B detector.

Figure A. Checking Electronic Components of a Dual Detector System



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5. If the electrical systems are functioning properly, refer to the following sections and carefully check all components of the *gas supply system*. Verify that the following flow rates are correct:
   - column flow
   - linear gas velocity
   - split flow
   - septum purge flow
   - make-up gas flow
   - detector air and hydrogen flows

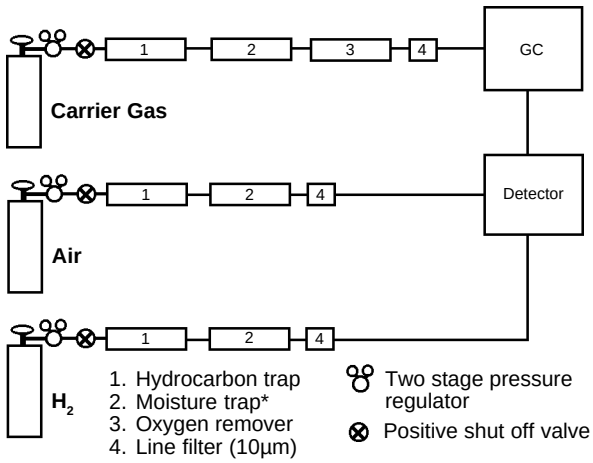
The split, make-up, and detector flows are high enough to be measured with the same bubble flow meters used with packed column systems. The septum purge and column flow rates will require a capillary column flow meter. The average linear gas velocity through the column is determined from the holdup time for an unretained compound.

Check the Carrier Gas Supply for Leaks

Figure B shows a gas supply system for a capillary GC equipped with a flame ionization detector. To prevent problems, all fittings in this system must be leak tight and each component must be functioning correctly. Preventive maintenance is an important part of a trouble-free gas supply system. You should use only high quality gases, and pass these through high performance purifying devices to remove any trace of oxygen, moisture, and hydrocarbons. For more information about purifying gases, request **Bulletin 848**.

Begin checking the carrier gas supply by reading the pressure gauge, to ensure that the chromatograph is being properly supplied. The pressure at the gauge should exceed the pressure needs of the chromatograph by at least 15psig. Next, check the external fittings along the carrier gas line for leaks, using an electronic leak detector. *Never use liquid leak detectors with your capillary GC equipment – you risk seriously contaminating the system.* Carefully and slowly move the probe around each connection. Check around column loops to be sure the column is not broken.

Figure B. Typical Gas Supply for a Capillary GC System (Flame Ionization Detector)



NOTE: The functions of a moisture trap and an oxygen remover are combined in our OMI-2 and High Capacity gas purifiers (see products pages).

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As a further check for leaks, turn off the carrier gas supply at the cylinder. In a leak free system, the line pressure will fall slowly (30 seconds or longer). If there is a valve connecting the carrier gas line to the chromatograph, allow the chromatograph to cool, turn off the valve, *then* turn off the cylinder valve. Under these conditions, the pressure in a leak free system will drop only one or two pounds over several hours. A rapid loss of pressure indicates leaks are present. Recheck the fittings using the electronic detector. Seal any leaks you find and recheck the system's ability to maintain pressure. Repeat this process as necessary until the system maintains pressure.

Verify Column Flow and Linear Gas Velocity

Connect a capillary bubble or digital flow meter to the column or detector outlet. Determine the flow rate through the column from the formula below. Adjust the rate as necessary.

$$\text{flow rate (mL/min.)} = \frac{\text{distance bubble travels (mL)}}{\text{time (sec.)}} \times 60$$

Remember that gas flow through most capillary gas chromatographs is pressure regulated. Because gas viscosity changes with temperature, it is important to know the temperature at which the average linear velocity, u, is set. Turn on the instrument and adjust the oven temperature accordingly. Inject an unretained compound onto the column and use the holdup time to determine the linear gas velocity, u, from the formula below. A good rule is to make three injections of small amounts of the unretained compound, with short time intervals between them, and compare the retention times for consistency. If the retention times are consistent, compare the average value to the column manufacturer's recommendations.

$$\text{u(cm/sec.)} = \frac{\text{column length (cm)}}{\text{holdup time for unretained compound (sec.)}}$$

Use methane as the unretained compound when you are using a flame ionization or thermal conductivity detector. Inject 25-50µL of 1% methane in nitrogen onto the column. For other detectors, other compounds provide better response than methane: Freon® 12, sulfur hexafluoride, dichloromethane, or air for electron capture detectors, acetonitrile for nitrogen-phosphorous detectors, air for GC/MS systems. These compounds are virtually unretained by most columns.

If the linear gas velocity does not meet the requirements of your method, adjust the flow or pressure controls. If you cannot obtain an adequate gas flow by adjusting the flow controller, the source pressure probably is too low. Increase this pressure until the flow is adequate. Normally, a source pressure of 60psig is sufficient. If not, there may be a leak or other problem in the system.

Use an electronic gas leak detector to check for leaks around fittings, columns, flow controllers, and valves. Also check to make sure the column is not broken within the fittings. You can repair a broken column by using a capillary butt connector, but the column could have been damaged by exposure to oxygen and moisture in the air before you discovered the break. If you repair a column, evaluate its performance with a test mix (see Appendix) before using it to analyze your samples.

If the gas flow at the detector end of the column is inadequate, but the column is intact, the column or the inlet liner may be blocked with septum or ferrule fragments or dirt from the sample. Remove the column and examine it for obstructions. Check several loops at each end and cut off plugged segments, making a clean, square cut. Exposed polyimide at a jagged edge can adsorb sample components. If your column has a bonded phase, you can use certain solvents to flush out small particles and remove soluble, adsorptive deposits. Consult the column manufacturer's instructions before rinsing the column.

If the column is clean, remove the inlet liner, examine it, and replace or clean and redeactivate it if necessary.

NOTE: The surface of an untreated inlet liner contains active metal ions and silanol groups. Chromatography of active solutes (i.e., free acids, compounds containing O, S, N, or other electron donors) will be improved if liners and liner packings are properly deactivated with a suitable silanizing reagent, such as dimethylchlorosilane. We deactivate every liner we provide, but some suppliers do not.

If the gas flow is adequate at the column outlet, but not at the detector outlet, the detector jet may be blocked. Remove the jet and clean or replace it. When measuring flow out of the detector, be sure the detector is neither undertightened nor overtightened. Some detectors do not have gas-tight seals. Consult your instrument manual for sealing instructions.

Check the Make-up Gas Supply

Most flame ionization detectors have been designed for packed column instruments. Capillary column gas flows do not adequately sweep the internal volumes in these detectors. To eliminate dead volumes and ensure an adequate flow for optimal detector operation, flow from the capillary column is supplemented with a flow of make-up gas. Check the make-up gas flow with the column-detector connection closed off, and adjust the flow to the detector manufacturer's recommendations. Total detector flow (column flow plus make-up gas), in most cases, should be about 30cc/minute.

It is not always advantageous to use the same gas as both the carrier gas and the make-up gas. The appropriate make-up gas can increase the signal-to-noise ratio of a detector. For best sensitivity, we recommend nitrogen for FIDs (it increases sensitivity, relative to helium, because it cools the flame), argon/methane or nitrogen for ECDs, and helium for nitrogen/phosphorus and flame photometric detectors. Be sure to use only high purity gases and high performance gas purifiers.

Minimize Dead Volumes

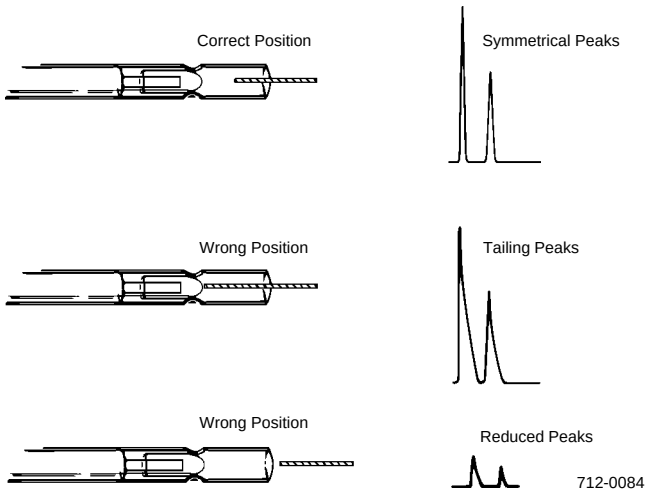
Inexperienced users of capillary columns often forget that dead or unswept volumes in the sample flow path can be a significant source of problems. Dead volume can make the efficiency of an excellent column appear to be lower than that of an average column. Because flow rates through capillary columns are usually very low (as low as 0.3mL/minute), sudden changes in the volume of the gas path must be avoided or the consequences of such changes must be minimized. For example, if you are using a sample splitter, you must position the column inlet end in the mixing chamber, where gas velocities are high (Figure C). Consult your instrument manual for correct insertion distances.

To eliminate dead volume and avoid active sites in the detector transfer line, insert the outlet end of the column into the detector jet. Every instrument has an optimum insertion distance that can be determined by consulting the instrument manual or the

manufacturer, or by experimentation. Inserting the column incorrectly into some detectors, such as an electron capture detector (ECD), or into the ion source of a GC/MS system, can reduce sensitivity.

For installation into a flame ionization detector, the column end should be about 2mm below the jet tip. If you are using a small diameter fused silica column, be sure the column does not extend through the jet tip and into the flame (Figure D). This would

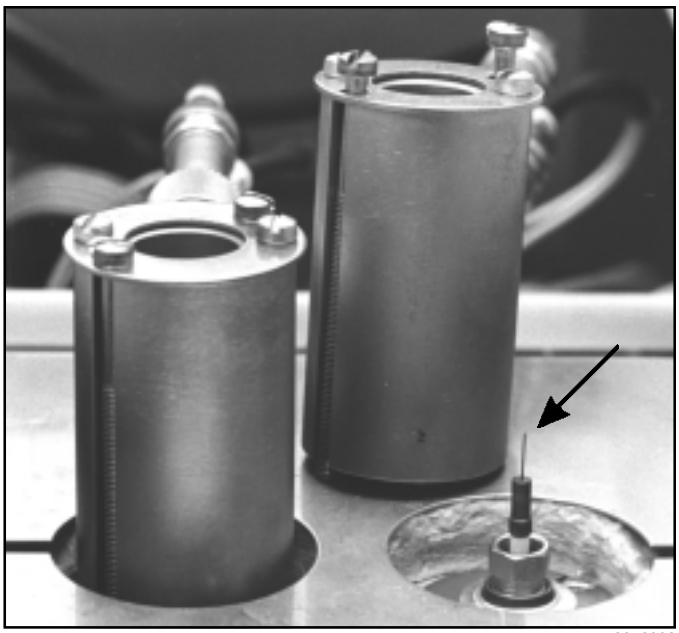
Figure C. Correctly Position Column Inlet in Sample Splitter to Minimize Dead Volume Effects



pyrolize or carbonize both the stationary phase and the column's outer coating, producing a highly adsorptive outlet. In some detectors, transfer lines with right angle turns or very small inner diameters prevent insertion of the column into the detector. Consult the manufacturer about overcoming this problem, perhaps with a part substitution.

Wide bore (0.53mm and 0.75mm ID) capillary columns are often used in instruments designed for packed columns. The outlet end of a 0.53mm ID column, or a 0.75mm ID without a 0.32mm ID

Figure D. Column Outlet Extending Beyond the Jet Tip in a Flame Ionization Detector



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guard column, will not fit into the detector beyond the hydrogen (FID) or scavenger (ECD) inlets in most packed column instruments. Therefore, make-up gas must be added to the column effluent to rapidly flush the effluent toward the detector.

To connect a wide bore capillary column to the injection port of your packed column GC, use a Direct Injection Conversion Kit (Figures E and F). In two-column instruments, the make-up gas can be introduced from the second injection port and regulated with the flow controller for that port. Alternatively, make-up gas can be supplied from an external source and controlled with a needle valve (Figure F). Regardless of the plumbing, be sure that the column end is in the high velocity area.

Figure E. Cross Section of Direct Injection Conversion Kit and Column After Installation

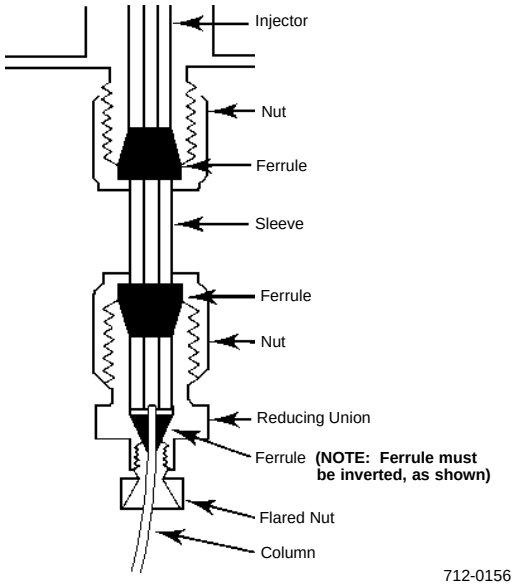
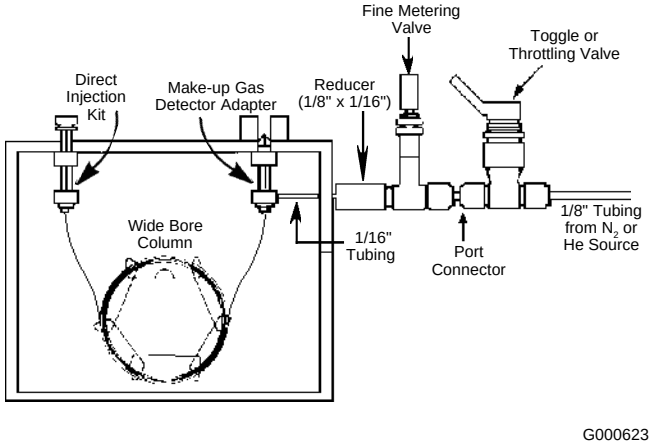


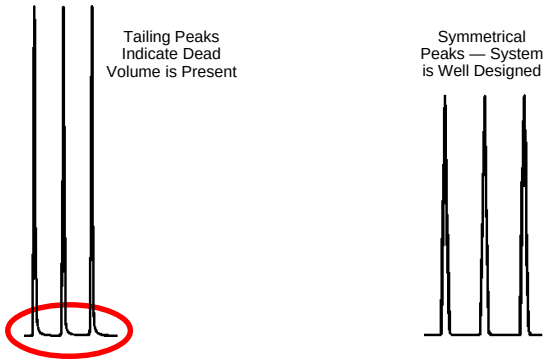
Figure F. Packed Column Chromatograph Converted for Use with Wide Bore Capillary Column



Use Methane to Reveal Dead Volumes

In addition to providing information needed to calculate the linear gas velocity, injections of methane or another unretained compound show the quality of the column installation. If the system is properly connected, the peak will be symmetrical. Dead volumes, leaks, or obstructions will produce tailing peaks (Figure G). A high chart speed makes it easier to accurately evaluate peak shape.

Figure G. Methane Injections Reveal Dead Volume



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Tips to Help Prevent Problems

Selecting a Carrier Gas

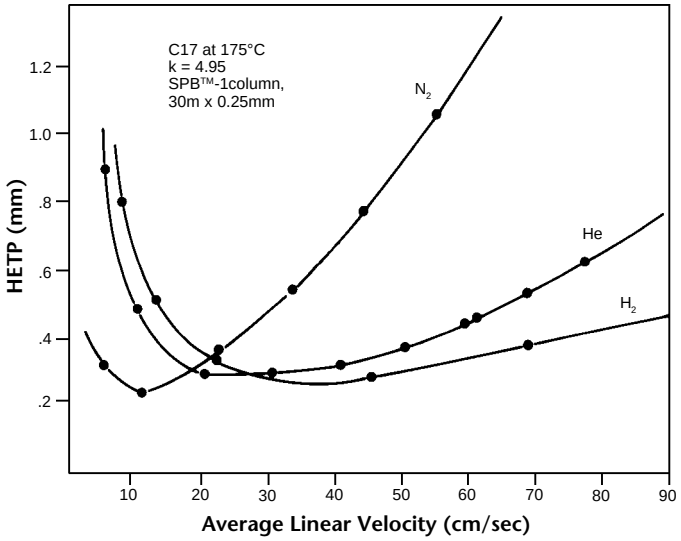
With few exceptions, hydrogen is by far the most suitable carrier gas for capillary chromatography. The van Deemter curves in Figure H show that deviations from the optimum linear velocity for hydrogen have a smaller effect on column efficiency than comparable changes for helium or nitrogen. Furthermore, because the optimum velocity for hydrogen is higher than that for helium or nitrogen, hydrogen can provide better separations in less time than either of the other gases.

Helium is the second best carrier gas, and is popular because no safety precautions are needed. In a few situations, helium is the preferred carrier gas. It is preferred with GC/MS systems, for example, because it has a high ionization potential. Compared to hydrogen flow, helium flow is less dependent on temperature, and thus is usually the preferred carrier for plasma-type nitrogen/phosphorus detectors, for which response is critically dependent on carrier gas flow.

Nitrogen's lower diffusivity makes it possible to obtain high efficiency only within a very narrow range of low rates (Figure H). Thus, nitrogen should rarely be used as a carrier gas with capillary columns.

REMEMBER: Hydrogen forms explosive mixtures with air at concentrations of 4-10%. Although its high diffusivity makes it unlikely that such concentrations will be attained, do not disregard the danger. Precautions are especially important if your instrument is fitted with rigidly locked (rather than spring-loaded) doors. Install a flow-limiting controller before the pressure regulator that supplies the inlet. This will limit hydrogen discharge, even if the column is broken or disconnected.

Figure H. Linear Velocity Changes for Hydrogen Have Least Effect on Column Efficiency



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Selecting an Injection Method

Several injection methods can be used to transfer a sample to a capillary column. Some methods are better suited than others to the sample being analyzed (Table 1). The injection methods are:

- *Split Injection* – A splitter divides the injected, vaporized sample into two parts. One part is directed onto the column and the other (usually the larger) is vented. This method allows you to use convenient sample volumes without overloading the column. Split injections are used with most capillary columns (0.1mm-0.75mm ID). This is the most commonly used injection method.

  One drawback to the split injection method, however, is that the splitter can discriminate among sample components if the components have widely different boiling points. This causes inaccurate quantitation. To promote uniform vaporization of all sample components, you can use a liner with a baffle or a cup design. For best performance, the column inlet end should usually be centrally oriented 1-2cm in from the inlet end of the liner. Check your instrument manual for the proper distance.

Table 1. Matching Injection Method to Sample Characteristics

	Sample Characteristics			
Injection Method	Approx. Range	Conc. (ng)	Thermal Stability	Activity
Split	C1-C19	10+	good	low
Splitless	C1-C40	<20	good	low
Direct	C1-C100	<20	good	high
On-column	C1-C40	<20	poor	high
Programmed Temp. Vaporization	C1-C140	<20	poor	low

- *Splitless Injection* – The sample is injected into the hot inlet with the splitter vent off. After some time (determined by experimentation), the vent is turned on. Sample components will be concentrated on the column, while a major portion of the solvent will have been vented. The solvent used and the initial oven temperature are critical. The technique is especially valuable for concentrating very low level compounds on the column, to obtain more symmetric peaks. The method requires slow injection into a special straight bore injector liner. For best results, the inlet end of the column should be inserted into the liner to 1-2cm below the maximum insertion distance of the syringe needle.

- *Direct Injection* – The sample is injected into the liner and is, in turn, transferred onto the column. Because there is no splitter, discrimination is avoided. The liner is a straight tube, or a tube with restrictions on both ends, to keep the sample from backing up into the injection port. Insert the column into the liner according to the instrument manufacturer's recommendation (usually 1-2cm below the maximum insertion distance of the needle). The method is best used with 0.53mm and 0.75mm ID columns.

- *On-Column Injection* – The sample is injected directly into the inlet end of the column. Standard needles can be used with 0.53mm ID fused silica columns and 0.75mm ID glass columns that are *not* connected to fused silica guard columns. Special inlets and fused silica needles must be used with 0.25mm and 0.32mm ID fused silica columns, and 0.75mm ID glass columns connected to fused silica guard columns. On-column injection is used with very active, thermally liable, or high boiling compounds to minimize sample contact with possibly adsorptive surfaces. A slow injection rate is required.

- *Cold Trapping* – Allows large solvent peaks to elute well before the first peak of interest. The column temperature is set 10°C higher than the solvent's boiling temperature and at least 20°C lower than the boiling temperature of the lowest boiling sample component. Consequently, the solvent passes through the column while the compounds to be analyzed are held back. The column is subsequently heated to allow the compounds of interest to elute.

- *Programmed Temperature Vaporization* – The sample is injected onto a liner that is then temperature programmed. Each compound vaporizes at its own boiling point. Low boiling compounds elute at temperatures well below those that could promote decomposition. To ensure the best chromatography, column temperature is very low initially and programmed upward.

Selecting an Injection Technique

There are many different types of syringes and injection techniques available. We recommend several combinations for differing or overlapping purposes (Table 2). The most popular syringes are:

1µL plunger-in-needle syringes – For injecting small samples (0.1-1.0µL). The entire sample resides in the needle and is pushed out of the needle by a plunger. The syringe is filled by capillary action – pull the plunger back and the vacuum draws the sample into the needle. Pumping the plunger will cause the sample to get between the wire and the wall, producing false (larger than measured volume) and very irreproducible sample sizes.

10µL liquid syringes – The most commonly used syringes for gas chromatography. There are several techniques for using them:

- *Filled Needle Technique* – The sample is held in both the needle (1-2µL) and the syringe barrel, and is injected rapidly or slowly, depending on the injection mode. This technique, used with all injection modes and employed by all autosamplers, offers the poorest repeatability of all injection techniques.

- *Cold Needle Technique* – The sample is drawn completely into the syringe barrel and the needle filled with air. Depress the plunger as soon as the needle is fully inserted into the injection port. Complete the injection as rapidly as possible. Like the filled needle technique, this technique is used with all modes of injection.

- *Air Plug Technique* – Similar to the cold needle technique, except that approximately 3µL of air is drawn into the syringe before the sample. Insert the needle into the injection port and allow it to heat for 2-4 seconds, then depress the plunger as rapidly as possible. This technique can be used with all modes of injection and offers good reproducibility.

Table 2. Sample Parameters for Different Injection Techniques

Injection Method	Injection Time (sec.)	Sample Size (µL)	Split Ratio	Splitter	Inlet Temp. (°C)	Initial Oven Temp.
Split	<1	0.1-2	1:30-1:500	on	100-350	NR
Splitless	<20	1-3	1:30-1:500	off 20-60 sec.	100-350	10° < solvent BP
Direct Injection	<3	0.1-2	splitter off	off	100-350	NR
On Column (hot) (cold)	<1	0.1-2	splitter off splitter off	off off	= solvent BP	= solvent BP
Cold Trapping	<1	1-3	1:30-1:500	on	100-350	20° > solvent BP
Programmed Temp. Vaporization	<1	0.1-2	1:30-1:500	on	20° < solvent BP	20° < solvent BP

NR = No Recommendation

- *Solvent Flush Technique* – The needle is first filled with pure solvent, then about 1µL of air, the sample, and another 1µL of air are drawn in succession. Depress the plunger as soon as the needle is in the injection port. The method offers good reproducibility, but the solvent must be pure, and the extra solvent volume must be considered in the analysis. Keep solvent and sample volumes small, to prevent overloading the injection port or column.
- *Sandwich Technique* – Takes the solvent flush technique one step further. Solvent, air, sample, air, and solvent are drawn into the syringe in succession. Injection is made as soon as the empty needle is in the injection port. To prevent overloading the injection port and column, total liquid volume must not exceed 3µL. This technique provides excellent repeatability, but the large volumes involved preclude using it with on-column or direct injection.

Syringe Cleaning Tips

Poorly maintained syringes lead to ghost peaks, poor sample quantification, short septum life, and septum fragments in the liner. For repeatable sample quantification and long syringe life, clean the syringe after every injection and *visually examine* (no fingers) the point for burrs. Remove all sample residue from both barrel and needle. We recommend the following procedure:

Draw pure, particle-free solvent into the full volume of the syringe. Wait a few minutes for the solvent to dissolve soluble materials. Discard this material, preferably by inserting the needle into a heat/vacuum syringe-cleaning device. Repeat the process. It is often helpful to switch solvent types, using first a nonpolar (e.g., hexane), then a highly polar (e.g., acetone) solvent.

Remove the plunger and gently wipe it with a clean tissue. (Do not remove the plunger from wire-in-the-needle syringes.) Before replacing the plunger, insert the needle into a hot injection port, allowing hot carrier gas to dry the needle and barrel (avoid drawing air into the system). Do not dry the needle and barrel with room air. Dust particles in the air can accumulate in the barrel and block it.

Using Test Chromatograms to Evaluate A New Column or Routinely Monitor Column Performance

When you receive a new column, evaluate its performance in your instrument. Use the test mixture and conditions described on the data sheet. The test results, in combination with information from methane injections, will be valuable for evaluating the overall quality of the installation, and the column's efficiency, percent coating efficiency, capacity factor, and inertness.

Compare your results to the test chromatogram and data sheet provided with your column. If you cannot duplicate the results onthe data sheet, a problem exists. Interpretation of the test mix results are covered on the test mix instruction sheet. See the Appendix of this guide for details on calculating performance values from the test data.

Dead volumes will be uncovered by the methane test, as described in a previous section.

Depending on use, capillary columns can, in time, show tailing, broadening peaks, or retention changes. To ensure that your chromatographic system is performing at optimum, make a weekly injection of the test mix or other appropriate isothermal mix. This information will be extremely useful for troubleshooting the system. Expect to duplicate the test chromatogram from week to week. If you cannot, a problem exists.

If a nonbonded column gradually loses efficiency or retention times change, there are two possible causes: phase washing off the inlet end or gradual bleeding from the column, causing a film thickness gradient along the column. Both conditions are gradual processes that can arise from a continual one-directional flow through the column. These problems can be avoided by periodically (about every two weeks) reversing the inlet and detector ends of the column, and thus the direction of flow. This procedure should be used as a preventive measure, not a cure.

Rinsing a Bonded Phase/Removing Contaminated Column Ends

If your column produces tailing peaks (and you know the injector liner is clean and properly deactivated), the problem could be adsorptive sample residue or septum fragments contaminating the inlet end of the column. You can cure this problem by cutting two loops from the column inlet.

Alternatively, you can rinse Supelco bonded phase capillary columns (except those with phase films of 1.5µm or more) with pentane, methylene chloride, or acetone to remove soluble contaminants and septum fragments. Rinsing also removes polymer fragments formed by thermal degradation of the phase while the column is in use. Therefore, each time the column is rinsed and subsequently heated, a small loss in performance (decrease in k') results.

Columns can be rinsed in a variety of ways, but solvent should *always* flow from the column outlet to the inlet, so septum particles and other contaminants are flushed from the inlet, not further into the column. Usually, the easiest way to rinse a column is to attach the inlet end to a vacuum source and pull the solvent through the column. Alternatively, solvent can be pushed through the column, from a pressurized reservoir, at approximately 30psig. To effectively rinse a column, use 5-10mL of solvent for a 0.25mm or 0.32mm ID column, or 20-25mL of solvent for a 0.53mm or 0.75mm ID column.

Under some conditions, contaminants polymerize in the column inlet. Rinsing with solvents will not restore column performance, and it will be necessary to break off approximately 1/2 meter from the column inlet. If your samples are excessively dirty, you can prevent column contamination by attaching a 1/2 meter guard column of fused silica tubing to the column inlet, using a capillary butt connector. Contaminants will be trapped in the guard column. For more information about guard columns, refer to the Supelco catalog.

Troubleshooting Table

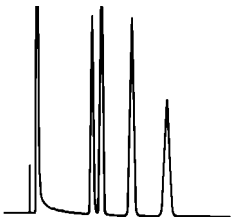
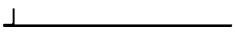
To use this table, locate the symptom your chromatograms are showing, then read the entire list of possible causes. Because a specific remedy can be performed easily on one instrument and with difficulty on another, the causes are not listed in any particular order. Eliminate them one by one, beginning with the easiest and working toward the most difficult.

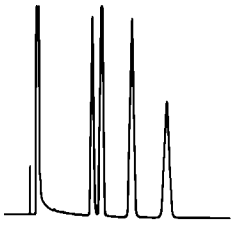
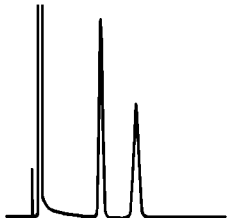
Symptoms Index

Abbreviations

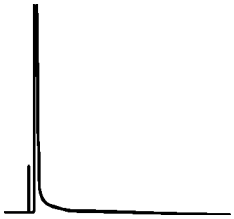
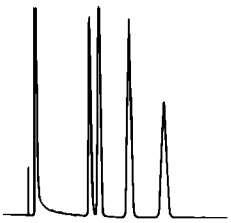
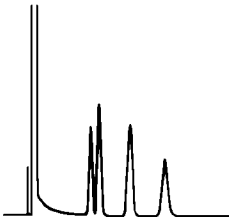
ECD — electron capture detector **FTD** — flame photometric detector **TCD** — thermal conductivity detector **FID** — flame ionization detector

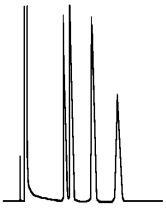
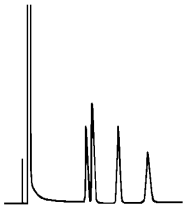
Troubleshooting Table

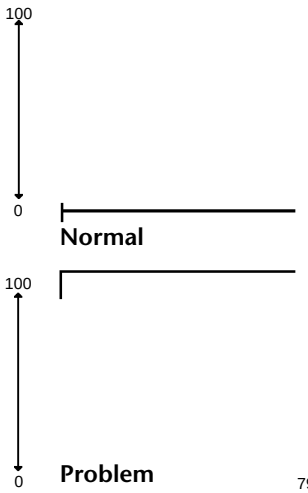
Symptom	Possible Cause	Remedy
Symptom No. 1 — No Peaks		
 **Normal**  **Problem** 795-0314	1. Detector or electrometer power off, or fuse blown.	1. Check detector and electrometer settings. Check fuses.
	2. FID: flame not lit.	2. Hold mirror over FID exhaust. Water condenses on mirror if FID lit. Light flame, if unlit, and adjust hydrogen and air flows.
	3. Sample injected onto wrong column (multiple column chromatograph).	3. Reinject sample onto proper column.
	4. Syringe defective.	4. Inject sample with a new syringe. If problem disappears, discard old syringe.
	5. Injection port temperature too low (sample not vaporized).	5. Verify temperature with an accurate thermometer, adjust if necessary. If instrument setting and thermometer agree, increase temperature (do not exceed stationary phase limit) or inject sample directly onto column.
	6. Oven not heated or column temperature too low (sample condensing in column).	6. Verify temperature with an accurate thermometer, adjust if necessary. If instrument setting and thermometer agree, increase temperature (do not exceed stationary phase limit).

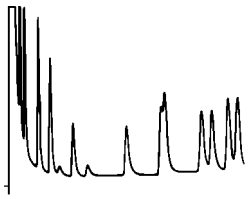
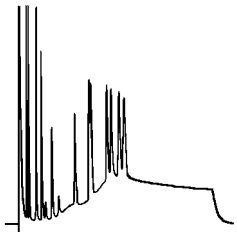
Symptom	Possible Cause	Remedy
Symptom No. 1 — No Peaks *(cont'd.)*		
	7. Recorder malfunctioning.	7. Check recorder connections and adjust if necessary. Set attenuation to infinity. If recorder pen does not go to electrical zero, troubleshoot recorder according to manual.
	8. No carrier gas flow.	8. Check for leaks at column connections and septum (see page 3), replace septum or tighten connections if necessary. Check for obstructions at column inlet (an overtightened septum can obstruct column or end of splitter liner, cutting off gas flow). Be sure pressure at gas source exceeds pressure need at head of column by at least 15psig at maximum temperature. Replace cylinder before pressure falls below 300 psig. Replace regulator or flow controller if defective.
	9. Detector, electrometer, or cables defective.	9. Check collector voltage and connections per instrument manual.
	10. FID: faulty connection between collector and voltage source.	10. Clean contacts as recommended by instrument manufacturer.
	11. Column broken.	11. If broken at inlet or detector end, inspect first few coils for column or septum fragments. If fragments are present, rinse column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). Make clean cut and reinstall column. Repair mid-column break by using a connector. Evaluate column for oxidation or other damage, replace column if necessary.
Symptom No. 2 — Missing peaks/solvent peak only		
 **Normal**	1. Syringe dirty or partially plugged.	1. Inject sample with a new syringe. If problem disappears, thoroughly clean old syringe.
	2. Sample too dilute. Split ratio too high.	2. Inject a standard. If results are satisfactory, increase sensitivity or inject larger or more concentrated sample.
	3. Carrier gas leak at septum or column connection.	3. Check for leaks (see page 3). Replace septum or tighten connections if necessary.
 **Problem**	4. Incorrect injection port or column temperature: • Injection port or column too cold, sample not properly vaporized. • Injection port too hot, thermally labile sample decomposing. • Column too hot, sample components eluting in solvent peak.	4. Verify temperatures with an accurate thermometer, adjust if necessary. If instrument settings and thermometer agree, increase or reduce temperature(s) as necessary (be sure new temperatures are appropriate for column and sample).

795-0315

Symptom	Possible Cause	Remedy
Symptom No. 2 — Missing peaks/solvent peak only *(cont'd.)*		
 **Problem** 795-0317	5. Incorrect linear velocity/column flow rate.	5. Measure column flow and adjust if necessary (see page 4).
	6. Sample components adsorbed by column or inlet liner.	6. Inject standard on column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically. Remove inlet liner and check cleanliness. Replace glass wool and packing or use new, deactivated liner. If sample has never been analyzed and is chemically active, you may need a column with a different (or specialized) stationary phase.
	7. Column cannot separate components from solvent.	7. Change solvent or column.
Symptom No. 3 — Detector response low for all peaks (retention times correct)		
 **Normal**	1. Sample size too small or split ratio too large.	1. Increase sample size or reduce split ratio.
	2. Sensitivity setting wrong.	2. Check sensitivity setting, adjust if necessary. Inject standard for comparison.
	3. Makeup gas flow inadequate.	3. Adjust flow to detector manufacturer's specifications.
	4. Poor injection technique.	4. Use correct syringe size and appropriate injection technique for sample and analysis.
	5. Syringe defective.	5. Inject sample with a new syringe. If problem disappears, discard old syringe.
 **Problem** 795-0316	6. Carrier gas leak at septum or column connection.	6. Check for leaks (see page 3). Replace septum or tighten connections if necessary.
	7. Injection port temperature too low (sample not vaporized).	7. Verify temperature with accurate thermometer, adjust if necessary. If instrument setting and thermometer agree, increase temperature (do not exceed stationary phase limit).
	8. FID: hydrogen or air flow incorrect.	8. Adjust gas flows to detector manufacturer's specifications.
	9. FID: low oxygen level in compressed air.	9. Replace air tank.

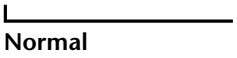
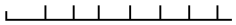
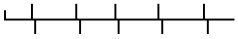
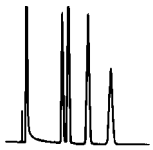
Symptom	Possible Cause	Remedy
Symptom No. 3 — Detector response low for all peaks (retention times correct) *(cont'd.)*		
	10. FID: faulty connection between collector and voltage source.	10. Clean contacts as recommended by instrument manufacturer.
	11. ECD: detector dirty.	11. Clean per instrument manual.
	12. TCD: incorrect carrier gas flow rate.	12. Adjust flow rate to detector manufacturer's specifications.
	13. TCD: incorrect cell voltage.	13. Refer to instrument manual.
	14. Sample components adsorbed by column or inlet liner.	14. Inject standard on column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically. Remove inlet liner and check cleanliness. Use new, deactivated liner or replace glass wool and packing.
	15. FPD: hydrocarbon eluting with sample, diminishing response (quenching effect).	15. Check with hydrocarbon-free standard. If necessary, change to column that will separate hydrocarbons from components of interest.
Symptom No. 4 — Detector response low for all peaks, retention times prolonged		
 **Normal**	1. Linear velocity/flow rate too low.	1. Measure linear velocity and adjust if necessary.
	2. Carrier gas leak at septum or column connection.	2. Check for leaks (see page 3). Replace septum or tighten connections if necessary.
	3. Column temperature too low.	3. Verify temperature with accurate thermometer, adjust if necessary. If instrument setting and thermometer agree, increase temperature (do not exceed stationary phase limit).
 **Problem**	4. Low carrier gas flow/high pressure drop: • overtightened septum • source pressure insufficient (may be most noticeable with temperature programmed analyses) • inlet carrier gas line, or gas purifier plugged	4. Loosen septum. Increase pressure at gauge by 10psig. Flow control must be set at least 15psig above need at maximum temperature of program. Replace liner, tubing, or purifier as necessary.
795-0316		

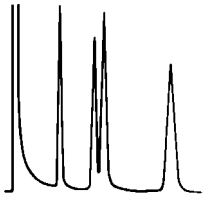
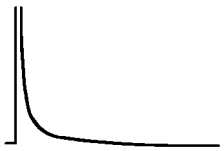
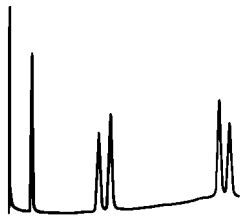
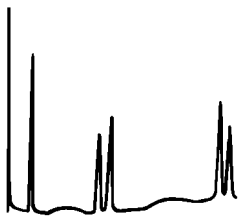
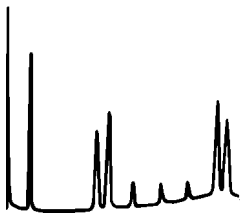
Symptom	Possible Cause	Remedy
Symptom No. 5 — Quantitation not reproducible		
1. Retention times correct, but normalization techniques produce low values for components with longest retention times.	1. Sample parameters incorrect. a. Incomplete sample injection b. Injection port or column temperature too low. c. Incorrect slope sensitivity with electronic integrator. d. Splitter discrimination.	1. Verify parameters, using a standard. a. Use appropriate syringe and injection technique for sample and analysis (see page 6-7). b. Verify temperatures with an accurate thermometer, adjust if necessary. If instrument settings and thermometer agree, increase temperature(s) as necessary (do not exceed stationary phase limit). c. Adjust slope sensitivity. d. Check split rate and adjust if neces sary. Make sure liner is appropriate for injection technique. Make sure injection technique and syringe are correct for sample (see Tables 1 and 2). If necessary, increase injection port temperature.
2. Retention times correct, but equal amounts of different components on-column result in unequal peak areas.	2. a. Detector response differs for different components. b. Components adsorbed by inlet liner (i.e., by glass wool or liner packing) or transfer lines. c. Components adsorbed by polyimide on poorly cut fused silica tubing end or by ferrule fragments in column.	2. a. Determine correction factors and/or use internal standards technique. b. Change liner (use new, deactivated liner). Delete glass wool or packing, if necessary. Clean or replace transfer lines. c. Make clean, square cuts at column ends. Remove ferrule fragments by rinsing column (bonded phase only) or removing 1-2 coils at contaminated end.
3. Quantitation for one component varies among several injections, even when using internal standard technique.	3. a. Internal standard not compensating for all components in sample. b. Slope sensitivity of integration not high enough for late eluting sample components.	3. Use multiple internal standards.
4. Quantitation inconsistent for same sample on successive analyses.	4. Peaks insufficiently resolved, peak tailing.	4. Modify operating parameters or use a column with a different stationary phase.
5. Low values for minor compounds.	5. Sample too small for accurate counting by integrator.	5. Increase sample size or electrometer range setting.
6. Peak response increases with successive injections.	6. Active sites on column adsorbing sample components until saturated (priming the column).	6. Use a deactivated column.
Symptom No. 6 — Baseline off scale, cannot zero		
 795-0318	1. Column temperature too high, column contaminated or improperly conditioned.	1. Verify column temperature with an accurate thermometer. Confirm that it is within limits for stationary phase and analysis. Reduce column temperature to ambient. If problem is the column, baseline will return to normal. Recondition column. If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically.
	2. Recorder malfunctioning.	2. Set attenuation to infinity. If recorder pen does not go to electrical zero, trouble-shoot recorder according to manual.

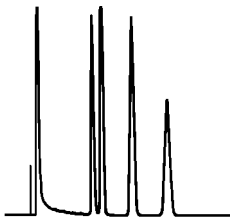
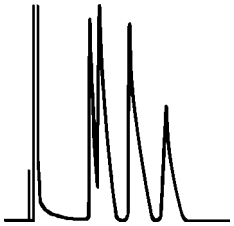
Symptom	Possible Cause	Remedy
Symptom No. 6 — Baseline off scale, cannot zero *(cont'd.)*		
	3. Carrier gas leak at septum.	3. Check for leaks (see page 3). Replace septum or tighten connections if necessary.
	4. Wrong gases used with instrument and detector (e.g., argon/methane with FID).	4. Verify gases are suitable for instrument and detector (see instrument manual).
	5. FID: column outlet end extending through flame jet into flame.	5. Look for column end in detector, flame discoloration. Remove column, cut off burnt end, clean or replace jet. Reinstall column with end below top of jet.
	6. Injection port contaminated.	6. Turn off injection port heat. If zeroing capability returns, replace injection port liner.
	7. Carrier gas flow too high or too low.	7. Measure flow and adjust to within instrument specifications.
	8. TCD: imbalance in column reference and sample flows.	8. Measure flows and adjust until equal.
	9. Detector contaminated (e.g., NPD with Snoop®, ECD with chlorinated solvent).	9. Disconnect column from detector and seal detector inlet. If problem is not eliminated, refer to detector manual for cleaning instructions. Avoid sources of contamination.
	10. Electrometer or detector malfunctioning.	10. Troubleshoot per instrument manuals.
Symptom No. 7 — Baseline drift		
 **Normal**	1. Carrier gas flow changing with temperature during temperature programming.	1. Be sure pressure at gas source exceeds pressure need at head of column by at least 15psig at maximum temperature. Replace cylinder before pressure falls below 300psig. Replace regulator or flow controller if defective.
	2. Carrier gas leak at septum or column connection.	2 Check for leaks (see page 3). Replace septum or tighten connections if necessary.
	3. Septum bleed.	3. Turn off injector heater. If drift disappears, use higher temperature septum or analyze sample at lower injection temperature.
 **Problem**	4. Septum fragments in column.	4. Inspect column ends for septum fragments. If fragments present, rinse column (bonded phase only) or remove 1-2 coils from contaminated end (see page 8). Always use a sharp needle and inject in same spot on septum (use a needle guide).
795-0319		

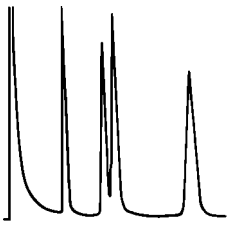
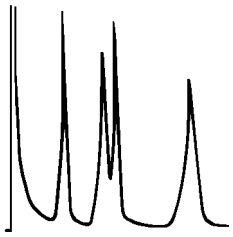
Symptom	Possible Cause	Remedy
Symptom No. 7 — Baseline drift *(cont'd.)*		
	5. Detector contaminated (e.g., NPD with Snoop®, ECD with chlorinated solvent).	5. Disconnect column from detector and seal detector inlet. If problem is not eliminated, refer to detector manual for cleaning instructions. Avoid sources of contamination.
	6. Column bleed or contamination (e.g., residual components from previous injection).	6. Inject standard on column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically.
	7. Some nonbonded phases: rapid temperature changes can cause phase to puddle (form droplets within column), causing high bleed.	7. Replace column. To prevent problem, always heat and cool column gradually to avoid thermal rearrangement of phase. Use bonded phase columns whenever possible.
	8. Gas flows (including hydrogen or air for an FID) not within minimum/maximum limits; flows poorly regulated.	8. Adjust flows to within instrument specifications. Replace regulator or flow controller if defective.
	9. Insufficient instrument and/or column warm-up time or temperature equilibration time.	9. Allow sufficient time for instrument and/or column to equilibrate when changing columns or operating temperatures.
	10. Electrometer or detector defective.	10. Troubleshoot according to steps described on page 2.
	11. Injection port contaminated.	11. Turn off injection port heat. If drift disappears, replace injection port liner.
	12. Makeup or detector gas contaminated.	12. Use only high quality gases: install high performance gas purifiers.
Symptom No. 8 — Irregular or unstable baseline		
Normal	1. Column bleed or contamination, improperly conditioned column.	1. Inject standard on column known to be performing well. If results are good, recondition or rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically.
 **Problem**	2. Contaminated injector liner.	2. Turn off injection port heat. If baseline stabilizes, replace injection port liner.
795-0323		

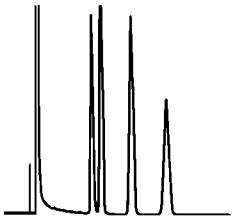
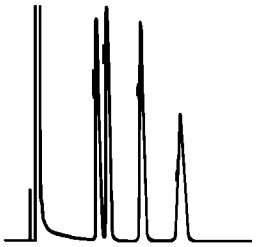
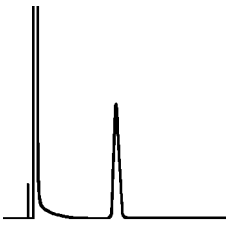
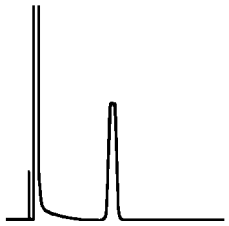
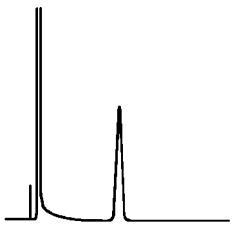
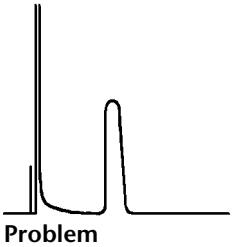
Symptom	Possible Cause	Remedy
Symptom No. 8 — Irregular or unstable baseline *(cont'd.)*		
	3. Detector contaminated (e.g., NPD with Snoop®, ECD with chlorinated solvent).	3. Disconnect column from detector and seal detector inlet. If problem is not eliminated, refer to detector manual for cleaning instructions. Avoid sources of contamination.
	4. Carrier gas leak at septum or column connection.	4. Check for leaks (see page 3). Replace septum or tighten connections if necessary.
	5. Carrier gas flow insufficient or poorly regulated.	5. Be sure pressure at gas source exceeds pressure need at head of column by at least 15psig at maximum temperature. Replace cylinder before pressure falls below 300psig. Replace regulator or flow controller if defective.
	6. Gas or gas line contaminated.	6. Change gas cylinder. Use only high quality gases; install high performance gas purifiers. Replace gas line if necessary.
	7. Gas flows (including hydrogen or air for an FID) not within minimum/ maximum limits; poorly regulated flow.	7. Adjust flows to within instrument specifications. Replace regulator or flow controller if defective.
	8. Defective electrometer, detector, or cable.	8. Troubleshoot according to steps described on page 2.
	9. FID: collector incorrectly aligned.	9. Consult instrument manual, realign collector as required.
	10. ECD: heater wire too close to detector wire (causes AC noise).	10. Consult instrument manual, reposition heater wire.
	11. FID: column outlet end extending through flame jet into flame.	11. Look for column end in detector, flame discoloration. Remove column, cut off burnt end, clean or replace jet. Reinstall column with end below top of jet.
Symptom No. 9 — Cycling baseline drift		
 **Normal**	1. Instrument poorly located (drafts or other changes in ambient temperature, etc.).	1. Relocate instrument, close windows, etc.
	2. Defective oven or detector temperature controller.	2. Monitor temperature with an accurate thermometer. If temperature varies more than specification in instrument manual, replace temperature sensing probe.
 **Problem**	3. Carrier gas flow insufficient or poorly regulated.	3. Be sure pressure at gas source exceeds pressure need at head of column by at least 15psig at maximum temperature. Replace cylinder before pressure falls below 300psig. Replace regulator or flow controller if defective.
795-0324		

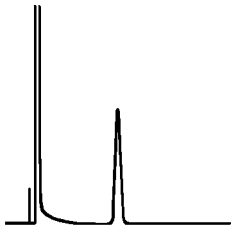
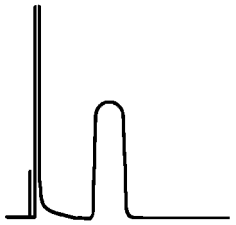
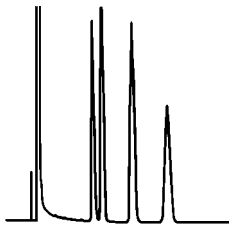
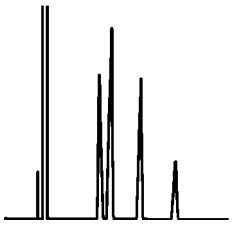
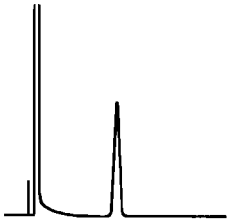
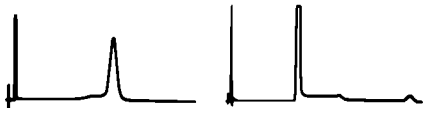
Symptom	Possible Cause	Remedy
Symptom No. 9 — Cycling baseline drift *(cont'd.)*		
	4. FID (when a hydrogen generator is hydrogen source): hydrogen pressure varying, causing sensitivity to vary.	4. Increase output pressure from generator.
	5 Column improperly conditioned or contaminated with very high boiling compound.	5. Inject standard on column known to be performing well. If results are good, recondition original column (rinse bonded phase column, then recondition). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically.
Symptom No. 10 — Spikes (regular)		
 Normal	1. FID: condensate or dust particles in detector.	1. Clean detector and check ends of column for particles. If particles are present, rinse column (bonded phase only) or remove 1-2 coils from contaminated end (see page 8).
 Problem	2. Contaminated carrier, makeup, or other gas.	2. Use only high quality gases; install high performance gas purifiers.
 Problem 795-0320	3. Defective electronics or detector.	3. Troubleshoot electronics and detector according to steps described on page 2.
Symptom No. 11 — Spikes (irregular/erratic)		
Normal	1. Defective detector cable (intermittent short-circuiting).	1. Troubleshoot according to steps described on page 2. Replace cable if necessary.
	2. ECD: heater wires and detector wires loose or too close together.	2. Check wire connections and positions, relocate if necessary.
	3. FID: insufficient hydrogen flow.	3. Monitor hydrogen flow, increase if necessary.
 Problem 795-0321	4. Electronic interference from external source.	4. Relocate instrument, identify interference source (e.g., nearby transmitter site, etc.)
Symptom No. 12 — Negative peaks		
 Normal	1. Recorder improperly connected or polarity reversed.	1. Reverse recorder connections or polarity switch.
	2. Sample injected onto wrong column (multiple column chromatograph).	2. Reinject sample onto correct column.
 Problem 795-0322	3. TCD: impurities in carrier gas.	3. Use only high quality gases; install high performance gas purifiers.

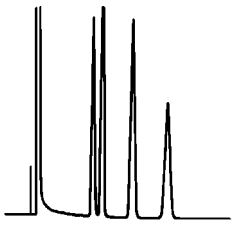
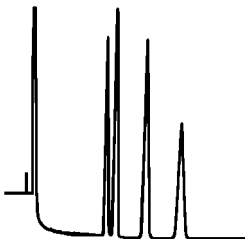
Symptom	Possible Cause	Remedy
Symptom No. 13 — Ghosts peaks (peaks corresponding to previous sample appear when solvent alone is injected)		
 **Previous Sample**  **Normal (solvent injected after sample)**  **Problem (solvent injected after sample)** 795-0336	1. Dirty syringe. 2. Sample adsorbed, then desorbed, by column (particularly in temperature program) or inlet liner. 3. Sample too large (backflashes into areas of injector where it can condense).	1. Inject pure solvent, using a new syringe. If peaks are absent, clean syringes more thoroughly. 2. Inject sample on an identical column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically. Remove inlet liner and check cleanliness. Replace glass wool and packing or use new, deactivated liner. 3. Reduce sample size. Flame heat or solvent rinse transfer lines to remove contaminants.
Symptom No. 14 — Extra peaks		
 **Normal**  **Problem**  **Problem** 795-0337	1. Septum bleed (particularly in temperature program). 2. Peaks from previous runs carried over (commonly appear as very broad peaks with short retention times). 3. Impurities from sample, solvent, sample container or other labware, reagents used in sample preparation. Contaminants in excess reagent can be concentrated during sample preparation. 4. Condensed carrier gas impurities eluting during temperature programming. 5. Trace impurities in lab atmosphere (e.g., cigarette smoke). 6. TCD: air or water peak. 7. Multiple or incomplete derivatives formed during sample workup.	1. Turn off injector heater. If extra peaks disappear, use higher temperature septum or analyze sample at lower injection temperature. 2. Let analysis run longer before repeating. Change temperature program, if possible. 3. Inject solvent alone, using a clean syringe. If extra peaks appear, use higher quality solvent. If only solvent appears, run solvent blank through entire sample preparation process. If extra peaks appear, analyze solvent blank at each step of workup to isolate source. If only solvent peak appears, extra peaks are from sample. 4. Use only high quality gases; install high performance gas purifiers. 5. Analyze lab environment, take corrective action if necessary. 6. Normal condition when using syringe injection or an aqueous sample. 7. Reevaluate derivatization procedure.

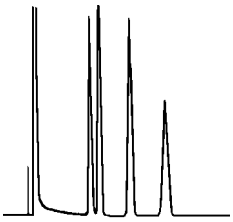
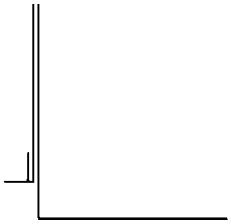
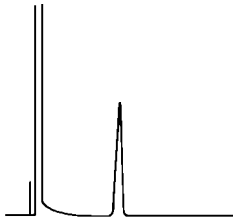
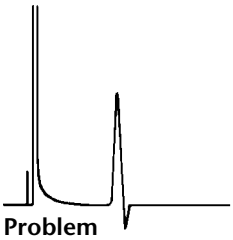
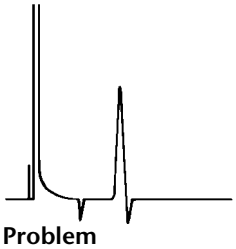
Symptom	Possible Cause	Remedy
Symptom No. 14 — Extra peaks *(cont'd.)*		
	8. Sample decomposing.	8. Verify injector, detector, and oven temperatures with an accurate thermometer, adjust if necessary. If instrument settings and thermometer agree, reduce temperature(s). Inject sample on an identical column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically. If necessary, use a different type of column. Remove inlet liner and check cleanliness. Use new, deactivated liner or replace glass wool and packing. Make sure makeup gas flow is adequate.
Symptom No. 15 — Tailing peaks		
 **Normal**  **Problem** 795-0339	1. Column or injection port temperature too low (hydrocarbons tail). 2. Sample components adsorbed by column or inlet liner (active components in Grob mix tail). 3. Two compounds eluting simultaneously.	1. Verify temperatures with an accurate thermometer, adjust if necessary. If instrument settings and thermometer agree, increase temperature(s) as necessary (do not exceed stationary phase limit). 2. Inject standard on column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically. Remove inlet liner and check cleanliness. Use new, deactivated liner or replace glass wool and packing. If sample has never been analyzed and is chemically active, you may need a column with a different (or specialized) stationary phase. 3. Increase sensitivity and reduce sample size. Reduce column temperature approximately 20°C and look for partial separation. Change temperature program (or consider using one if analysis currently is isothermal). If sample has never been analyzed and is chemically active, you may need a column with a different (or specialized) stationary phase.

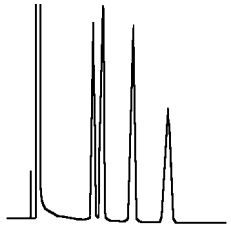
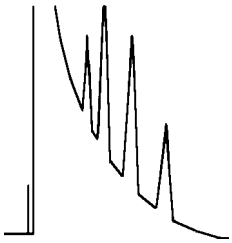
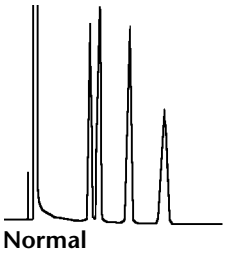
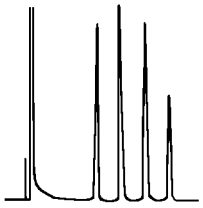
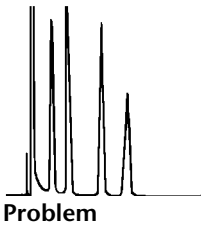
Symptom	Possible Cause	Remedy
Symptom No. 15 — Tailing peaks *(cont'd.)*		
	4. Needle hitting and breaking packing in inlet liner.	4. Partially remove packing from liner.
	5 Column end poorly cut (polyimide adsorbing sample).	5. Remove column, make clean, square cut, reinstall.
	6. Some nonbonded phases: rapid temperature changes cause phase to puddle (form droplets within column), causing drastic loss of efficiency.	6. Replace column. To prevent problem, always heat and cool column gradually to avoid thermal rearrangement of phase. Use bonded phase columns whenever possible.
Symptom No. 16 — Leading peaks		
 **Normal**	1. Column overloaded.	1. Decrease sample size.
	2. Two compounds eluting simultaneously.	2. Increase sensitivity and reduce sample size. Reduce column temperature approximately 20°C and look for partial separation. Change temperature program (or consider using one if analysis currently is isothermal). If sample has never been analyzed and is chemically active, you may need a column with a different (or specialized) stationary phase.
 **Problem** 795-0340	3. Sample condensing in injector or column.	3. Verify injection port and oven temperature with an accurate thermometer, adjust if necessary. If instrument settings and thermometer agree, increase temperature(s) as necessary (do not exceed stationary phase limit).
	4. Sample decomposing.	4. Verify injector, detector, and oven temperatures with an accurate thermometer, adjust if necessary. If instrument settings and thermometer agree, reduce temperature(s). Inject sample on an identical column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically. If necessary, use a different type of column. Remove inlet liner and check cleanliness. Use new, deactivated liner or replace glass wool and packing. Make sure makeup gas flow is adequate.

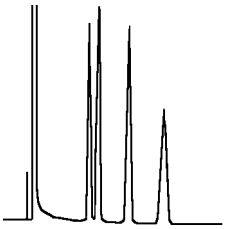
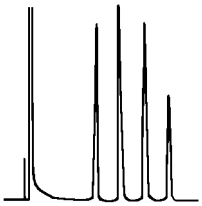
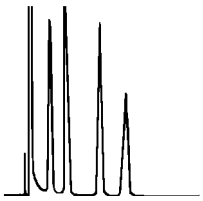
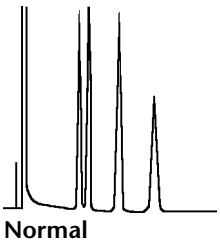
Symptom	Possible Cause	Remedy
Symptom No. 17 — Split peaks		
 Normal  Problem 795-0341	1. Detector grossly overloaded. 2. Sample flashing prior to injection, simulating two injections. 3. Older instruments: column temperature fluctuations ("Christmas tree" effect).	1. Reduce sample size. Make sure makeup gas flow is adequate. 2. Use solvent flush injection technique (see page 8). Use less volatile solvent. 3. Insulate column by wrapping with aluminum foil.
Symptom No. 18 — Square or flat top peaks		
 Normal  Problem 795-0342	1. Electrometer saturated (normal for solvent peak). 2. Recorder defective.	1. Reduce sample size. 2. Troubleshoot recorder per instruction manual.
Symptom No. 19 — Round top peaks		
 Normal  Problem 795-0343	1. FID: detector overloaded. 2. Recorder gain too low.	1. Decrease sample size. 2. Adjust gain.

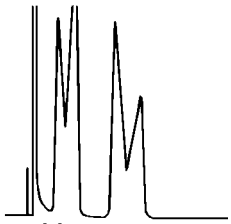
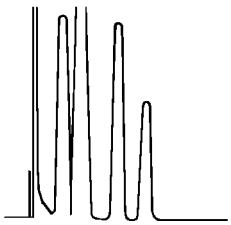
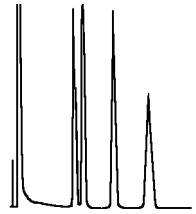
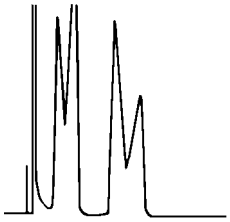
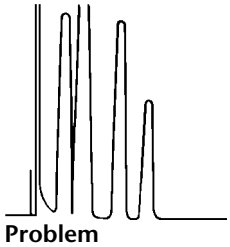
Symptom	Possible Cause	Remedy
Symptom No. 20 — Cigar top peaks		
 Normal  Problem 795-0344	1. ECD: detector overloaded.	1. Reduce sample size.
Symptom No. 21 — Clipped peaks (column efficiency abnormally high)		
 Normal  Problem 795-0345	1. Recorder or instrument zero below movement range of recorder pen.	1. Disconnect leads from recorder and set zero of recorder baseline adjustment to approximately 5% of full scale. Check threshold limit on integrator and adjust accordingly.
Symptom No. 22 — Baseline rise before or after peak		
 Normal  Problem Problem 795-0346	1. Sample decomposing.	1. Verify injector, detector, and oven temperatures with an accurate thermometer, adjust if necessary. If instrument settings and thermometer agree, reduce temperature(s). Inject sample on an identical column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically. If necessary, use a different type of column. Remove inlet liner and check cleanliness. Use new, deactivated liner or replace glass wool and packing. Make sure makeup gas flow is adequate.

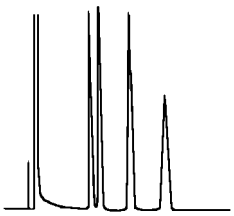
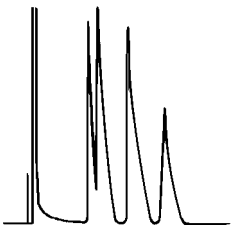
Symptom	Possible Cause	Remedy
Symptom No. 23 — Baseline change after large peak		
 **Normal**	1. Water or large component stripping contaminant from column.	1. Inject sample on an identical column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically.
	2. Column improperly conditioned, stationary phase being stripped (nonbonded phase columns).	2. Inject standard on column known to be performing well. If results are good, recondition original column. If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically.
 **Problem (flow increase)**	3. Pressure imbalance when gas sampling valve activated: • flow controller malfunctioning • sample loop partially plugged	3. Replace flow controller (which adjusts slowly to pressure surges) with a pressure regulator. Clean or replace sample loop. Clean connection ports in valve.
	4. Column and inlet liner misaligned.	4. Check installation of column end and inlet liner, adjust if necessary.
	5. Incorrect zero adjustment on detector amplifier.	5. Balance amplifier according to instrument manual.
	6. Flow controller defective or operating below designed flow range.	6. Replace flow controller if defective. Increase flow or replace controller with one that functions at the desired flow rate. Be sure pressure at gas source exceeds pressure need at head of column by at least 15psig at maximum temperature. Replace cylinder before pressure falls below 300psig.
 **Problem (flow decrease)**	7. TCD: reference and sample gas flows not correctly adjusted.	7. Adjust flows until equal, according to instrument manual.
	8. Injection port temperature too low (sample not vaporized properly).	8. Verify temperature with an accurate thermometer, adjust if necessary. If instrument setting and thermometer agree, increase temperature (do not exceed stationary phase limit) or inject sample directly onto column.
795-0347	9. Large leak at septum during injection and for a short time thereafter (common with large diameter needles).	9. Replace septum and use smaller diameter needles.

Symptom	Possible Cause	Remedy
Symptom No. 24 — Baseline drop after peak: FID flame extinguished		
 Normal  Problem 795-0348	1. Sample too large. 2. Carrier gas, hydrogen, or air flow incorrect. 3. Flame tip plugged. 4. Collector and tip not located properly (whistling or humming sound often heard). 5. Column broken.	1. Decrease sample size. 2. Adjust gas flows to detector manufacturer's specifications. 3. Clean or replace tip. 4. Adjust collector position according to instrument manual. 5. If broken at inlet or detector end, inspect first few coils for column or septum fragments. If fragments are present, rinse column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). Make clean cut and reinstall column. Repair mid-column break by using a connector. Evaluate column for oxidation or other damage, replace column if necessary.
Symptom No. 25 — Negative dip after solvent and/or sample peaks		
 Normal  Problem  Problem 795-0349	1. After large peak only (e.g., solvent): sample too large or component concentration too high for detector. 2. After all peaks: pressure imbalance when gas sampling valve activated: • flow controller malfunctioning • sample loop partially plugged 3. ECD: compounds in carrier gas or sample adding to background current. 4. ECD after all peaks: detector cell dirty. 5. TCD: peak(s) eluting from reference column.	1. Decrease sample size. 2. Replace flow controller (which adjusts slowly to pressure surges) with a pressure regulator. Clean or replace sample loop. Clean connection ports in valve. 3. Use only high quality carrier gases; install high performance gas purifiers. Remove extraneous sample components if possible. 4. Clean detector according to manual. 5. Condition reference column to remove interfering peak(s).

Symptom	Possible Cause	Remedy
Symptom No. 26 — Broad solvent peaks		
 Normal	1. Column installed poorly, producing dead volume in injection port.	1. Check column connections and correct if necessary. Use care when making column connections, especially when changing from one column diameter to another. Use on-column injection.
	2. Normal condition with very dilute sample, as in trace analysis.	2. Ignore.
	3. Poor injection technique.	3. Review injection technique (see pages 6-8).
	4. Injection port temperature too low.	4. Verify temperature with an accurate thermometer, adjust if necessary. If instrument setting and thermometer agree, increase temperature (do not exceed stationary phase limit) or inject sample directly onto column.
 Problem	5. Sample solvent interacting with detector.	5. Change solvent.
	6. Sample solvent retained by column (e.g., methanol by active column).	6. Change solvent.
	7. Septum purge plugged or turned off.	7. Check purge, adjust if necessary.
795-0350	8. Incorrect split flow in split injection mode.	8. Measure split flow, adjust if necessary.
Symptom No. 27 — Retention times prolonged or shortened		
Column previously provided good results	1. Column temperature higher (lower) than previous analyses.	1. Verify temperature with an accurate thermometer, adjust if necessary.
 Normal	2. Carrier gas flow rate lower (higher) than previous analyses.	2. Measure flow rate, adjust if necessary.
	3. Carrier gas leak at septum or column connection.	3. Check for leaks (see page 3). Replace septum or tighten connections if necessary.
	4. Column contaminated or deteriorated.	4. Rinse column (bonded phase only) or remove 1-2 coils at inlet. If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically.
 Problem	5. Some nonbonded phases: rapid temperature changes cause phase to puddle (form droplets within column), causing drastic loss of efficiency.	5. Replace column. To prevent problem, always heat and cool column gradually to avoid thermal rearrangement of phase. Use bonded phase columns whenever possible.
 Problem	6. Recorder chart speed incorrect or recorder defective.	6. Verify chart speed, troubleshoot recorder according to manual.
795-0351		

Symptom	Possible Cause	Remedy
Symptom No. 27 — Retention times prolonged or shortened *(cont'd.)*		
	7. Column overloaded.	7. Reduce sample size.
	8. Column flow varies from run to run.	8. Be sure pressure at gas source exceeds pressure need at head of column by at least 15psig at maximum temperature. Replace cylinder before pressure falls below 300psig. Replace regulator or flow controller if defective.
New column (previous column of same composition provided good results)  **Normal**  **Problem**  **Problem** 795-0352	1. Column of different description ordered incorrectly (e.g., you ordered a 1.0µm phase film when you should have ordered a 0.5µm phase film) or supplied incorrectly by manufacturer (e.g., you received a 45-meter column when you ordered a 60-meter column).	1. Compare column tags or other manufacturer's information. Column lengths, IDs, film thicknesses, etc. must be identical. Contact manufacturer if you find differences.
	2. Column with same description not identical to previous column: • Column longer (shorter) than previous column • Phase film thicker (thinner) than previous column • Stationary phase different, or varies in composition (common problem when using non-GC grade chemicals as phases)	2. Contact manufacturer about column-to-column inconsistency. You may need special purpose or specially tested columns. If phase film thickness is slightly different, you can compensate by adjusting column temperature (higher with thicker film, lower with thinner film). Flow adjustments may also correct problem. NOTE: Most column-to-column differences in length, etc. can be avoided by using standardized columns from reliable manufacturers.
	3. Linear velocity incorrect.	3. Adjust linear velocity.
	4. Carrier gas different from that used with previous column.	4. Change carrier gas.
Symptom No. 28 — Unresolved peaks		
Column previously provided good results  **Normal** 795-0353	1. Column temperature higher (lower) than previous analyses.	1. Verify temperature with an accurate thermometer, adjust if necessary.
	2. Carrier gas flow rate lower (higher) than previous analyses.	2. Measure flow rate, adjust if necessary.
	3. Sample larger or more concentrated than in previous analyses. Minor peak swamped by major peak.	3. Reduce sample size or increase split ratio.
	4. Injections slower than in previous analyses.	4. See injection techniques on pages 6-8.

Symptom	Possible Cause	Remedy
Symptom No. 28 — Unresolved peaks *(cont'd.)*		
 **Problem**  **Problem** 795-0354	5. Column or inlet liner contaminated or column deteriorating. 6. Some nonbonded phases: rapid temperature changes cause phase to puddle (form droplets within column), causing drastic loss of efficiency.	5. Inject standard on column known to be performing well. If results are good, rinse original column (bonded phase only) or remove 1-2 coils from inlet end (see page 8). If column performance is not restored, replace column. To prolong column life, use guard columns (see page 8) and reverse column periodically. Remove inlet liner and check cleanliness. Use new, deactivated liner or replace glass wool and packing. 6. Replace column. To prevent problem, always heat and cool column gradually to avoid thermal rearrangement of phase. Use bonded phase columns whenever possible.
New column (previous column of same composition provided good results)  **Normal**  **Problem**  **Problem** 795-0355	1. Column of different description ordered incorrectly (e.g., you ordered a 1.0µm phase film when you should have ordered a 0.5µm phase film). or supplied incorrectly by manufacturer (e.g., you received a 45-meter column when you ordered a 60-meter column). 2. Column with same description not identical to previous column: • Column longer (shorter) than previous column • Phase film thicker (thinner) than previous column • Stationary phase different, or varies in composition (common problem when using non-GC grade chemicals as phases) 3. Linear velocity incorrect. 4. Carrier gas different from that used with previous column.	1. Compare column tags or other manufacturer's information. Column lengths, IDs, film thicknesses, etc. must be identical. Contact manufacturer if you find differences. 2. Contact manufacturer about column-to-column inconsistency. You may need special purpose or specially tested columns. If phase film thickness is slightly different, you can compensate by adjusting column temperature (higher with thicker film, lower with thinner film). Flow adjustments may also correct problem. ***NOTE:*** Most column-to-column differences in length, etc. can be avoided by using standardized columns from reliable manufacturers. 3. Adjust linear velocity. 4. Change carrier gas.

Symptom	Possible Cause	Remedy
Symptom No. 29 — Column deteriorates too soon after installation (poor resolution, tailing peaks, etc.)		
 **Normal**	1. Column operated at near or above maximum temperature limit of phase.	1. Use higher temperature phase (e.g., bonded phase). • Use shorter column and lower temperature, if possible. • Reduce temperature when column is not in use. • Remove column from oven when another column is used at a higher temperature.
	2. Water or oxygen in carrier gas damaging phase.	2. Use only high quality gases and high-performance purifiers. Replace cylinders before pressure falls below 300psig.
 **Problem**	3. Oxygen from leaks at column connections damaging phase.	3. Check for leaks (see page 3). Tighten connections if necessary. Allow column to cool before removing from GC, to prevent exposing hot column to air.
	4. Water from water-based samples (serum, plasma, other complex samples) stripping phase from column or chemically reacting with phase, nonvolatile components building up in column inlet.	4. Rinsing probably will not cure problem, but cutting 1-2 coils from inlet end may be effective. *Prevent* problem by installing a guard column (see page 8).
	5. Sample too acidic or basic.	5. Adjust sample pH.
	6. Sample contamination at column inlet.	6. Rinse column (bonded phase only) or cut 1-2 coils from inlet end. Install a guard column (see page 8).
	7. Some nonbonded phases: rapid temperature changes cause thermal shock, producing thermal rearrangement of phase with subsequent drastic loss of efficiency. Phase sometimes puddles (forms droplets) within column.	7. Replace column. To prevent problem, always heat and cool column gradually. Use bonded phase columns whenever possible.

795-0356

Appendix

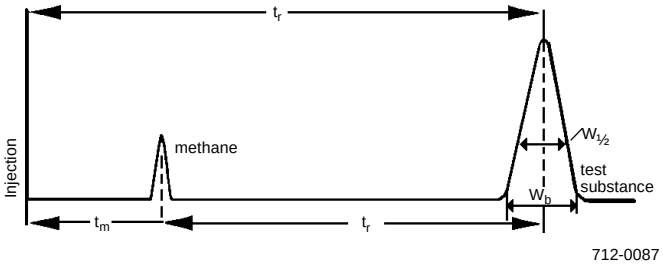
Efficiency

The relationship between the time a solute spends in the column and the width of the peak when the solute elutes. For a given elution time, the narrower the peak, the more efficient the column.

The most common measure of efficiency is the *theoretical plate number*, n. This is defined by Equations 1 and 2, and is measured from a test chromatogram. The t_r and W values can be measured in distance (e.g., mm) or time (e.g., sec.), but both values must be in the same units. $W_{1/2}$ is usually easier to measure than W_b.

$$n = 16\left(\frac{t_r}{W_b}\right)^2 \qquad \textbf{Equation 1}$$

$$n = 5.54\left(\frac{t_r}{W_{1/2}}\right)^2 \qquad \textbf{Equation 2}$$



where t_r = peak retention time (uncorrected)

W_b = peak width at baseline

$W_{1/2}$ = peak width at 1/2 peak height

The gas holdup or dead volume is accounted for by modifying Equations 1 and 2 to include measurements on a compound that is unretained by the phase. Equations 3 and 4 provide the number of *effective plates*, N_{eff}, for the column.

$$N_{eff} = 16\left(\frac{t_r - t_m}{W_b}\right)^2 \qquad \textbf{Equation 3}$$

$$N_{eff} = 5.54\left(\frac{t_r - t_m}{W_{1/2}}\right)^2 \qquad \textbf{Equation 4}$$

where t_m = holdup time for unretained compound.

Column efficiency also can be expressed in terms of the length of column (usually in mm) that is equivalent to one theoretical plate. This value, the *height equivalent to a theoretical plate* (HETP), is determined from Equation 5. HETP values are useful for comparing efficiency among columns of differing length.

$$HETP = \frac{L}{n} \qquad \textbf{Equation 5}$$

where L = column length in mm

The *capacity ratio*, k', for a sample component is the ratio of the time the component spends in the stationary phase to the time it spends in the gas phase. Efficiency is only meaningful if measured on a well-retained peak with a k' of at least 5. This is especially important for comparing different systems. k' is calculated from Equation 6.

$$k' = \frac{t_r - t_m}{t_m} \qquad \textbf{Equation 6}$$

The *Trennzahl or separation number*, TZ, is probably the most realistic way to express the separation efficiency of a column. TZ is the number of completely separated compounds that can be eluted between two sequential peaks of a homologous series, usually n-paraffins differing by one CH_2 unit. The TZ number can be used with temperature programmed analyses, for which n is meaningless. TZ is calculated from Equation 7.

$$TZ = \left(\frac{t_r \text{ peak 2} - t_r \text{ peak 1}}{W_{1/2} \text{ peak 1} + W_{1/2} \text{ peak 2}}\right) - 1 \qquad \textbf{Equation 7}$$

where 1 and 2 are the sequential homologs

Coating Efficiency

The ratio of the theoretical minimum HETP, $HETP_{min}$, to HETP, determined by experiment. Calculate $HETP_{min}$ from Equation 8, then determine coating efficiency (% CE) from Equation 9. ($HETP_{exp}$ is determined from Equation 5)

$$HETP_{min} = r\left(\frac{1 + 6k' + 11k'^2}{3(1 + k')^2}\right) \qquad \textbf{Equation 8}$$

where r = column radius

$$\%\ CE = \left(\frac{HETP_{min}}{HETP_{exp}}\right) \times 100 \qquad \textbf{Equation 9}$$

Column inside diameter and phase film thickness have far more effect on efficiency than does column length. Table 3 shows the effect of column ID on $HETP_{min}$ and the maximum number of effective plates, N_{max}, at k' = 10.

Table 3 -Effect of Column ID on $HETP_{min}$ and N_{max} at k' = 10

	Column ID			
	0.25mm	**0.32mm**	**0.50mm**	**0.75mm**
$HETP_{min}$ (mm)	0.225	0.290	0.450	0.680
N_{max} (plates/m)	4440	3500	2200	1500

Selectivity

The distance between the apexes of two peaks is a measure of the column's selectivity, or ability to separate the two compounds. The *selectivity factor*, α, is determined from Equation 10.

$$\alpha = \frac{t_r \text{ peak 2} - t_m}{t_r \text{ peak 1} - t_m} \qquad \textbf{Equation 10}$$

Resolution

Resolution, R, encompasses both efficiency and selectivity. An R value of 1.5 or greater indicates complete or baseline separation of two peaks. Resolution and the Trennzahl (separation) number are closely related, as shown by the similarity of the equations for determining R (Equation 11) and TZ (Equation 7). A second equation for determining R (Equation 12) also clearly shows the relationship between resolution and selectivity and efficiency. (α is determined from Equation 10 and N_{eff} is determined from Equation 3 or 4)

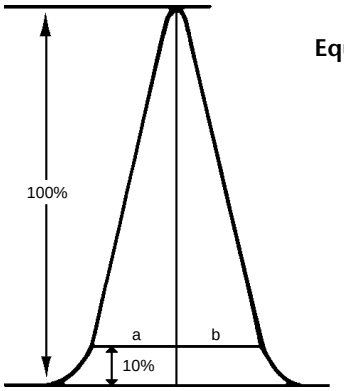
$$R = 2\left(\frac{t_r \text{ peak 2} - t_r \text{ peak 1}}{W_b \text{ peak 1} + W_b \text{ peak 2}}\right) \qquad \textbf{Equation 11}$$

$$R = 1/4\left(\frac{\alpha - 1}{\alpha}\right) N_{eff} \qquad \textbf{Equation 12}$$

Peak Asymmetry

Asymmetric peaks indicate poor column inertness. *Peak asymmetry*, A_s, can be measured in several ways. Because A_s is most severe near the baseline, we determine the value at a point 10% of the peak height above the baseline (Equation 13). A high quality capillary column provides A_s values between 0.9 and 1.1 for appropriate samples.

$$A_s = b/a \qquad \textbf{Equation 13}$$



712-0088

where b = width of right hand side of peak

a = width of left hand side of peak

Helpful Capillary Accessories: Flow Meters

Humonics Veri-Flow 500 Electronic Flowmeter

994-0281

For effortless and continuous readings.

The leaders in GC flow measurement technology have developed an outstanding instrument for analysts who want a simple, continuous-reading flowmeter for general GC applications.

The Veri-Flow 500 is multiple-point calibrated to NIST-certified volumetric standards for nitrogen, helium, hydrogen, air, and 5% argon/methane (certificate supplied), for superior accuracy and to help you comply with ISO 9000, GLP, and other stringent quality control protocols. The display indicates flow in volume (mL/minute) or linear velocity (cm/second), or split ratio. Operation is pulse-free, unaffected by temperature or pressure changes, and fully compatible with electronic pressure control systems. Operates on internal rechargeable batteries. Very low power consumption and automatic shut-off. Features include:

- Calibrated for nitrogen, helium, hydrogen, air, 5% argon/methane
- Range of 1.0-500mL/min; accurate to within ±2% of reading (or 0.05mL)
- Continuous readings in volume, linear velocity, or split ratio
- Highly visible, oversized display
- 9-pin RS 232 communication port for recording data
- AC power adapter jack and recharger
- Only 4 x 5 x 3" (10 x 12.5 x 7.5cm)

Humonics Veri-Flow 500 Electronic Flowmeter **23143**

Humonics Laminar Micro-Flo 20 Flowmeter

994-0280

The perfect tool for flow calibrations in capillary GC.

The accuracy of any mass flow-type device, especially at low flow rates, depends on the instrument's full scale or maximum flow capacity – the larger the capacity, the larger the margin of error. This very affordable unit is the only dry-operation flowmeter specifically designed to provide continuous, accurate linear velocity and volumetric flow readings for capillary GC – flows of 20mL/minute or less.

The Micro-Flo 20 is calibrated to NIST-certified volumetric standards for helium and hydrogen. When the unit is turned on, the display defaults to linear velocity readings – display mL/minute readings simply by pushing a button. The unit is unaffected by temperature or pressure changes. The lifespan for a 9-volt battery is approximately 100 hours. Features include:

- Calibrated for helium and hydrogen
- Range of 0.10-20mL/min (read linear velocity to 999cm/sec)
- Accuracy: ±2% of volumetric reading, averaging ±1cm/sec in linear velocity mode
- Continuous readings in volume or linear velocity
- Highly visible display
- Capillary column adapter and 1/8 inch ID tubing provided

Humonics Laminar Micro-Flo 20 Flowmeter **23144**

A recalibration service is available for these flowmeters. Please inquire.

Helpful Capillary Accessories: Leak Detectors

GOW-MAC® Gas Leak Detectors

Help you find leaks quickly — without risk of contaminating your GC system.

Using liquids to detect gas leaks can be poor economy — especially in a capillary GC system. Even a small amount of liquid leak detector that seeps into a fitting, or through the septum, can damage your column. GOW-MAC gas leak detectors can easily and quickly pinpoint gas leaks too small to detect with soap solution.

GOW-MAC gas leak detectors operate on the same principle as a thermal conductivity detector. The instrument responds to any gas mixture that has a thermal conductivity value different from that of air. With an intrinsically high signal-to-noise ratio, amplification provides maximum usable sensitivity — helium leaks of 1 x 10^{-5} cc/sec and refrigerant leaks of 11 x 10^{-4} cc/sec are easily detected.

Easy to Operate

GOW-MAC gas leak detectors can be operated with little or no training. Turn on, adjust the zero and probe for leaks. The probe is designed to reach difficult and confined locations. A HIGH/LOW switch permits you to control sensitivity for very small leaks.

Model 21-250 Gas Leak Detector

912-0347

Specifications

Line Voltage: Internally selectable 115/230V, 50/60 Hz

Battery: Rechargeable lead/acid gel, 8V

Audio Output: Frequency changes with concentration; adjustable threshold and speaker volume

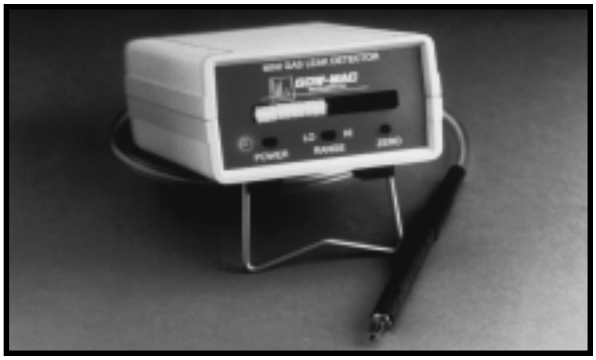
Range: High: x1; Low: x100

Dimensions: 27 x 21 x 9cm (10 3/4 x 8 1/4 x 3 5/8") (excluding handle)

Weight: 4.1kg (9lb) Shipping: 5.4kg (12 lb)

GOW-MAC Gas Leak Detector, 115/230 VAC **22409**

Miniature Gas Leak Detector



994-0274

The GOW-MAC Model 21-050 Mini Gas Leak Detector is a lightweight, hand-held instrument designed to easily and quickly pinpoint gas leaks. Like the full-sized GOW-MAC unit, it eliminates the risk of contaminating your column or system with liquids.

- Simple, one hand operation – turn on, zero, go – in less than 10 seconds
- Fast response (less than 2 seconds)
- Two sensitivity ranges: x1, x100
- Visual LED bar graph
- Rechargeable Ni-Cd battery; charger included

Specifications

Dimensions: 3 1/4 x 1 13/16 x 5 1/4" (8 x 4.5 x 13cm)

Weight: 474g (>1lb)

Mini Gas Leak Detector, 115VAC/60Hz **22807**

Mini Gas Leak Detector, 230VAC/50Hz **22808**

Carrying Case **22809**

Sensitivity of GOW-MAC Leak Detectors

Gas	cc/sec	feet³/year
Argon	1.0 x 10^{-4}	0.110
CO_2	1.1 x 10^{-4}	0.123
Fluorocarbons	1.1 x 10^{-4}	0.123
Helium	1.0 x 10^{-5}	0.012
H_2/He (40:60)	1.0 x 10^{-5}	0.012
Refrigerants	1.0 x 10^{-4}	0.123

NOTE: These GOW-MAC gas leak detectors are not intended for determining leaks of combustible gases. They are intended for nonspecific applications, to determine low level leaks of gases with thermal conductivity different from that of air. We recommend a combustible gas detector for monitoring combustible gases in possibly hazardous situations.

Helpful Capillary Accessories: Septa

Thermogreen™ LB-2 Septa

Inlet Temperature: 100°C to 350°C

- Conditioned, ready to use
- Extremely low bleed over a wide range of inlet temperatures
- Easier needle penetration and high puncture tolerance — ideal for autosamplers
- Silicone rubber formulation exclusive to Supelco

Disc Diameter mm	inch	Qty.	Cat. No.
5	3/16	50	**20638**
6	1/4	50	**20651**
9.5	3/8	50	**20652**
9.5	3/8	250	**20666**
9.5	3/8	1000	**20677**
10	13/32	50	**20653-U**
10	13/32	250	**23156**
10	13/32	1000	**23157**
11	7/16	50	**20654**
11	7/16	250	**23163**
11	7/16	1000	**23164**
11.5	11/24	50	**23154**
12.5	1/2	50	**20660-U**
12.5	1/2	250	**20678**
14	9/16	50	**20662-U**
16	5/8	50	**20663**
17	21/32	50	**23159**
Cylindrical, for Shimadzu® instruments			
Plug Type		10	**20608**
Plug Type		50	**20633**
Drilled, for Solid Phase Microextraction			
9.5	3/8	25	**23161**
9.5	3/8	50	**23162-U**
11	7/16	25	**23167**
11	7/16	50	**23168**

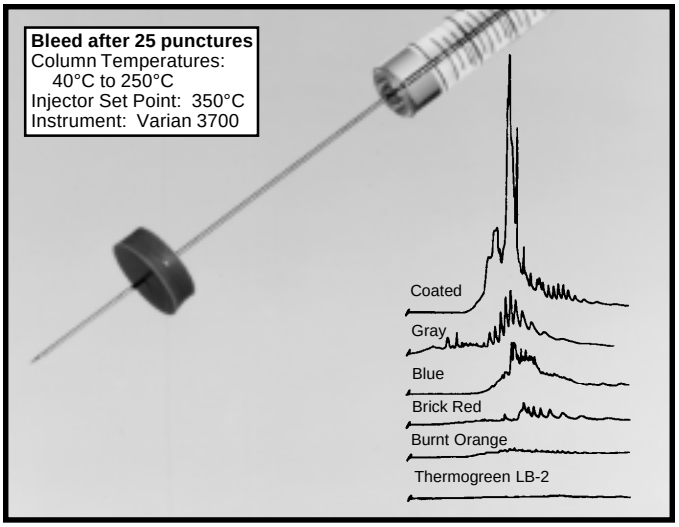
We recommend 9.5mm (3/8") sepa to those who previously used the 9mm size.

Free Mug with Thermogreen LB-2 Septa

P000331

Ask for your free mug when you order any package of Thermogreen LB-2 septa.

Mug **1714-U**

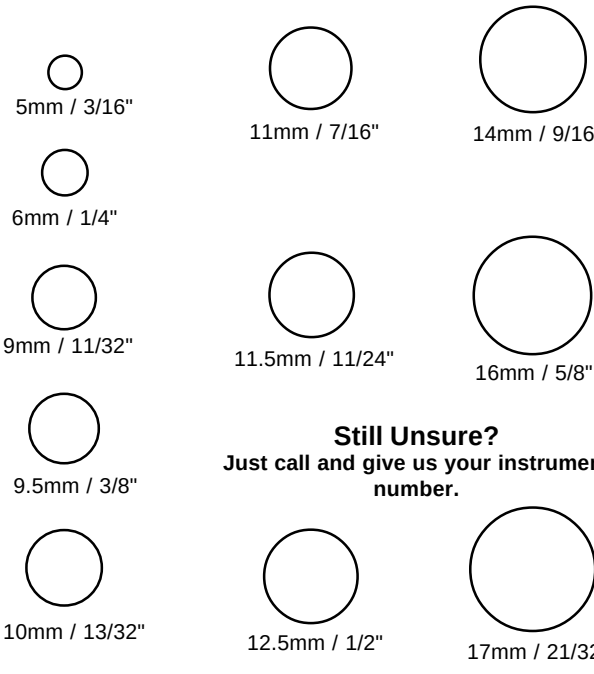


913-0180, 93-0132

Septa Sizes for Various Chromatographs

Manufacturer	GC Model	Size	
GOW-MAC	All models	3/8"	9.5mm
Finnigan	9600	3/8"	9.5mm
Hewlett-Packard	5880A, 5890	7/16"	11mm
	5700 series, 5880	3/8"	9.5mm
	5880/90, OCI ports	3/16"	5mm
Konir	All models	3/8"	9.5mm
Perkin-Elmer	Sigma series, 900 & 990, 8000, Auto Sampler	7/16"	11mm
Tracor	220, 222	1/2"	12.5mm
	550, 560	3/8"	9.5mm
Varian	Packed column injectors	3/8"	9.5mm
	SPI	7/16"	11mm
	3700/Vista, capillary injectors	7/16"/11/24	11/11.5mm
	Saturn GC/MS	11/24"	11.5mm
Fisons/Carlo Erba	8000 series	21/32"	17mm
Shimadzu		plug	

Measure Your Old Septum Here



Still Unsure?

Just call and give us your instrument number.

713-0464

Helpful Capillary Accessories: Fused Silica Tubing and Connectors

We have greatly expanded our lines of untreated tubing and tubing with surface deactivation. These products can be used as transfer lines, guard columns, or retention gaps, or to make your own columns.

Tubing can be coupled through the use of GlasSeal fused silica or glass connectors. If necessary, polyimide glue can be used to provide a permanent seal.

Tubing Treatment	Application
Untreated	General purposes, where high inertness is not necessary
Nonpolar (methyl)	Low polarity solvents (e.g., alkanes, carbon disulfide, ethers)
Intermediate Polarity (phenyl/methyl)	Intermediate polarity solvents (e.g., acetone, methylene chloride, toluene)
Polar (PEG)	Polar solvents (e.g., acetonitrile, methanol, water)

Fused Silica Tubing

ID (mm)	Untreated Cat. No.	Deactivated Tubing — Nonpolar Cat. No.	Deactivated Tubing — Intermediate Polarity Cat. No.	Deactivated Tubing — Polar Cat. No.
3 x 1-meter lengths				
0.10	25700-U	25704	25705	25710
0.20	–	24057	25706	25711
0.25	24024	24025	25707	25712
0.32	25702	24058	25708	25713
0.53	25703	25307	25709	25714
3-meter length				
0.10	25715	25720-U	–	–
0.20	–	25721	25726	25731
0.25	25717	25722	25727	–
0.32	25718	25723	25728	–
0.53	25719	25724	25729	25734
5-meter length				
0.10	25735	25740-U	25745-U	–
0.20	–	25741	25746	–
0.25	25737	25742	25747	–
0.32	25738	25743	25748-U	25752-U
0.53	25739	25744	25339•	25753
15-meter length				
0.20	–	25755	–	25763
0.25	24059	25756	25760-U	–
0.32	24062	25757	25761	25765
0.53	25306	25758	25762	25766
30-meter length				
0.20	25767	25768-U	25772	–
0.25	–	25769-U	–	25777
0.32	24063	25770-U	25774	25778
0.53	25308	25771	25775-U	25779
60-meter length				
0.20	–	–	25786	–
0.25	24061	25783	25787	–
0.32	24064	25784	25788-U	25792
0.53	25781	25785	25789	–

•Deactivated according to USP 467.

GlasSeal™ Column Connectors

GlasSeal connectors immediately connect fused silica tubing of the same or different diameter – no tools, no leaks. Use to connect a guard column or transfer line, repair a broken column, or connect columns having the same or different phases. "Y" connectors split a sample to two columns or a column effluent to two detectors. Silanized for an inert inside surface. Use with our 0.25mm-0.53mm ID tubing.

GlasSeal Capillary Column Connectors

borosilicate glass, pk. of 12	**20479**
fused silica, pk. of 5	**23627**
fused silica, pk. of 25	**23628**

"Y" GlasSeal Capillary Column Connectors

borosilicate glass, each	**20480**
fused silica, each	**23631**
fused silica, pk. of 3	**23632**

Polyimide Sealing Resin

A GlasSeal connector will form a perfect seal between two columns; a small drop of this resin makes the connection extremely durable. Also an excellent high temperature glue. Cure at 200°C; maximum temperature 350°C. Five gram bottle with handy applicator cap.

Polyimide Sealing Resin, 5g **23817**

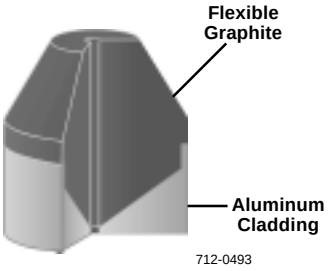
Helpful Capillary Accessories: Ferrules

CapSeal Bullet™ Ferrules

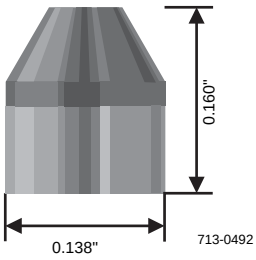
Will not adhere to fittings.

Reusable CapSeal Bullet Ferrules* consist of a graphite material captured in an aluminum base. This unique design keeps the ferrules from adhering to the fitting, making them easy to remove. Eliminate the headache of digging out a stuck ferrule and risking damage to your fittings.

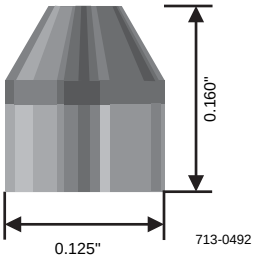
- 450°C temperature limit — temperature programmed or isothermal use
- Special end taper — reduces graphite extrusion into fitting
- Low-torque sealing (1/8 turn past fingertight)



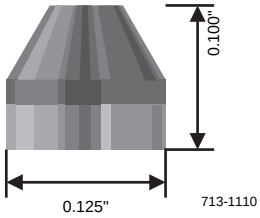
General Purpose



Varian



Hewlett-Packard On-Column Injection



*Patent pending.

Column ID	Ferrule ID	General Purpose (1/16" fitting)	Varian	Qty.	Hewlett-Packard On-Column Injection**	Qty.
0.20-0.25mm fused silica	0.4mm	**23480-U**	**23488**	12	**23864**	10
		23485	**23493**	48	**23867**	50
0.32mm fused silica	0.5mm	**23481**	**23489**	12	**23865**	10
		23486	**23494-U**	48	**23868**	50
0.53mm fused silica	0.8mm	**23482**	**23490**	12	**23866**	10
		23487	**23495**	48	**23869**	50
0.50-0.75mm glass	1.0mm	—	**23491-U**	12	—	—

**For capillary injection ports of HP5890 Series II.

Supeltex™ Capillary Column Ferrules (use with 1/16" fittings)

Ferrule Type and Temp. Limit	Qty.	Column ID: 0.75mm glass Cat. No.	0.50-0.75mm glass Cat. No.	0.53mm silica Cat. No.	0.32mm silica Cat. No.	0.20-0.25mm silica Cat. No
Supeltex M-4	10	**22460**	—	—	**22462**	—
(450°C)	10	—	**22494**	**20628**	—	**22498**
	50	—	—	**22479**	**22412**	**22480-U**
Supeltex M-2A	10	**22459**	—	**22489**	**22461**	**503258**
(400°C)	50	—	—	**22473**	—	**22474**
2 holes	5	—	—	—	**22463**	—
	5	—	—	—	—	**22467**
Indented blank (drill to fit your column)	10	—	—	—	**22488**	—
Supeltex M-1 (250°C)	10	—	—	**22499**	—	—
	Ferrule ID	1.2mm	1.0mm	0.8mm	0.5mm	0.4mm

Supeltex M-2B Ferrules for Varian SPI System (pk. of 10)

0.4mm ID	**22510-U**
0.5mm ID	**22511**
0.8mm ID	**22512**

Helpful Capillary Accessories: Tools

Capillary Starter Kit

913-0191

This kit contains all the tools you'll most likely need to use your capillary column. If you're a new capillary chromatographer or an experienced chromatographer tired of improvising with packed column tools, you can have everything you need right at hand.

Here's what you get:

- Tweezers
- Pin vise drill kit
- Flow calculator
- Pocket mirror
- Dispoz-a-lamp
- Capillary Cleaving Tool
- Chromatogram labels (100)
- Screw-type septum puller
- 1/4" x 5/16" open end wrench
- 6" (15cm) stainless steel ruler
- 6" (15cm) pipe cleaners (6)

Capillary Starter Kit **23639**

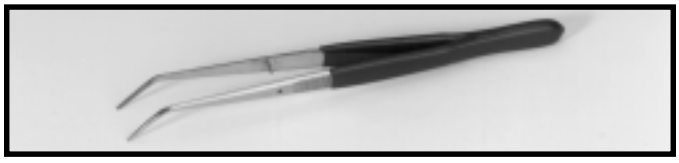
Mirror

913-0283

Ideal for examining inside of injection and detector fittings for ferrule or capillary fragments. Rotating head allows wide range of movement.

Mirror **22434**

Stainless Steel Tweezers

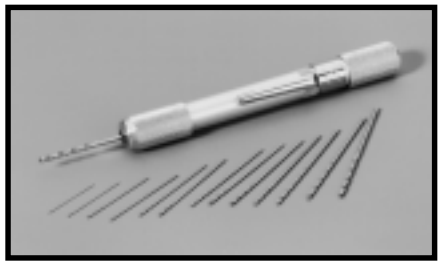


913-0284

Perfect for picking up hot injector liners and detector parts or for preventing finger oil contamination. Also useful for holding ferrules while inserting capillary tubing.

Tweezers **22435-U**

Pin Vise Drill Kit



913-0402

Drill the exact bore you need in hard or soft ferrules. This kit consists of a pin vise and 14 drill bits:

0.33mm/0.0135"	0.77mm/0.031"	1.06mm/0.042"
0.40mm/0.016"	0.83mm/0.033"	1.17mm/0.046"
0.56mm/0.022"	0.91mm/0.036"	1.40mm/0.055"
0.63mm/0.025"	0.97mm/0.038"	1.61mm/0.061"
0.72mm/0.028"	1.02mm/0.040"	

The handle holds all bits to keep them at your fingertips. The improved chuck is handy for gripping fine wire when cleaning FID jets, syringe needles or any other small orifice.

Pin Vise Drill Kit **23820-U**

Drill Bits

Diameter	Cat. No.
0.35mm	**23811-U**
0.40mm	**23810**
0.51mm	**23809**

Capillary Cleaving™ Tool

912-0177

Make scalpel-like cuts in both polyimide and fused silica — leaves no jagged edges to create problems. Industrial sapphire cutting edges remain sharp indefinitely.

The spring-loaded retractable blade version reduces the chance of breakage if the tool is dropped. Replacement blades are available.

Capillary Cleaving Tool with retractable blade **23814**

Replacement Blade for 23814 **23815**

Capillary Cleaving Tool with fixed blade **23740-U**

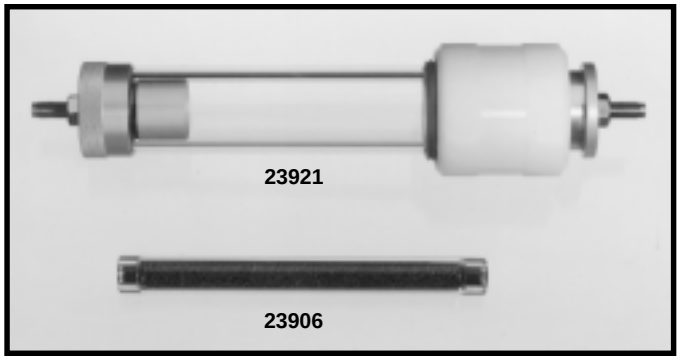
Files

Four-inch slim taper files to enlarge ferrule ID. Also handy for removing ferrules stuck in fittings.

Needle Files, pk. of 2 **23783**

Helpful Capillary Accessories: OMI™ Indicating Purifier

Irreversibly removes contaminants from carrier gas



913-0142

- Effectively purifies helium, hydrogen, nitrogen, argon/methane, and *ammonia*
- Glass body does not diffuse air or off-gas
- Ideal for Hall, ECD and GC/MS detection systems
- Color change indicates purifier exhaustion

Use this purifier for point-of-use gas polishing and final visual quality assurance before gas enters the GC. Simultaneously removes oxygen, water vapor, carbon monoxide, carbon dioxide, most sulfur compounds, most halogen compounds, alcohols, and phenols to less than 10ppb.

Install the OMI tube downstream from your gas purifying device, and tell at a glance whether or not oxygen and moisture are being effectively eliminated from your system. The tube contains Nanochem® resin, developed for the demanding gas purity needs of the semiconductor manufacturing industry. As little as 1ppm of oxygen or moisture will change the indicating resin from black to brown.

We have redesigned this popular purifier to allow easy replacement of spent tubes. Simply unscrew the end assembly from the tube holder and replace your OMI tube. The design prevents air from entering the new tube during installation, preserving the resin.

Our seal replacement kit contains two Teflon seals and a removal tool.

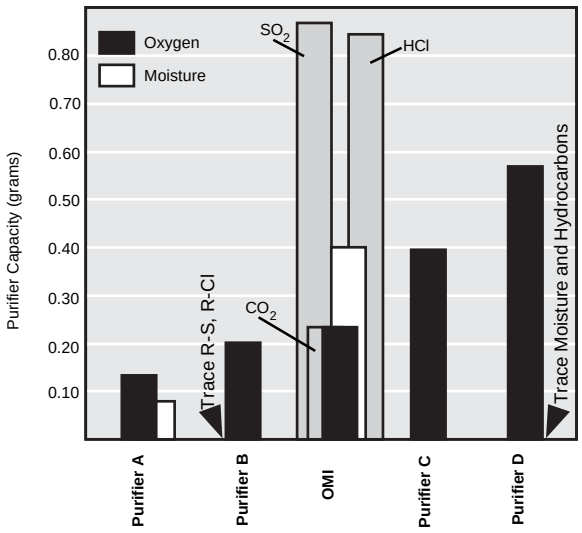
The OMI Purifier Protects Your Column from Many Carrier Gas Contaminants

Contaminant	OMI Purifier	Indicating Devices	Non-Indicating Oxygen Traps
Oxygen	Yes	Yes **	Yes **
Water	Yes	No	No
Carbon monoxide	Yes	No	No
Carbon dioxide	Yes	No	No
Alcohols/Phenols	Yes	No	No
Sulfur-containing compounds (R-S)	Yes	No	No ***
Halogen-containing compounds (R-Cl)	Yes	No	No ***

**If incoming oxygen level does not exceed 10ppm.
*** Corrosive compounds may poison some of these devices.

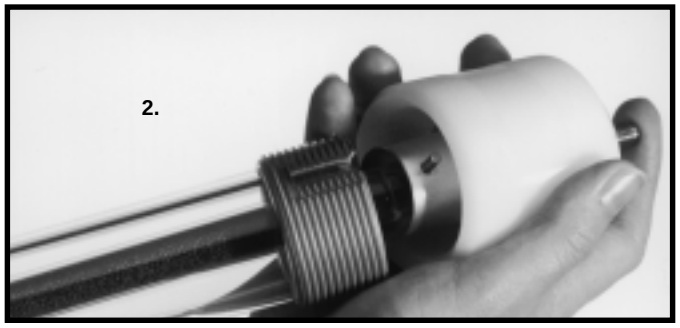
For more information about gas purification, request Bulletin 848. OMI purifiers contain Nanochem resin, licensed to Supelco for use in chromatographic applications. Nanochem is a registered trademark of Matheson Gas Products.

OMI Indicating Purifier Removes as Much Oxygen as Most *Nonindicating* Bulk Purifiers



795-0380

913-0148



913-0147

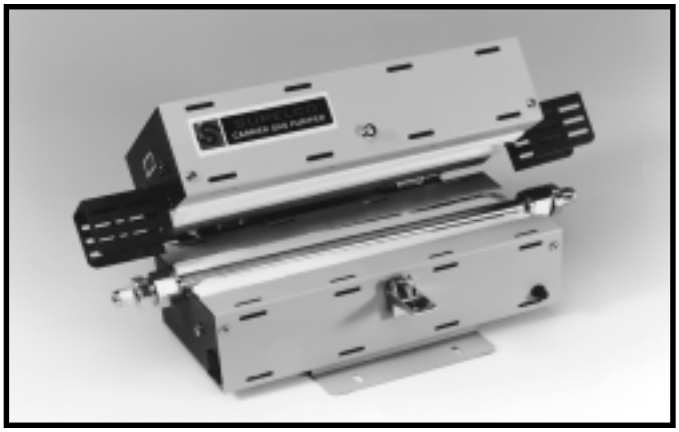
OMI-2 Purifier (tube only)	**23906**
OMI-2 Tube Holder Includes 1/8" fittings, does not include tube	**23921**
Seal Kit for OMI-2 Tube Holder Includes 2 Teflon seals and tool	**23917**
OMI-1 Replacement Tube*	**23900-U**

*Will not fit OMI-2 tube holder. Use with OMI-1 installation kits.

Note: First time users must order both OMI-2 holder and purifier tube.

Helpful Capillary Accessories

No other purifier removes both oxygen and water in such large quantities.



994-0140

UL and CSA listed; CE approved.

To fully protect your GC columns and detectors from oxygen and moisture damage, you should use a gas purifier specifically designed to ensure maximum gas purity. The Supelco High Capacity Gas Purifier tube is heated inside an oven, and oxygen and water react with the gettering material in the tube.* Unlike purifiers which remove contaminants by adsorption, reaction with the gettering material prevents these contaminants from returning to the gas stream — even when the temperature changes or as the material approaches saturation.

A single, replaceable high capacity purifier tube can remove 14L of oxygen or 35L of water vapor (STP). It removes oxygen and water from at least 60 tanks of heavily contaminated gas — gas containing 100ppm of oxygen and/or water. Efficiently removes oxygen and water at gas flow rates up to 1100mL/minute, and you can use it with any carrier gas except hydrogen.

The stainless steel converter tube is 10" x 1/2" OD. The split-sided heater is 10" long. The unit's integral mounting bracket allows you to bolt the unit to a bench top or wall. The 90 watt power consumption makes the unit as economical to operate as a light bulb.

High Capacity Gas Purifier, 115VAC

1/8" fittings	**23800-U**
1/4" fittings	**23802**

High Capacity Gas Purifier, 230VAC

1/8" fittings	**23801**
1/4" fittings	**23803**

Replacement Purifier Tubes

1/8" fittings	**22396**
1/4" fittings	**22398**

*The High Capacity Gas Purifier also removes carbon monoxide and carbon dioxide.

Column Test Mixes

After you install a column in your system, use a test mix to make sure you haven't also installed some surprises (such as ferrule or tubing fragments in the column, or small leaks). Weekly tests thereafter will keep little problems from growing into big problems. Test mixes are an inexpensive aid to obtaining high quality chromatograms.

Acidity Column Test Mix

Even a highly efficient column can adsorb acidic or basic compounds. To determine the acid/base affinity of your column, simply inject this mix and compare peak heights (Grob & Grob, Chromatographia, *4*:421, 1971). Instructions included.

0.05% each component in methylene chloride.

2,6-Dimethylaniline 2,6-Dimethylphenol

2mL **48255-U**

Hydrocarbon Test Mix

This easy-to-use mix is ideal for checking column installation when you use a capillary column in a modified packed column system. You also can use it to determine theoretical plates.

C12-C17 hydrocarbons, 500-2000µg/mL in chloroform.

2mL **48244**

Isothermal Test Mixes

Use to indicate column efficiency, leaks, dead volume, and sample adsorption. Simple, detailed instructions included.

Polar Column Test Mix

For SUPELCOWAX™ 10, SP™-1000, and other polar phases.

500µg/mL each component in methylene chloride.

2-Octanone
Pentadecane (C15)
1-Octanol
Hexadecane (C16)
Heptadecane (C17)
Octadecane (C18)
2,6-Dimethylaniline
2,6-Dimethylphenol
Eicosane (C20)

2mL **47302**

Intermediate Polarity Column Test Mix

For SPB™-20 and other intermediate polarity phases.

500µg/mL each component in methylene chloride.

Decane (C10)
2-Octanone
Undecane (C11)
1-Octanol
Dodecane (C12)
2,6-Dimethylphenol
Tridecane (C13)
2,6-Dimethylaniline
Tetradecane (C14)

2mL **47301**

Nonpolar Column Test Mix

For SPB-1, SPB-5, and other nonpolar phases.

500µg/mL each component in methylene chloride.

2-Octanone
Decane (C10)
1-Octanol
2,6-Dimethylphenol
Undecane (C11)
2,6-Dimethylaniline
Dodecane (C12)
Tridecane (C13)

2mL **47300-U**

Isothermal Test Mix Kit

2mL each of the following test mixes.

Polar Column Test Mix (47302)
Intermediate Polarity Column Test Mix (47301)
Nonpolar Column Test Mix (47300-U)

47303

Column Test Mixes

Methane Standard

Use 40µL injections of this dilute methane standard (100ppm in helium) for more accurate flow measurements than with smaller quantities of more concentrated methane. Use with the methane syringes, syringe adapter, and pressure regulator listed here. Disposable cylinder.

100ppm in helium.

14L **307200**

Accessories

Hamilton® 1725N Syringe	**20705**
Syringe Adapter	**609010**
Pressure Regulator	**513010**

Programmed Test Mix

This mix is for a sensitive, temperature programmed analysis (Grob, et al., *J. Chromatogr. 156*:1, 1978) that tests a column's affinity for many compounds. Prepared at concentrations convenient for setting split ratios and sample sizes. In use, on-column quantities are those recommended by Grob, et al.

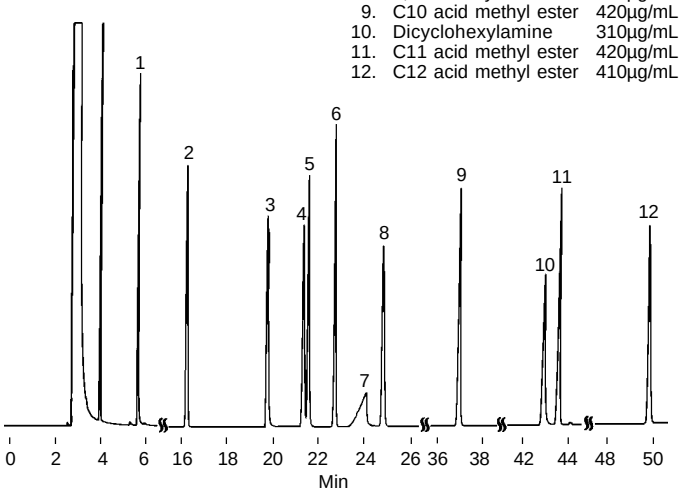
Each component at the quantity indicated in methylene chloride.

2mL **47304**

Programmed Test Mix

Column: **SPB-1, 30m x 0.25mm ID**
Cat. No.: **24029**
Oven: 50°C to 200°C at 2°C/min
Carrier: helium, 20cm/sec
Det.: FID, 250°C
Inj.: 1µL, 250°C, split 100:1

1.	2,3-Butanediol	530µg/mL
2.	Decane	280µg/mL
3.	1-Octanol	360µg/mL
4.	2,6-Dimethylphenol	320µg/mL
5.	Nonanal	400µg/mL
6.	Undecane	290µg/mL
7.	2-Ethylhexanoic acid	380µg/mL
8.	2,6-Dimethylaniline	320µg/mL
9.	C10 acid methyl ester	420µg/mL
10.	Dicyclohexylamine	310µg/mL
11.	C11 acid methyl ester	420µg/mL
12.	C12 acid methyl ester	410µg/mL



713-0256

Test Mixes for Specific Phases

For popular Supelco capillary columns. Active components and inactive hydrocarbons.

Carbowax® Amine Column Test Mix

1mL **48278**

α-DEX™ Column Test Mix

1mL **48013**

β-DEX Column Test Mix

1mL **48028**

Petrocol D2887 Column Test Mix

6 x 1mL **48889**

SPB-1 Thin Film Column Test Mix

For 0.10µm film SPB-1 columns.

1mL **48273**

SPB-1 Thick Film Column Test Mix

For 3µm and 5µm film SPB-1 columns.

1mL **48275-U**

SPB-50 Column Test Mix

1mL **48280-U**

Column Test Mixes

Omegawax™ Test Mixes

Use these mixes to periodically assess the performance of an Omegawax capillary column in analyses of fatty acid methyl esters. The Omegawax Column Test Mix and the Menhaden Oil standard are based on naturally occurring mixtures of fatty acids. Relative peak sizes may vary from lot to lot.

Omegawax Column Test Mix

1mL **48476**

Menhaden Oil

1mL **48473**

Sup-Herb™ Test Mixes

For Sup-Herb columns or herbicide columns of comparable selectivity.

Herbicides Mix 1

Eptam
Sutan
Tillam (Pebulate)
Ordram (Molinate)
Ro-Neet (Cycloate)
Treflan (Trifluralin)
Atrazine
Terbacil
Sencor
Bromacil
Paarlan (Isopropalin)
GOAL (Oxyfluorfen)
Velpar (Hexazinone)

1mL **49136**

Herbicides Mix 2

Vernam
Propachlor
Balan
Simazine
Propazine
Tolban (Profluralin)
Dual
Prowl
Oxadiazon

1mL **49138-U**

Trademarks

CapSeal Bullet, Cleaving, DEX, GlasSeal, Omegawax, OMI, Petrocol, SP, SPB, Supelco, SUPELCOWAX, Supeltex, Sup-Herb, Thermogreen — Sigma-Aldrich Co.
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GOW-MAC — GOW-MAC Instrument Co.
Hamilton — Hamilton Co.
Nanochem — Matheson Gas Products
Pressure-Lok — Precision Sampling Corp.
Shimadzu — Shimadzu Corp.
Snoop — Nupro Co.
Swagelok — Crawford Fitting Co.
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GOAL — Rohm & Haas Co.
Prowl — American Cyanamid Co.
Sencor — Bayer AG

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