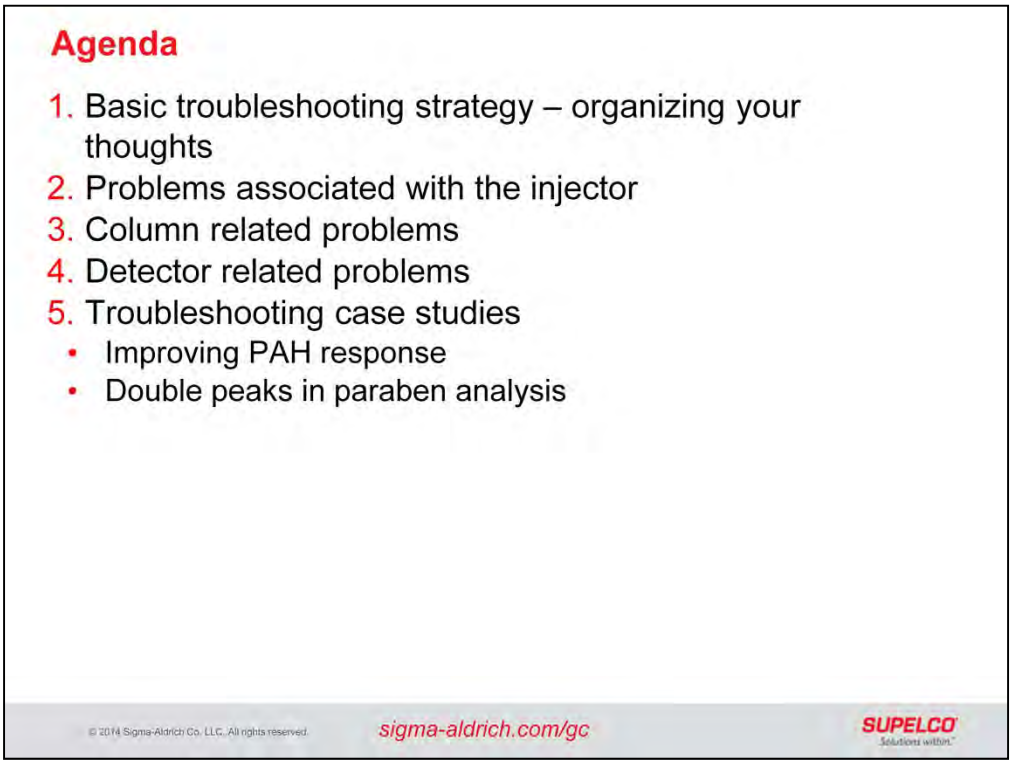
Hello everyone, and thank you for attending this webinar today. GC troubleshooting is a very broad topic, and we could easily devote an entire day to talking about it. In this webinar, I will be focusing on helping you to develop general troubleshooting strategies and give you some things to consider when things do not go as planned in a GC analysis.



- We will start with establishing a logical approach to troubleshooting, how to organize your thoughts, if you will.
- Next, I will focus on some specific problems related to the
    - injector
    - The column and
    - detector portions of the system.
- Finally, we will take a look at a couple of case studies in troubleshooting.

Troubleshooting Strategy

Approaching the problem…

- Stop, take a breather and think!
  - When did the problem start?
  - Has something changed?
- Check first to see if a "fix" for the problem is already known.
  - Talk to others in your lab
  - Check instrument maintenance/service log

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The easiest thing to do when first approaching a problem is to see if there is already a record of it happening in the past. There is no sense in "reinventing the wheel."

Sometimes taking a step back and talking to others, or checking instrument logs, is better to do before you start to take things apart or change them.

First thing, review your method parameters…

- For existing methods
  - Has anything changed?
  - What has occurred since things were working?
  - Does the instrument require maintenance?
- For new methods
  - Injection technique
  - Liner selection; is there something better out there?
  - Injector settings (EPC, temp., split vent time)
  - Column
  - Overall GC conditions (temps, flows)

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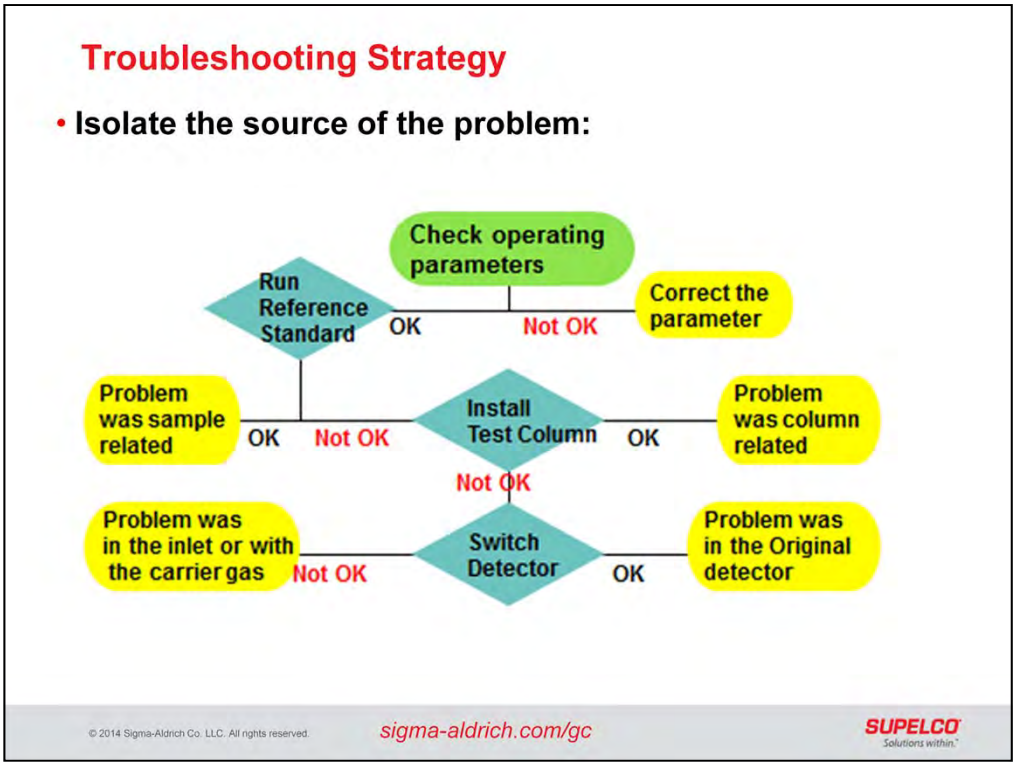
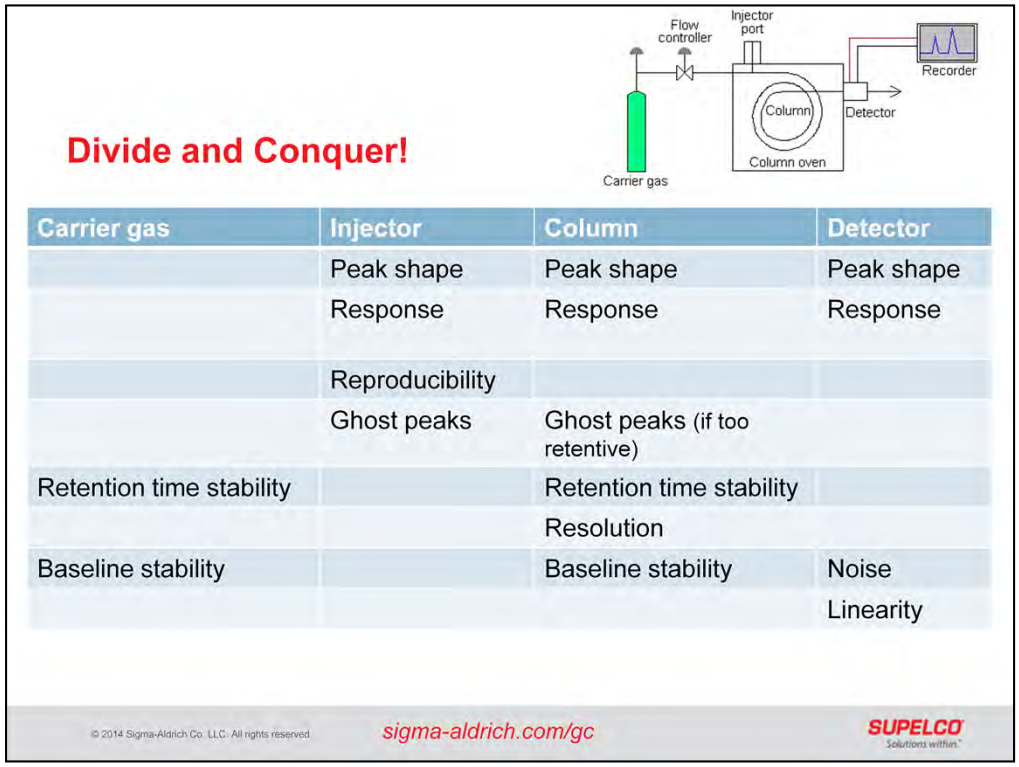
•Firstly, if a method stops producing acceptable data, make sure nothing has changed without your knowledge. This includes the method parameters and the condition of the instrument.

•If you are working on a new method, rule out operating parameters as the source of the problem, consider how things such as the liner type, injector settings, column choice, etc. can affect the chromatography.

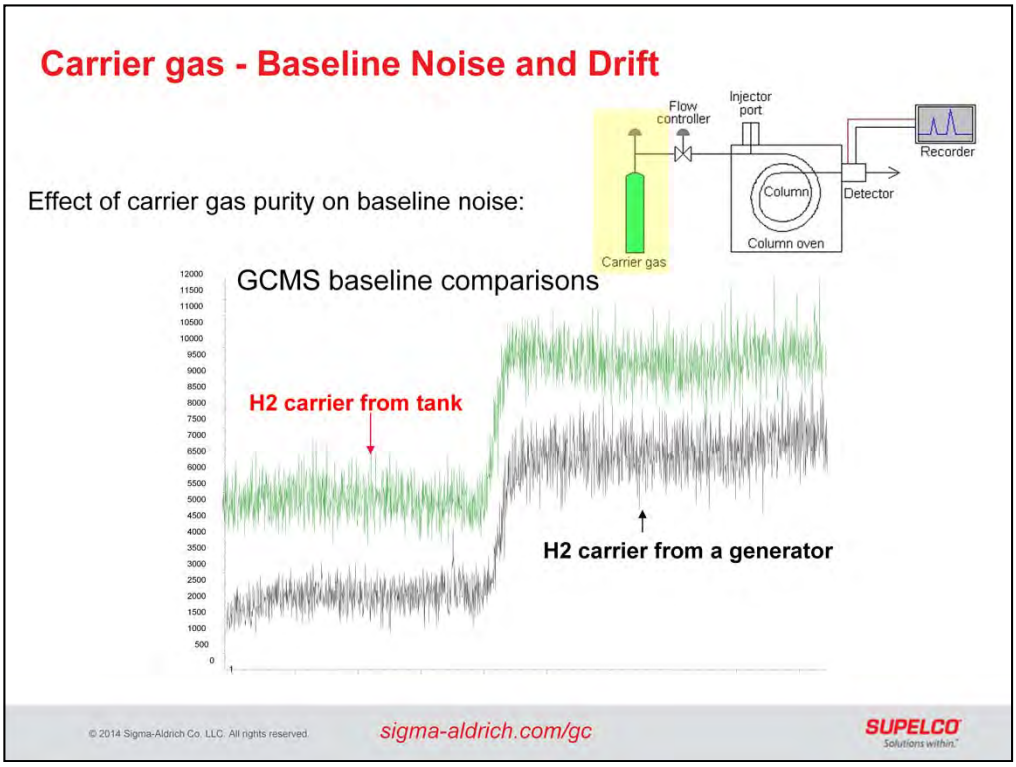


A systematic approach is always best. It is tempting to take a "shot-gun" approach to troubleshooting…that is, change a bunch of things at once and hope the issue is resolved. The problem with that is that often we do not know what change fixed the problem. This flowchart gives some guidance on how to isolate where the problem is coming from.

First, check operating parameters, if they look OK, run a reference standard…something you are very familiar with. In most cases, this is the point at which we actually know there is a problem..i.e. it is a standard of some sort that just does not look right. Next, install a test column if you have one…a column reserved just for this purpose that is in good conditions. If the problem persists, switch the detector or the inlet…that way you can isolate the problem to one or the other.



Or in a different format, this table summarizes some typical problems encountered in GC and the parts of the system often to blame. Some problems, such as peak shape, can originate from multiple sources. In cases like this, the flowchart approach on the last slide will help to isolate the source of the problem.



Let's start with the carrier gas in the GC system. Impurities in the gas are often the cause of baseline issues.

In this example, hydrogen carrier from two different sources, tank and generator, was compared in a GC-MS system. A higher baseline, i.e. noise, was observed with the hydrogen from the tank. The flow was increased halfway through the run to see if the pattern continued, which it did, confirming the carrier gas as the source.

To help prevent column bleed and other problems…remember gas purification

Minimum recommendations for removal

	Helium	Hydrogen	Air	Nitrogen	P-5
Oxygen	X				X
Water	X		X		
Hydrocarbons	X	X	X	X	X
Halocarbons					X

A wide variety of purifiers are available

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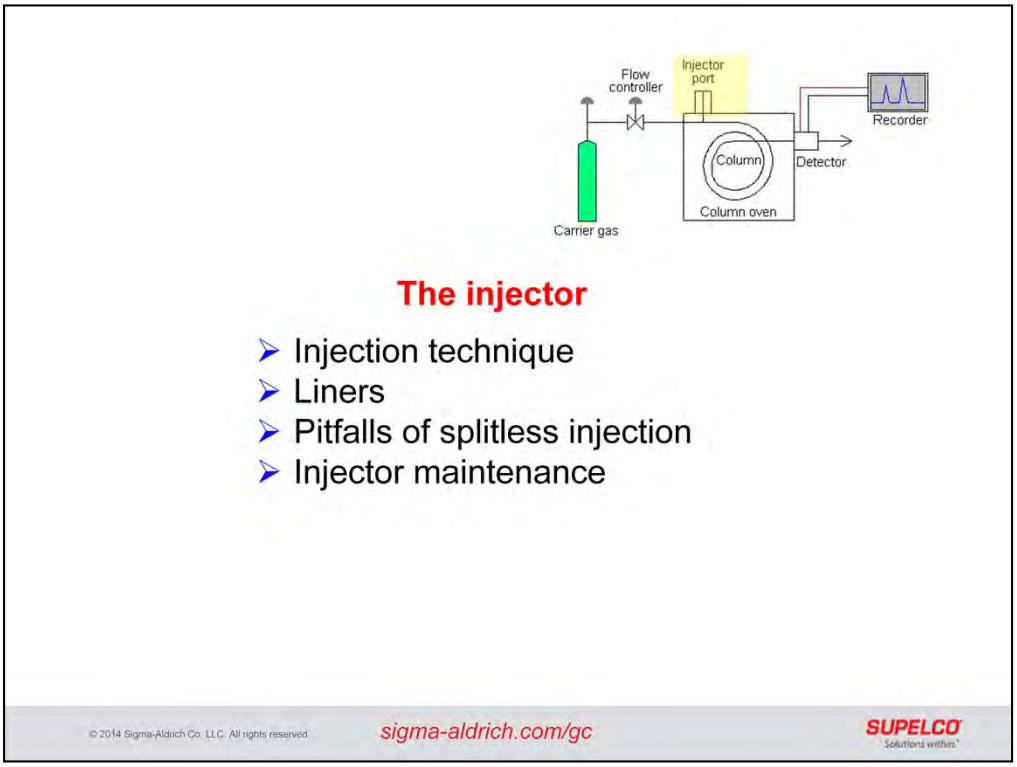
Protect your instrument with purification on the gases used to supply it. That includes carrier and detector gases.

Even high purity gas can become contaminated from activities after the gas is put into the tank by the supplier, things such as:

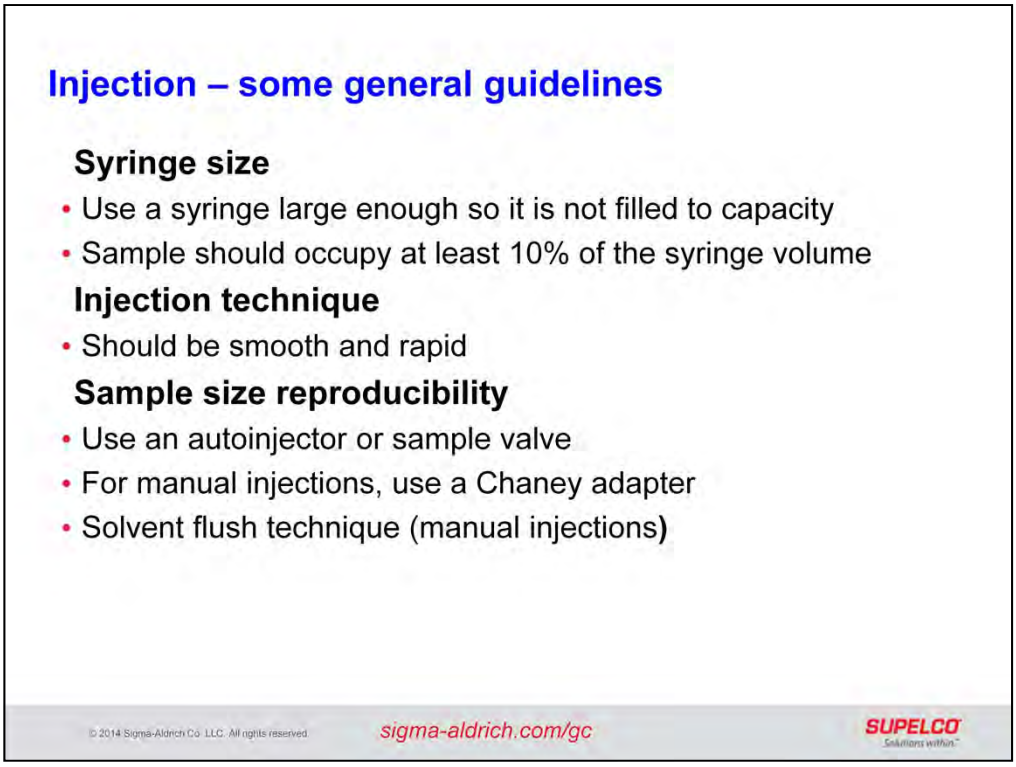
- Changing cylinders
- Dirty Gas lines
- Off-gassing from the regulator
- Or if the cylinder itself was dirty before the gas was put into it.

Clean carrier gas will extend column life, reduce noise, and increase sensitivity. Clean H2, air and detector makeup gases will result in better sensitivity

This chart shows some minimum recommendations for purification. Remember that purifiers do not have infinite capacity. They need to be replaced eventually. I like to use indicating traps if possible, especially down stream from other purifiers. That way, it is easy to tell when purifiers upstream need to be changed.



Moving on to the injector….this is probably the source of the majority of problems encountered in GC. There is a lot happening in the injector. We will take a look at injection technique, briefly at liner types, and at some of the issues associated with splitless injection. Finally I will talk about some routine injector port maintenance issues and what can happen if these are ignored.

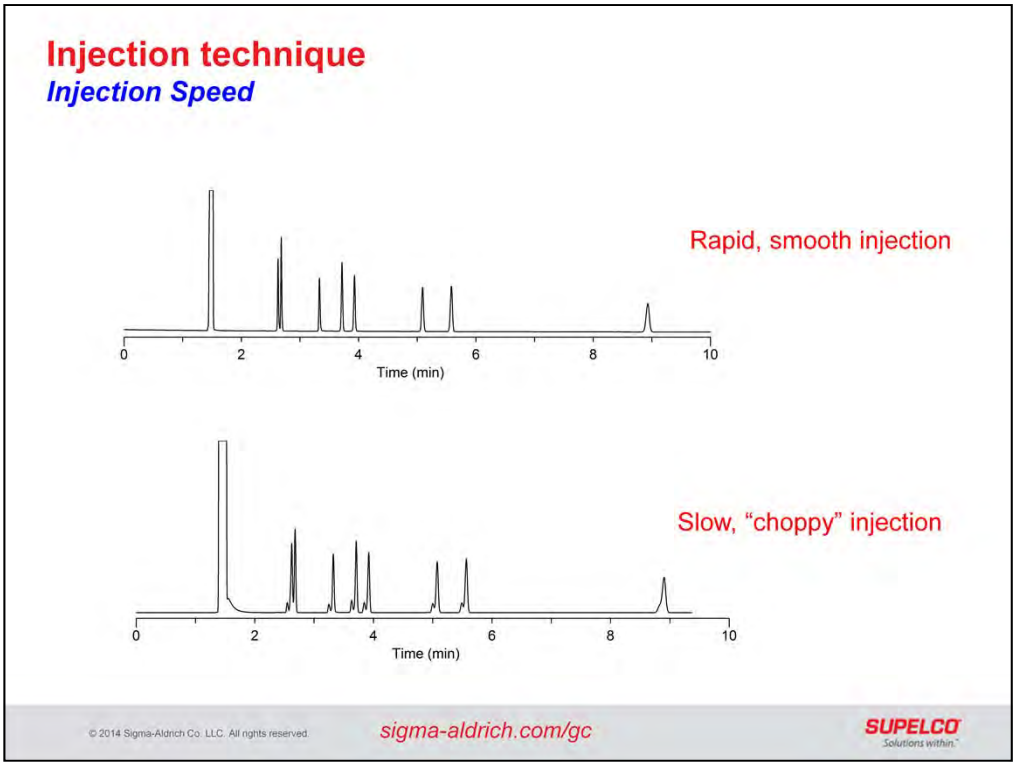


First, let's talk about some general guidelines for injection.

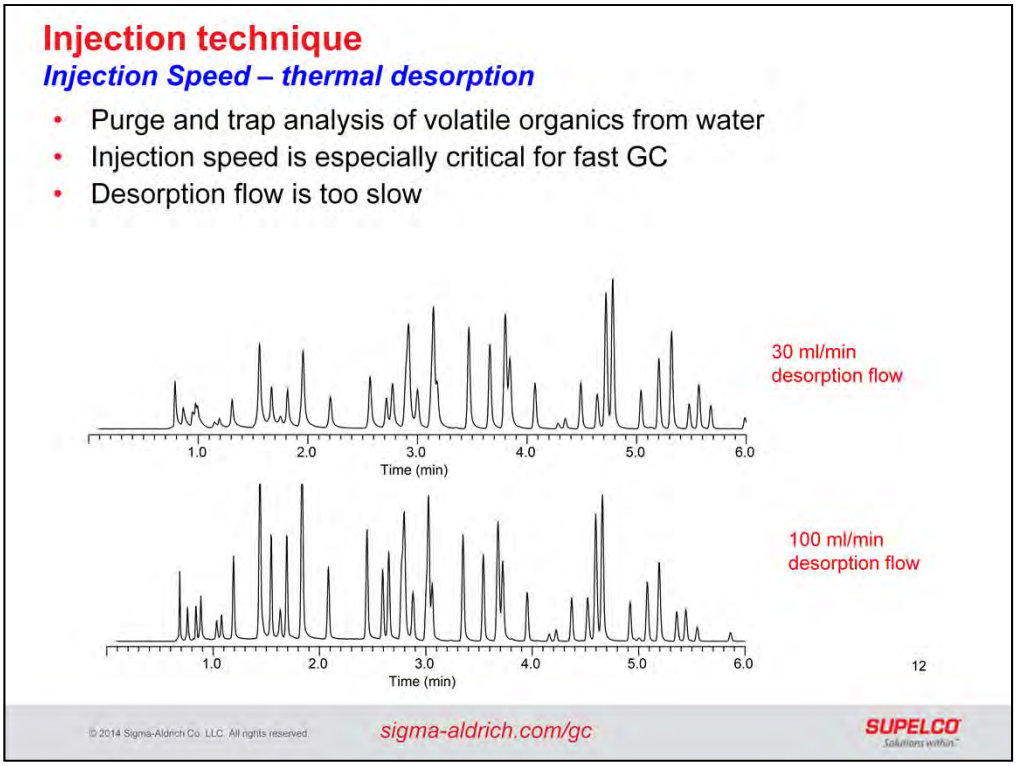
•Make sure the syringe size is appropriate…sample should occupy at least 10% of the total volume, but not more than 50%.

•The injection technique should be smooth and rapid. This is more of an issue for manual injection.

•Make sure injections are reproducible, i.e. you are getting the same response for the same sample when injected multiple times. If you are doing manual injection, use of the solvent flush technique and or a Chaney adapter will help with this.

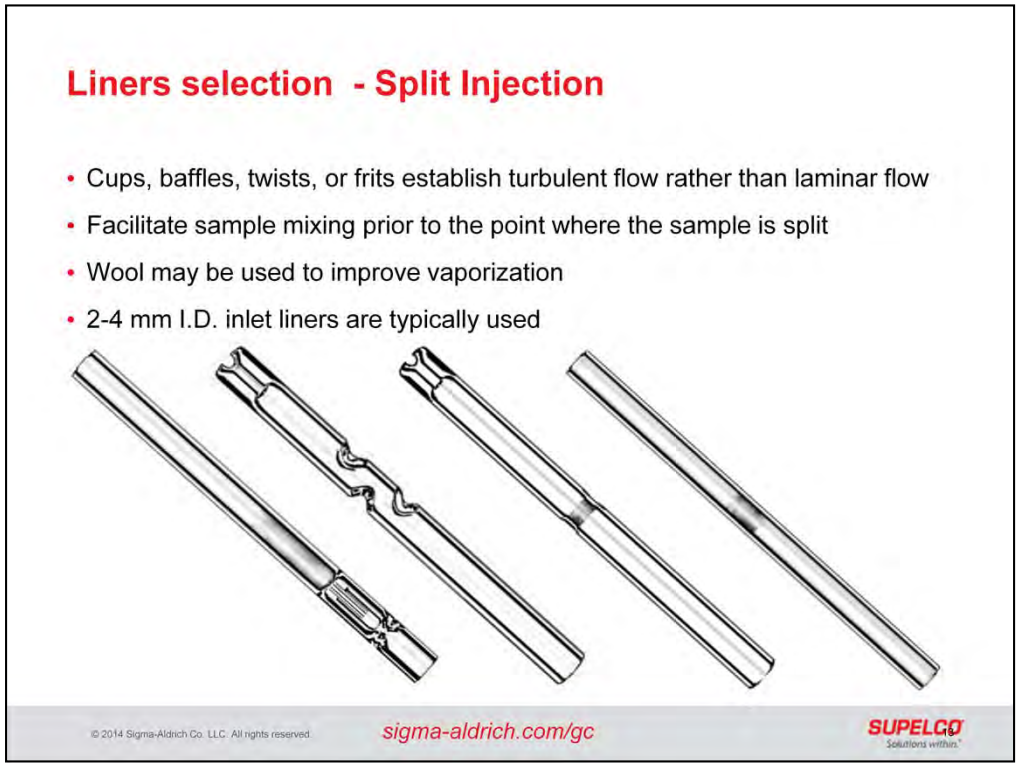


How about injection speed? This example shows the effect of a slow, choppy injection vs. a rapid smooth injection. We get double peaks in the first portion of the run if the injection is too slow. That is because sample is not delivered into the inlet in a single plug. Instead we get a main plug of sample followed by a small residual bit. The main plug of sample focuses and starts moving through the column followed by the smaller residual portion producing the double peak effect you see.

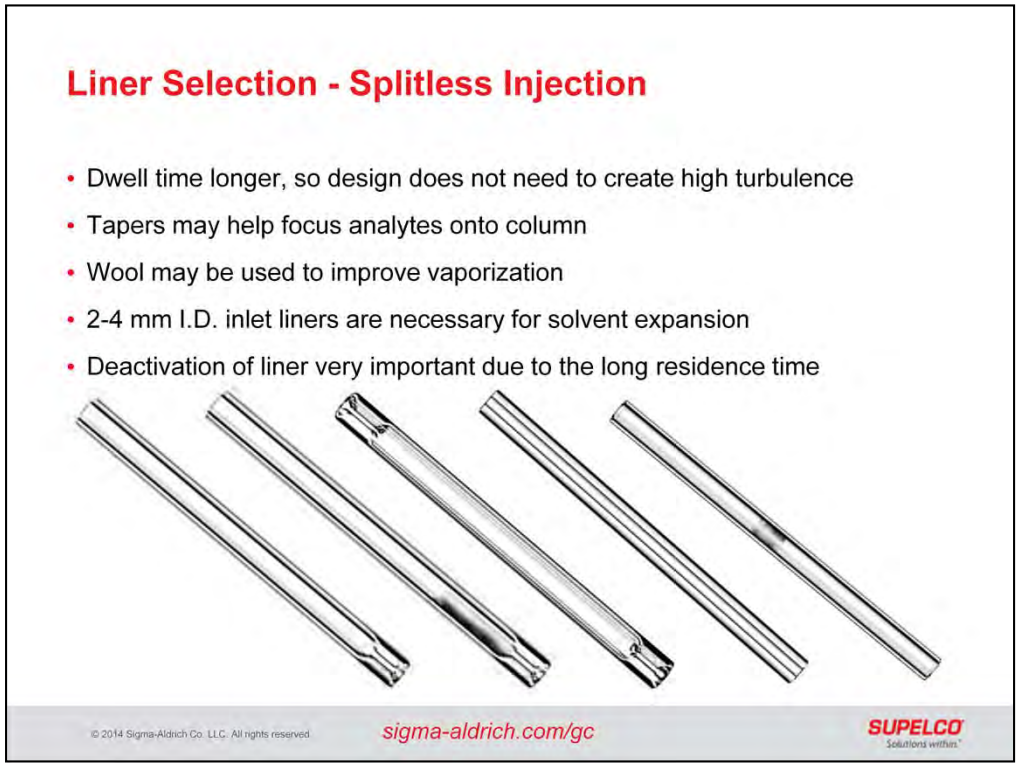


Even when no solvent is involved, such as in thermal desorption or purge and trap, sample transfer rate (which we can think of as “injection speed”) can affect peak shape. Instead of solvent delivering the sample, flow is sending it from an outside trapping source into the inlet. This must occur quickly, to keep analytes in a tight band after leaving the trapping source. If it occurs too slowly, analytes will broaden out inside the transfer line and inlet.

In this example, we can see the effect of desorption flow on peak shape in a purge and trap analysis. This is especially critical in fast GC because sample transfer must occur onto the column much quicker than a conventional analysis.

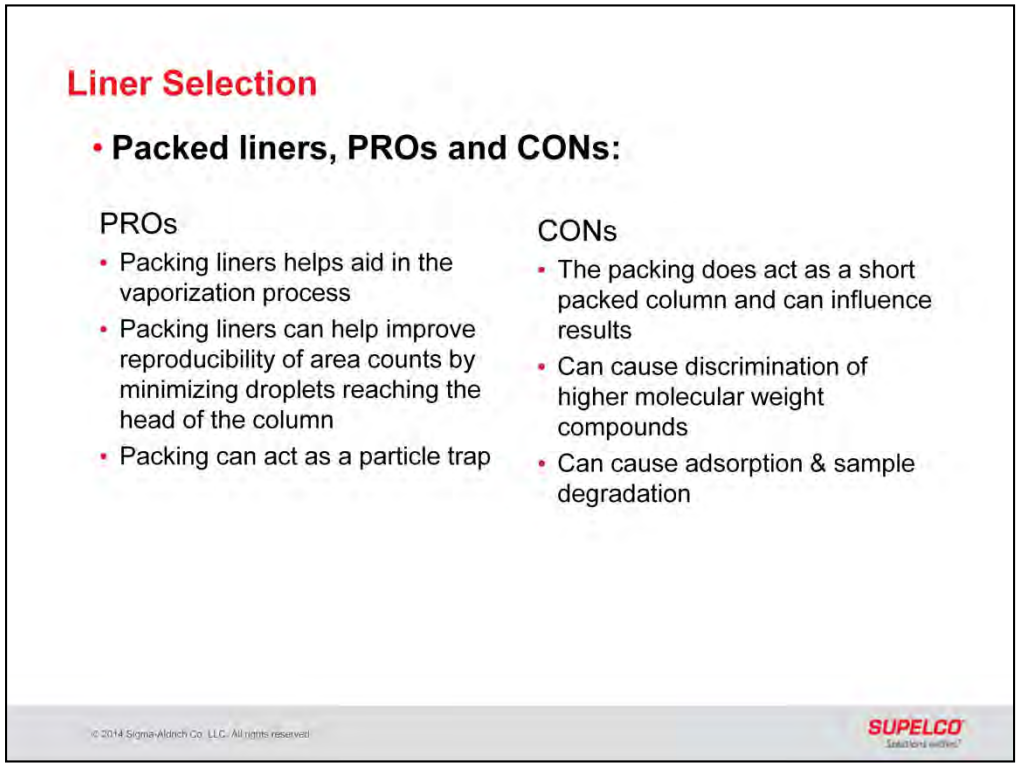


As for GC injection type, most people are doing either split or splitless. In either case, liner selection can impact response, and in some cases, peak shape. For split injections, these are a few liner types that are used. Split liners are often characterized by some sort of internal construction which aids in mixing of the sample with the high flow of carrier gas going through the inlet. This internal construction can be baffles, a cup-type design, or the traditional favorite, glass wool.

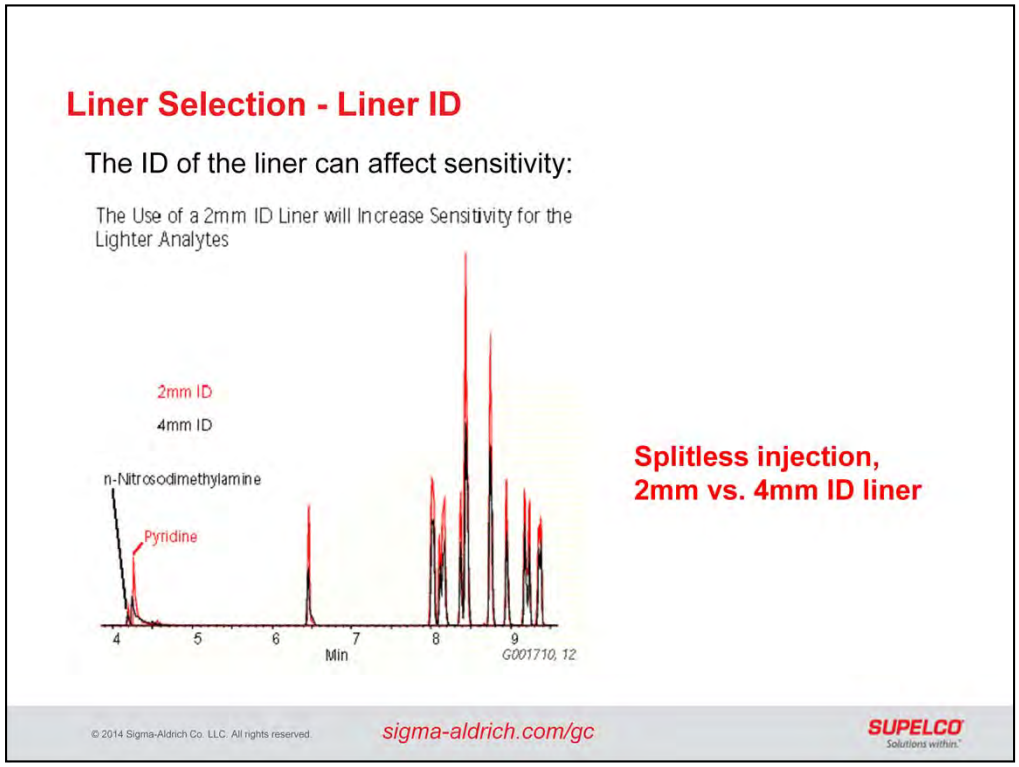


These are some liner styles typical for splitless injection. Flows through the liner are much lower during splitless than split, so residence time of the analytes in the liner will be longer. In this case, the use of tapers is helpful in focusing the analytes onto the column and helping to protect them from any active sites present at the bottom of the inlet.

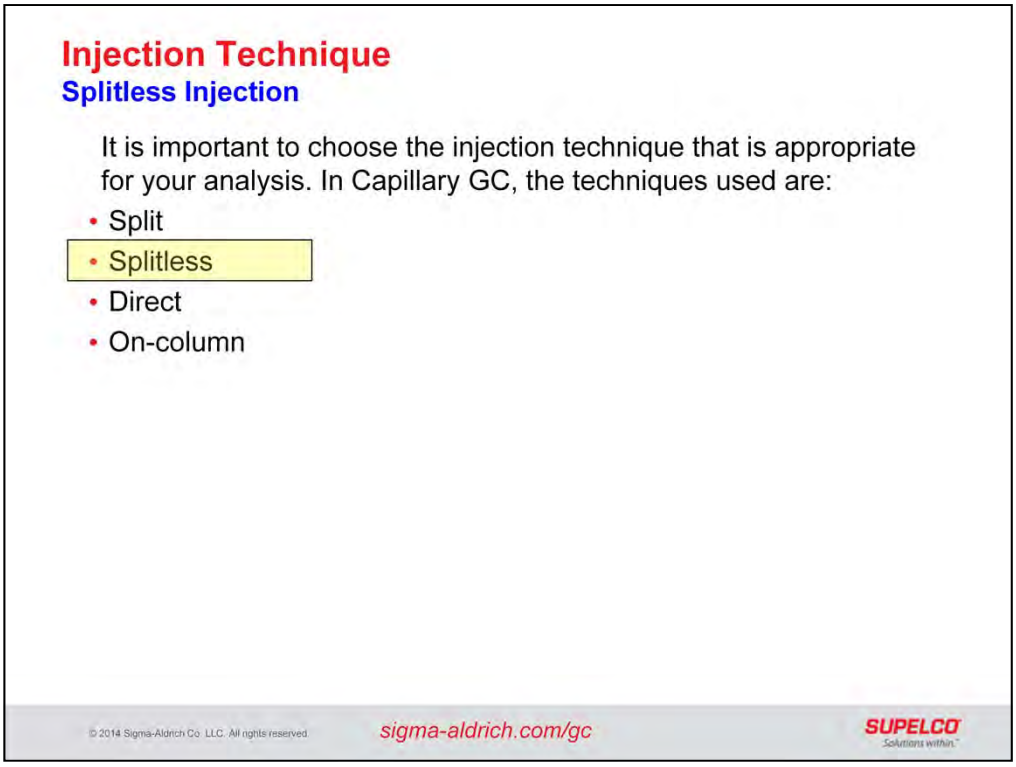
Wool inside the liner helps with vaporization of the solvent. Finally, deactivation is very important for all types of liners, but especially for splitless due to the longer residence time of analytes in the inlet.



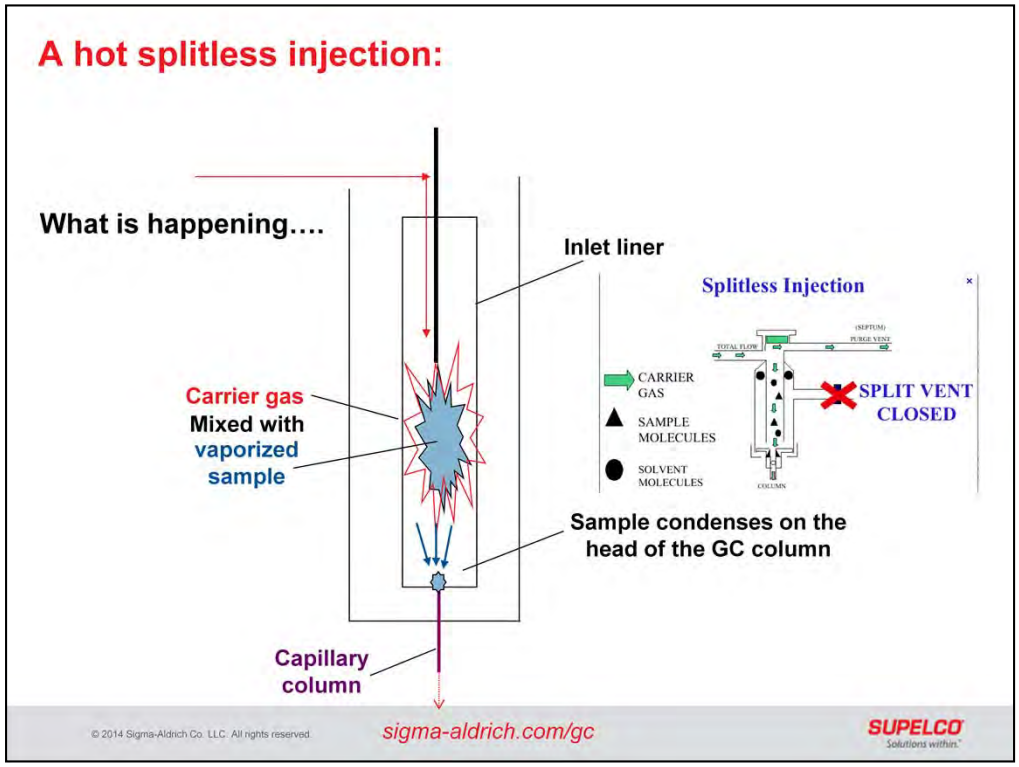
What about packing in liners? There are good and bad aspects of that. Packing is usually glass or quartz wool, and sometimes it can be a particle-type material. The positive aspects of packed liners is that the packing can aid in vaporization, improve injection reproducibility, and act as a trap for particles such as septa. The downside of packing is that it can cause MW discrimination for high boilers, and adsorption and sample degradation. In the case of particulate packings, the packing can act as a very short packed column and influence results.



When choosing a liner, be aware that the inner diameter or ID can impact peak shape. In this example we see the difference between 2 and 4 mm ID liners for lighter early eluting compounds. The smaller volume of the 2 mm ID liner increases the rate with which analytes reach the column – this provides for more efficient focusing. This really effects response for these early eluting analytes. However the downside when doing a splitless injection, as we will discuss, is the reduced capacity.

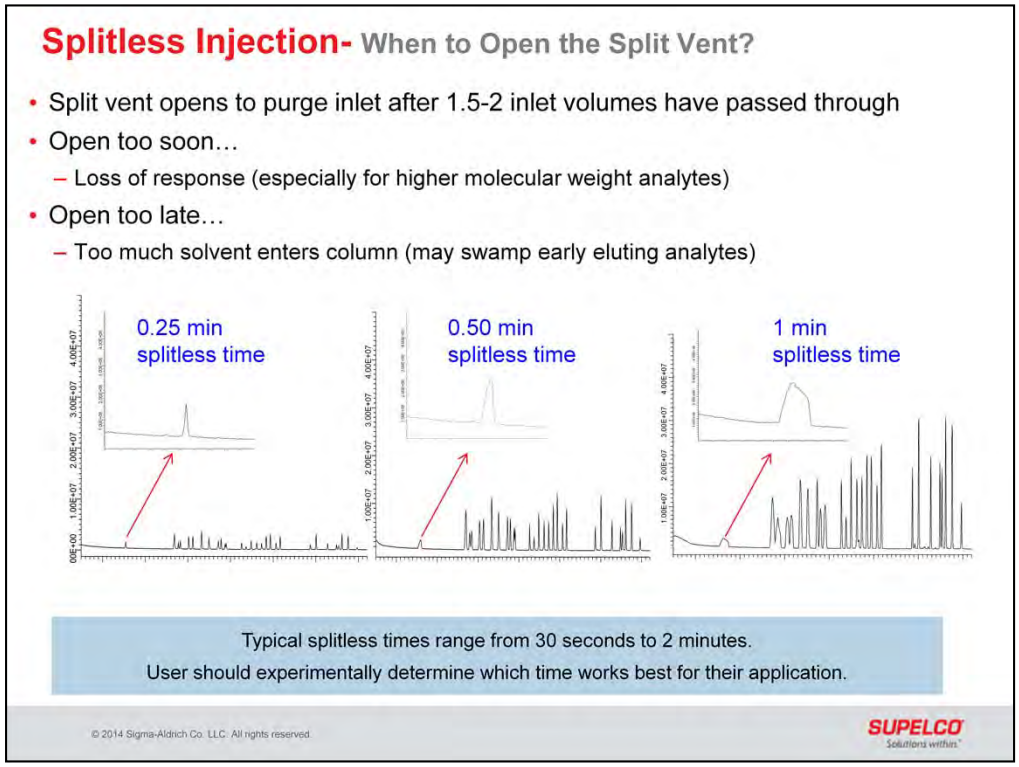


I have shown you liners for split and splitless injection, now I want to focus on splitless injection and some of the problems associated with it. This injection type is more challenging than split in that basically, more can go wrong.



Let's talk first about what happens during a splitless injection. With the split vent closed, all flow coming into the inlet is going onto the column. While it is closed, [CLICK] sample is injected into the hot inlet. It vaporizes [CLICK] and carrier gas is mixed with it. The sample and some solvent then condense on the head of the GC column [CLICK] (which is at a lower temperature than the inlet…this is called the solvent condensation effect). Sample enters the column and the separation begins. The split vent is then opened, and any solvent vapor remaining in the liner is flushed out.

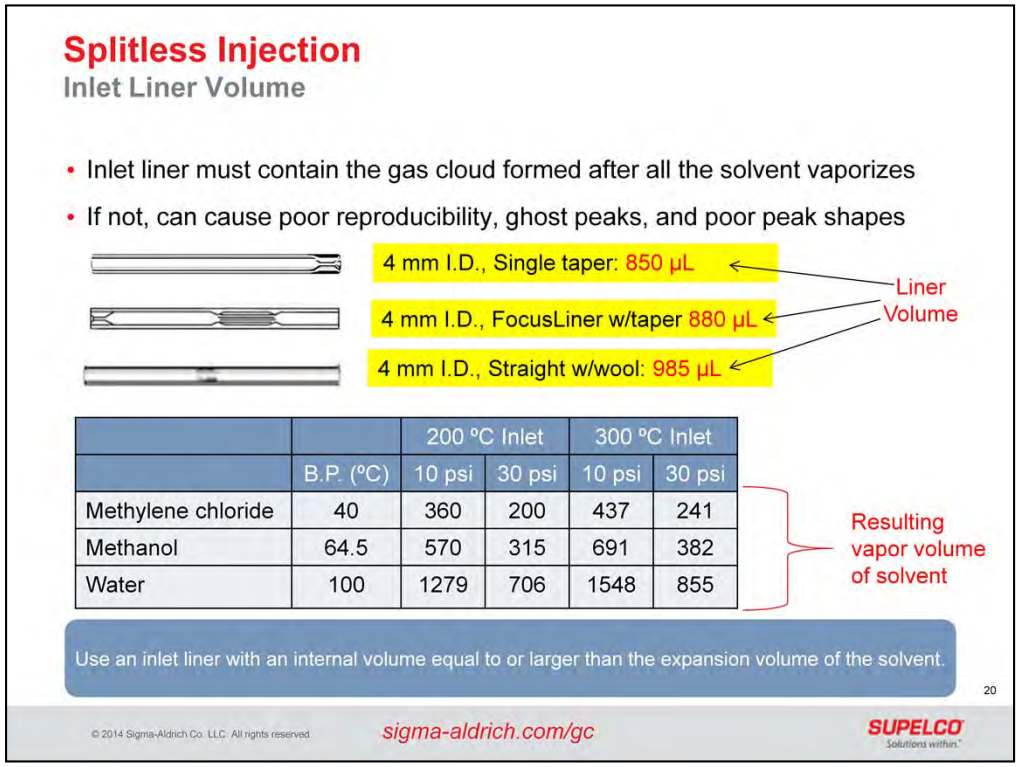
So, you can see there is a lot going on. Problems can be associated with vaporization, deactivation in the liner, focusing on the column, split vent times, etc.



When should the split vent be opened?

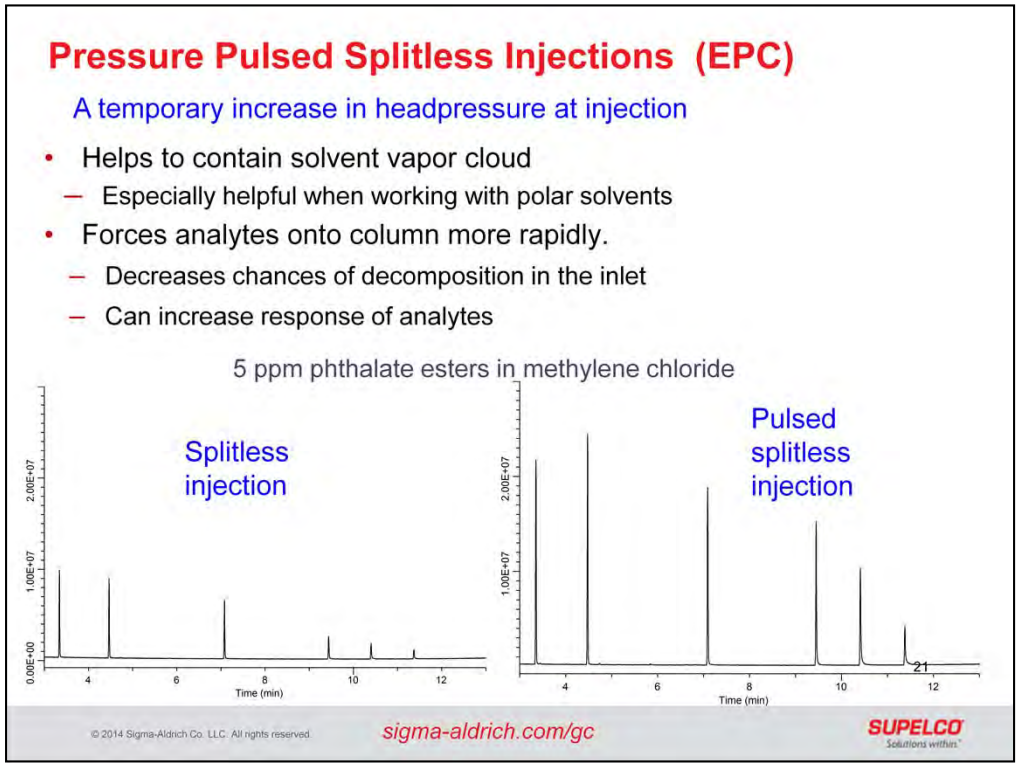
- Typically, it should be opened after 1.5 to 2 inlet volumes of has have passed through it.
- Too soon causes loss in response of heavier analytes
- Too late results in more solvent entering the column, which can swamp out or distort early eluting analytes.
- The time the splitter is opened is commonly referred to as “splitless time”. A typical range for this is from 30 secs to 20 min. You will have to determine what time works best for your application.

For example [CLICK], in this application increasing the splitless time from 0.25 min to 0.50 [CLICK] to 1.0 min [CLICK] definitely increases the response of these analytes. However, as we do this more solvent enters the column [CLICK] which distorts the peak shape for the first analyte. So, in order to use the longer splitless time you would have to change something else such as perhaps column film thickness or starting temperature to help with focusing of this analyte.



		200 °C Inlet		300 °C Inlet	
	B.P. (°C)	10 psi	30 psi	10 psi	30 psi
Methylene chloride	40	360	200	437	241
Methanol	64.5	570	315	691	382
Water	100	1279	706	1548	855

Let's talk for a minute about inlet liner volume. This is especially an issue in splitless injection because all the sample/solvent is going into and staying in the inlet. When liquids vaporize, they expand into a gas cloud. The liner must be able to hold the volume of this cloud. If it cannot, the result is often poor reproducibility, ghost peaks as sample backs up into gas lines, and poor peak shapes from difficulty in focusing sample. Three liner types are shown here with their internal volumes listed. In the table below, the volume of the vapor clouds formed at different temperatures and pressure have been determined for 3 different solvents. Water by far forms the largest vapor cloud. This is one of the reasons it makes a poor solvent in GC. In general, polar solvents will form larger vapor clouds than nonpolar solvents. This limits the liner style and injection volume that can be used with them.



A way to deal with a large vapor cloud, for example say you are working with a polar solvent…is a pressure pulsed injection. This can be done with any GC system equipped with electronic pressure control (EPC).

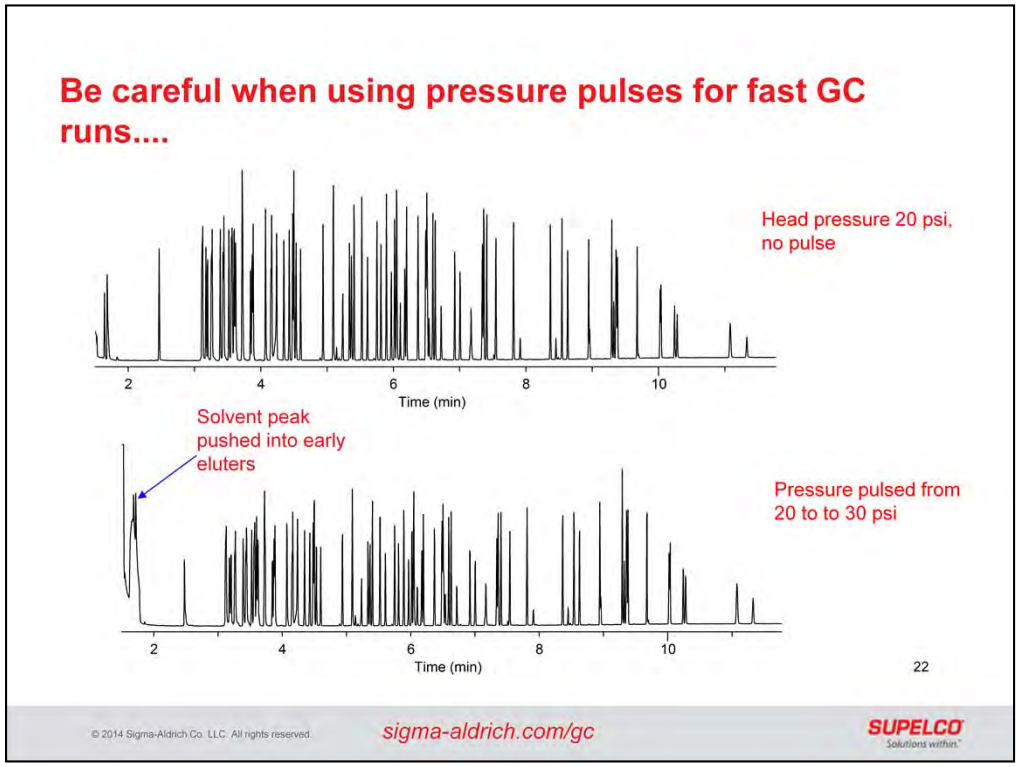
A pressure pulse is a temporary increase in headpressure at injection.

- This increase will help to contain the vapor cloud of the solvent,
- and it will force analytes onto the column more rapidly. Moving the analytes through the inlet faster decreases chances that they will decompose, and it can also increase analyte response.

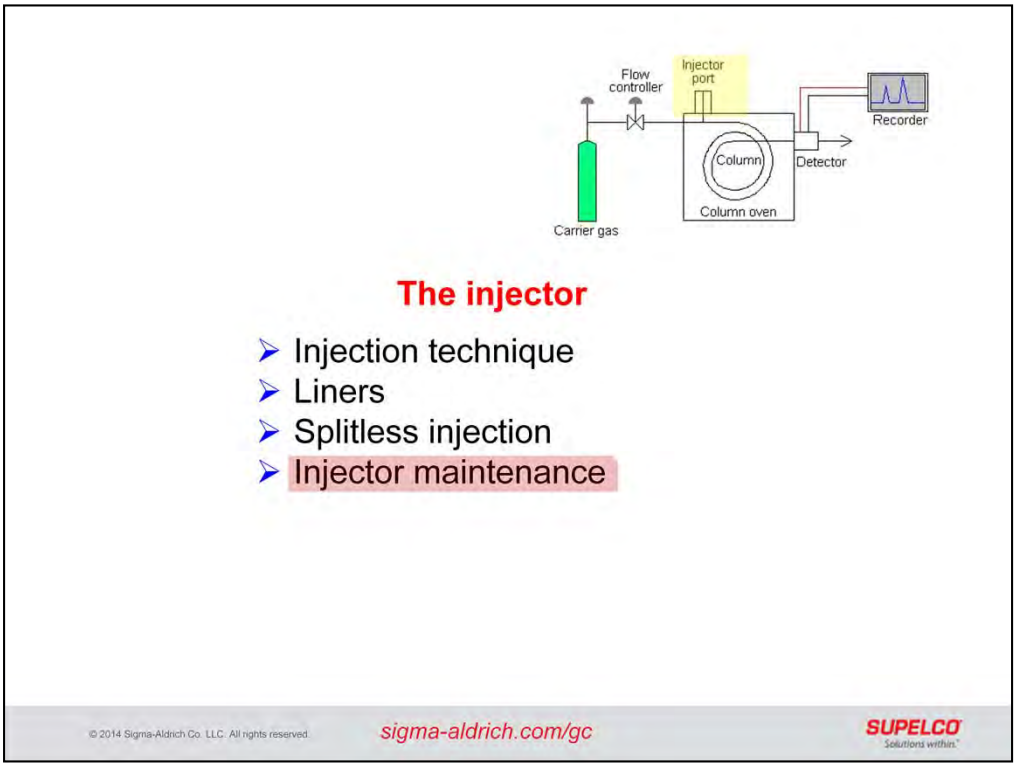
[CLICK]

In this example, we can see the difference in response for phthalates injected using a regular splitless injection and a pulsed splitless injection. [CLICK] In this case, the injection solvent was methylene chloride, so the vapor cloud could be contained by the liner even without the pulse. The pulse did increase response by forcing the analytes onto the column faster than a standard splitless injection.

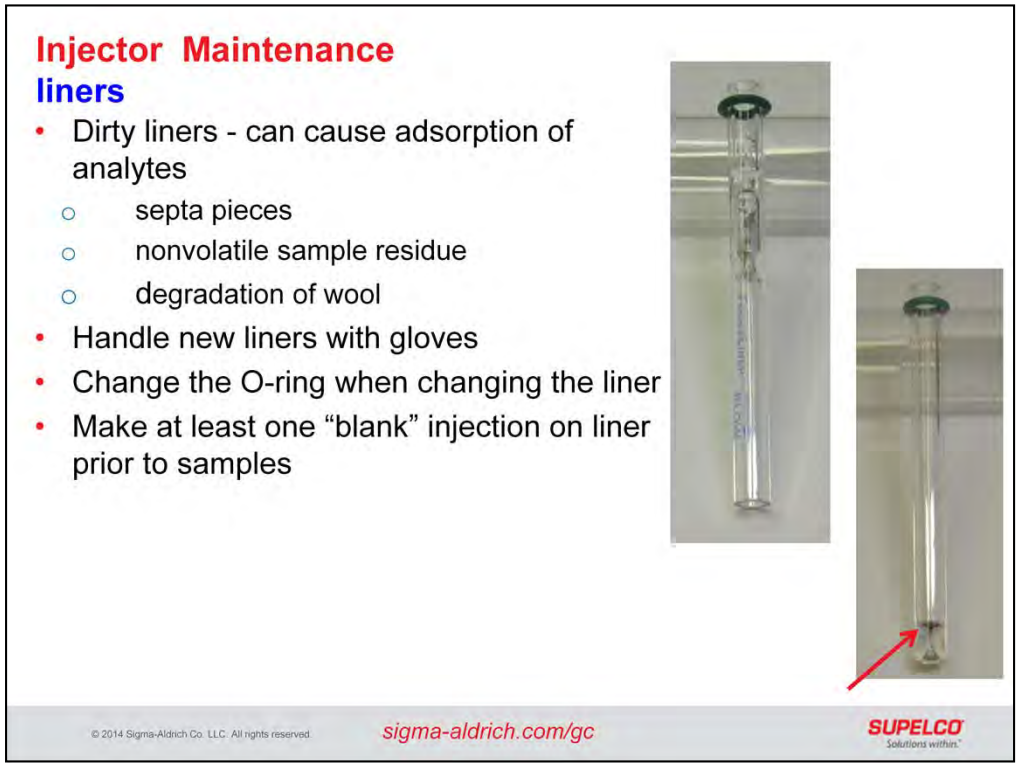
This is a 1 uL injection of a 5 ppm phthalate standard in methylene chloride.
Inj: 250C
Liner: 4 mm ID FocusLiner
Splitless time: 0.75 min.
For pulsed injection: 50 psi pulse until 0.75 min.
Column flow 1 mL/min
Column: SLB-5ms: 20 x 0.18 x 0.18
Oven: 65C (1), 15C/min to 325C, 10 min hold.
Detector: MS, full scan (interface at 330C)



Pressure pulsed injection is a very useful, but use some care with it as you will be putting much more solvent on the column than normal splitless. In some cases, this could interfere with early eluting peaks in your run as we see in this example.



Finally with regards to the injector, a little bit about maintenance.

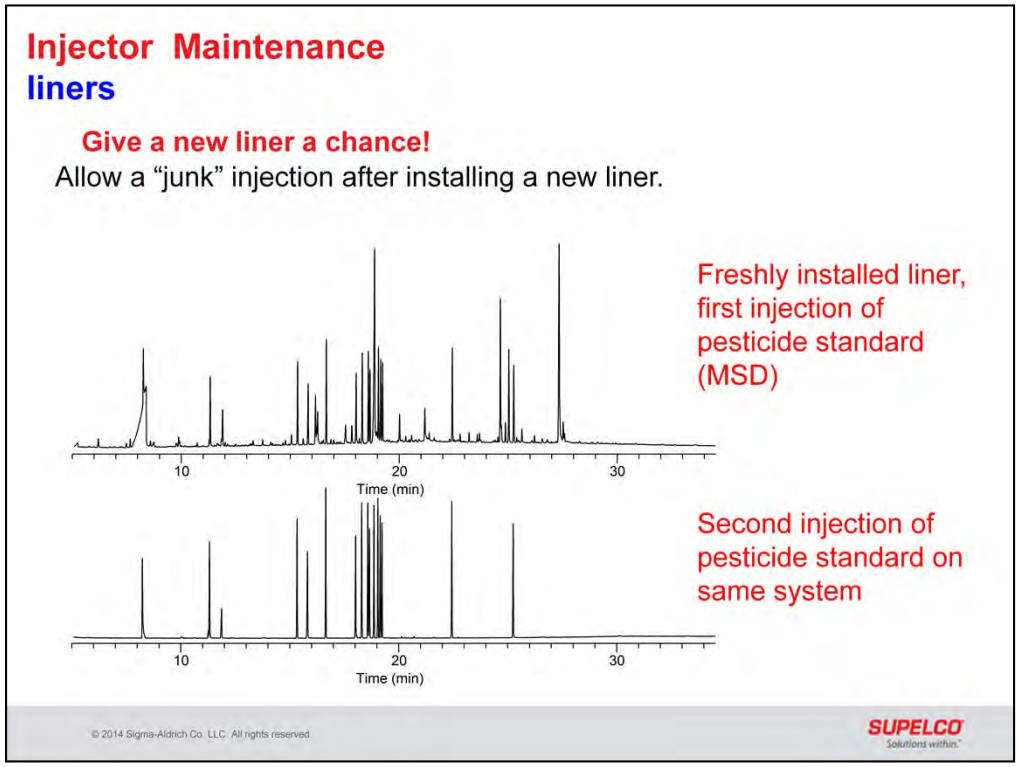


Liners get dirty…the matrix present in the samples will determine how quickly this happens. Even if your samples are clean, septa pieces can lodge in them. Also, quartz or glass wool in liners can degrade over time.

[CLICK] In this liner, you can see residue and small septa pieces in the middle portion. Also, the wool has compacted from being "hit" with the syringe many times.

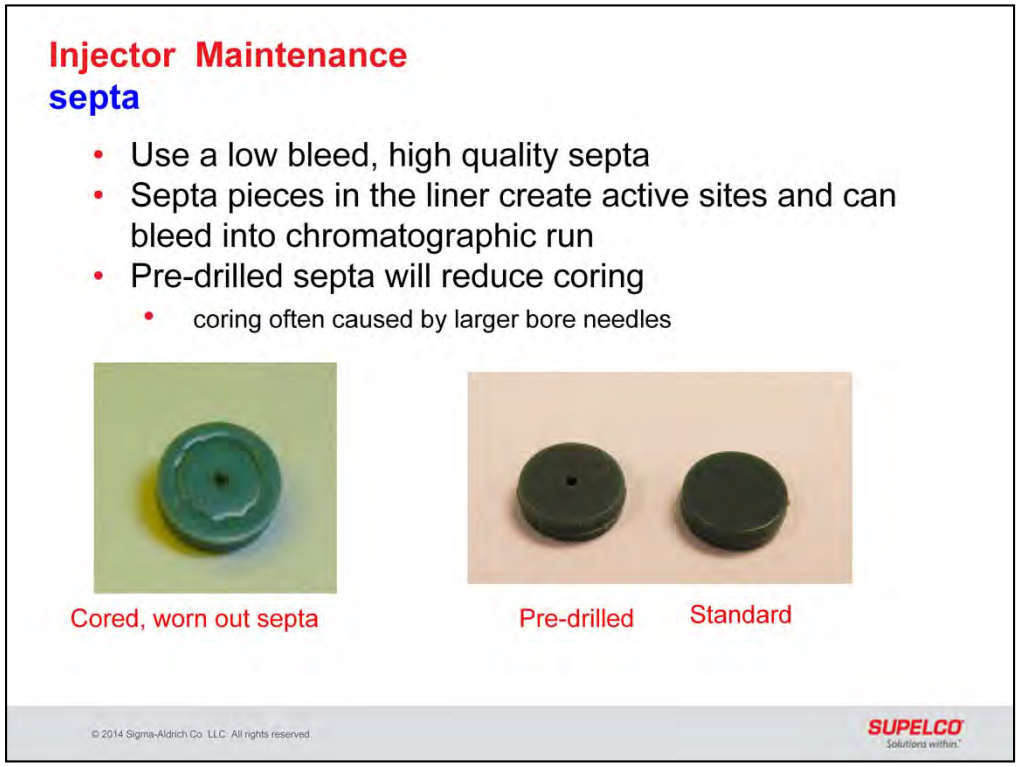
[CLICK] This liner is contaminated with residue at the tapered portion. Both of these liners would be impossible to clean and reuse.

- When changing a liner, use gloves to handle the new one. The oils on your hands can adhere to the glass and bleed off into your runs.
- Change the O-ring with the liner. The O-rings help to make an airtight seal at the top of the inlet. They will flatten with use. Flattened O-rings will not reseal as well as new, round O-rings.
- Also, as you will see, make at least one blank injection (solvent or whatever) on the liner before running samples.



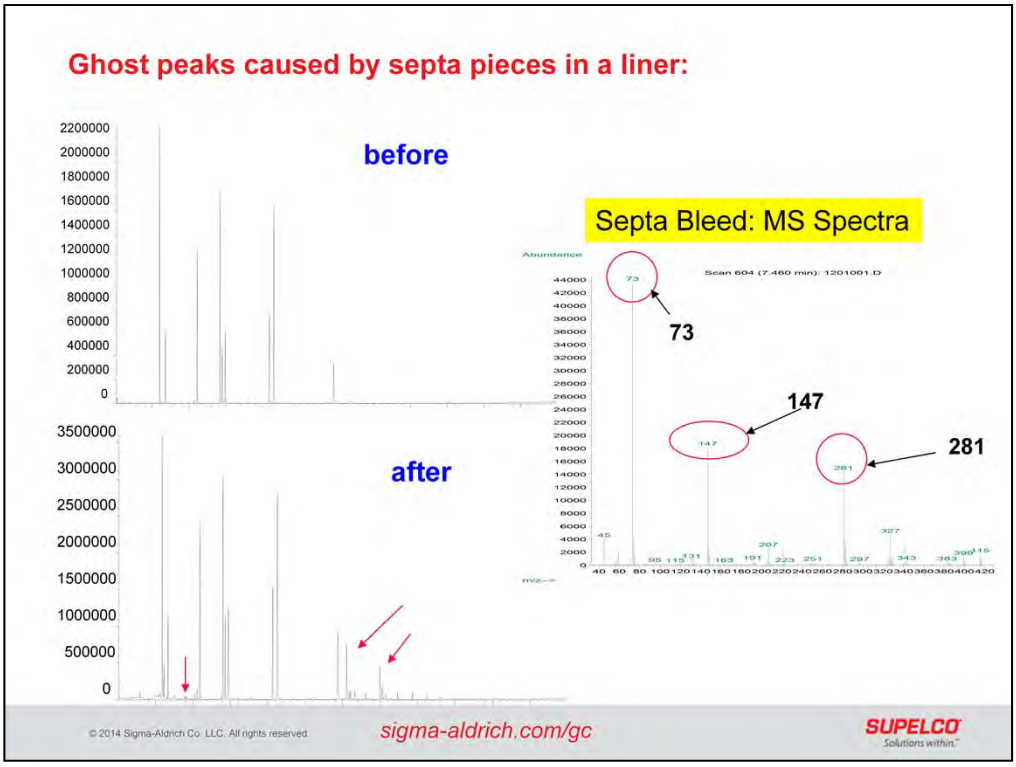
This is why a blank injection is recommended on a newly installed liner prior to running samples. New liners will often produce extraneous peaks as you see here. [CLICK] This goes for liners that you have "recycled" as well as brand new ones fresh out of the box.

These will usually go away after one or two runs.

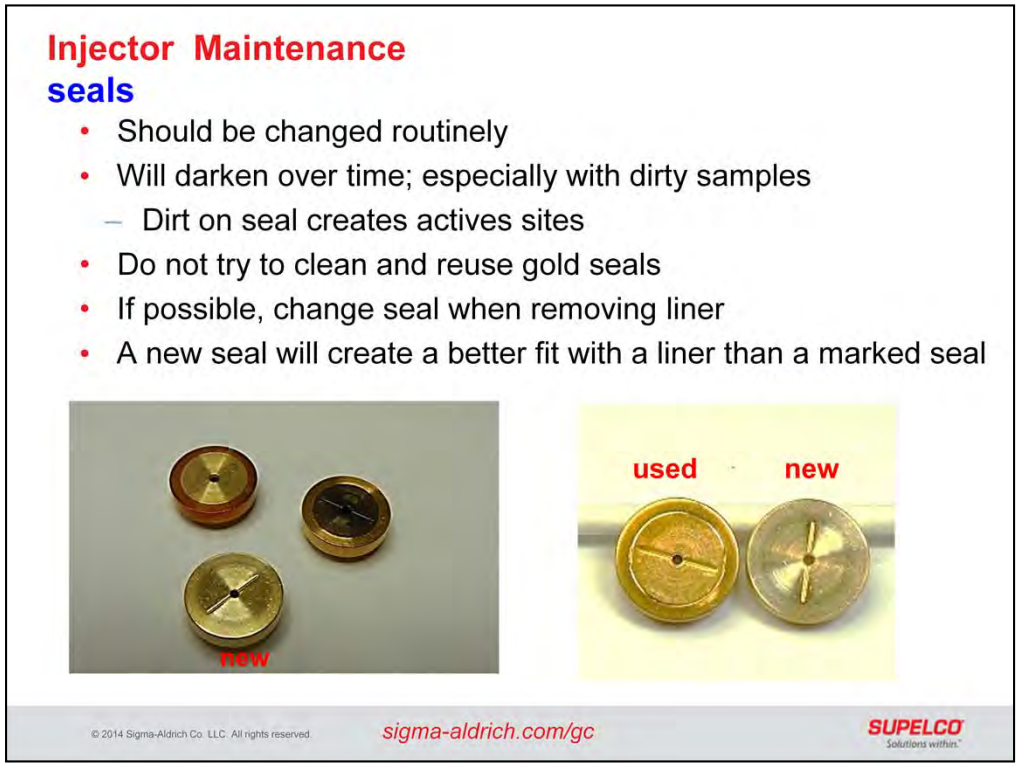


For septa, use a high quality, low bleed variety. Septa that are punctured in the same spot repeatedly, or with a large gauge needle, can become worn out, with a large injection hole forming, as you see on the left here…. This is called “coring”. When this happens, septa pieces often fall into the liner, creating active sites. Theses pieces can also bleed silane material that produces peaks in the GC run. If this occurs commonly with your application, consider using a pre-drilled septa. Pre-drilled are less likely to core due to the presence of a pre-formatted penetration site for the needle.

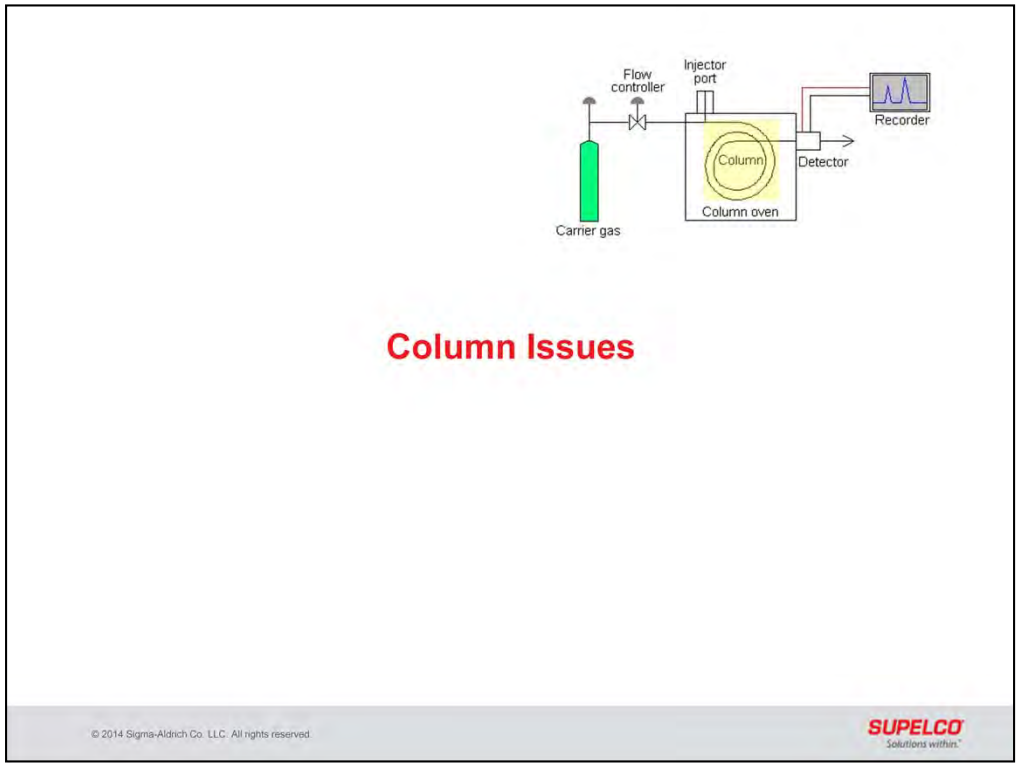
These septa on the right show the difference between standard and pre-drilled. The predrilled have this “injection hole” of sorts in the center.



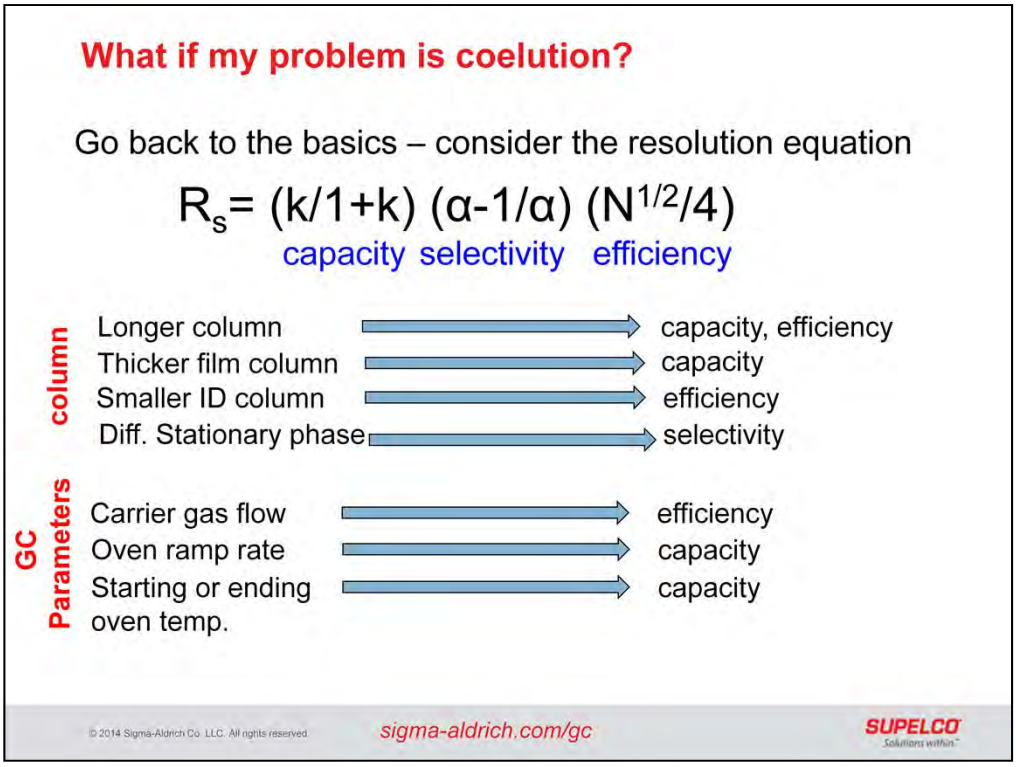
As I mentioned, when septa pieces start to bleed from the heat of the inlet, you will see extra peaks in the GC run. This is an example of what that looks like [CLICK] These peaks are silanes which bled off septa pieces, and were swept onto the column during an injection. If you are running a mass spec, these “septa” peaks as I call them, are easily identified [CLICK] by the presence of masses 73, 147, and 281 in their spectra.



- Seals sit at the bottom of the GC inlet, and should be changed routinely.
- [CLICK] The seals you see here are plated with gold to make them inert. These will darken over time; especially if exposed to dirty samples. The new seal is a shiny gold color vs. this used seal (point to upper one) with discoloration around the edge. This seal on the right is especially dirty – this dirt creates active sites.
- If you are using gold-plated seals, do not try to clean and reuse them- just replace them. You risk damaging the inert finish with cleaning.
- When you change the liner, it is a good idea to change the seal. A new seal will create a better fit in the inlet . [CLICK] If you look at the difference between a new and used seal, you'll see a circular indentation around the outside of the used seal. This forms when the liner seals down on the top. When the inlet is opened up, this seal is broken.



Now moving on to the column…..

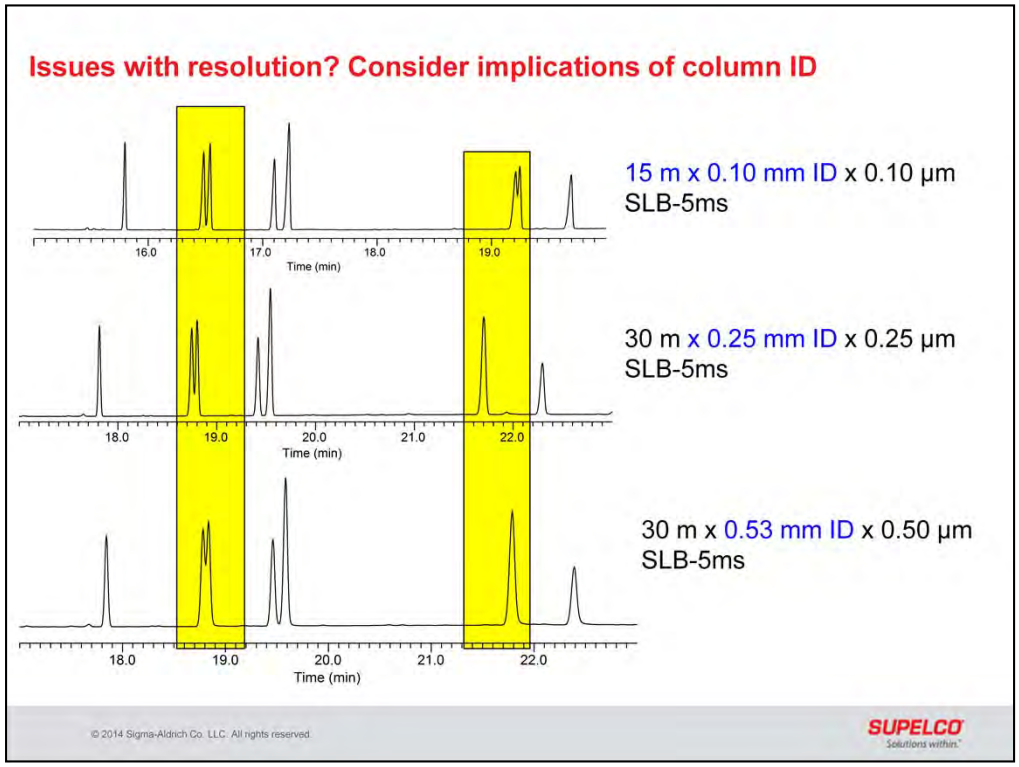


What if the problem is missing peaks? Consider that this could be due to coelution. In that case, go back to the basics. Look at the elements that contribute to an efficient separation. This is the resolution equation that describes these elements. Without going into a lecture on chromatography theory, just consider that resolution of peaks can be influenced by:

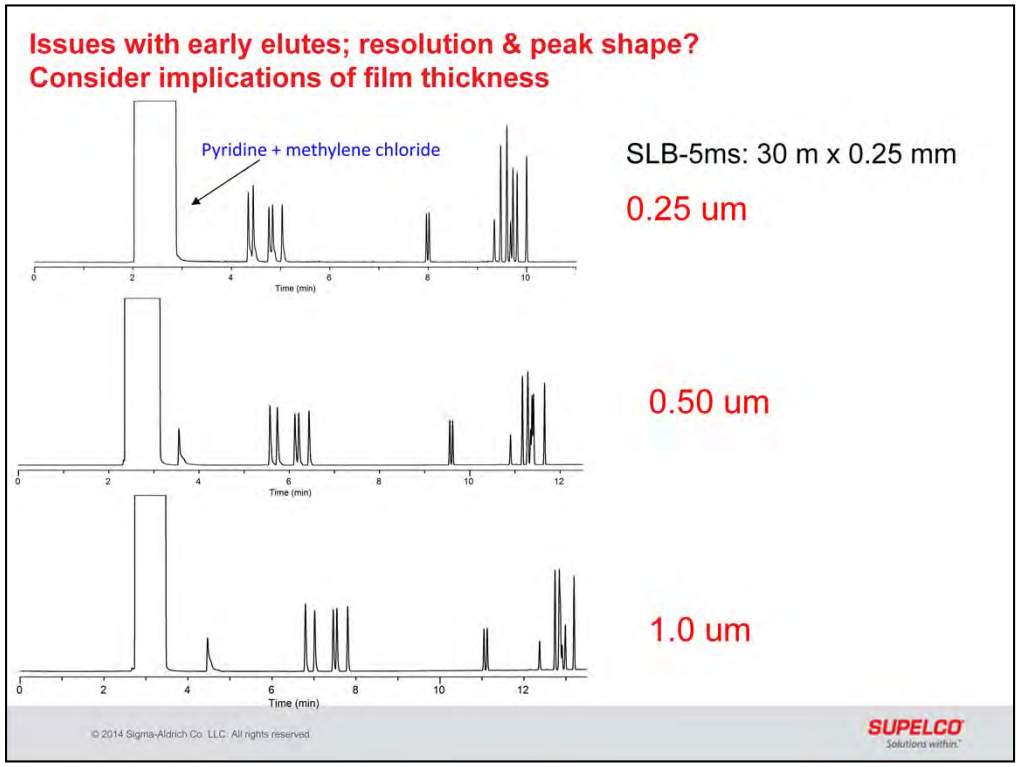
1. capacity, which is retention. This can be manipulated in different ways
2. Selectivity, which is manipulated by stationary phase chemistry
3. efficiency or plates, which can be manipulated by column dimensions and carrier gas

For example, By lowering the starting oven temperature to try and pull two peaks apart, you are manipulating the capacity term in the resolution equation.

Now we'll take a look at a couple examples of manipulating terms in this equation to increase resolution…

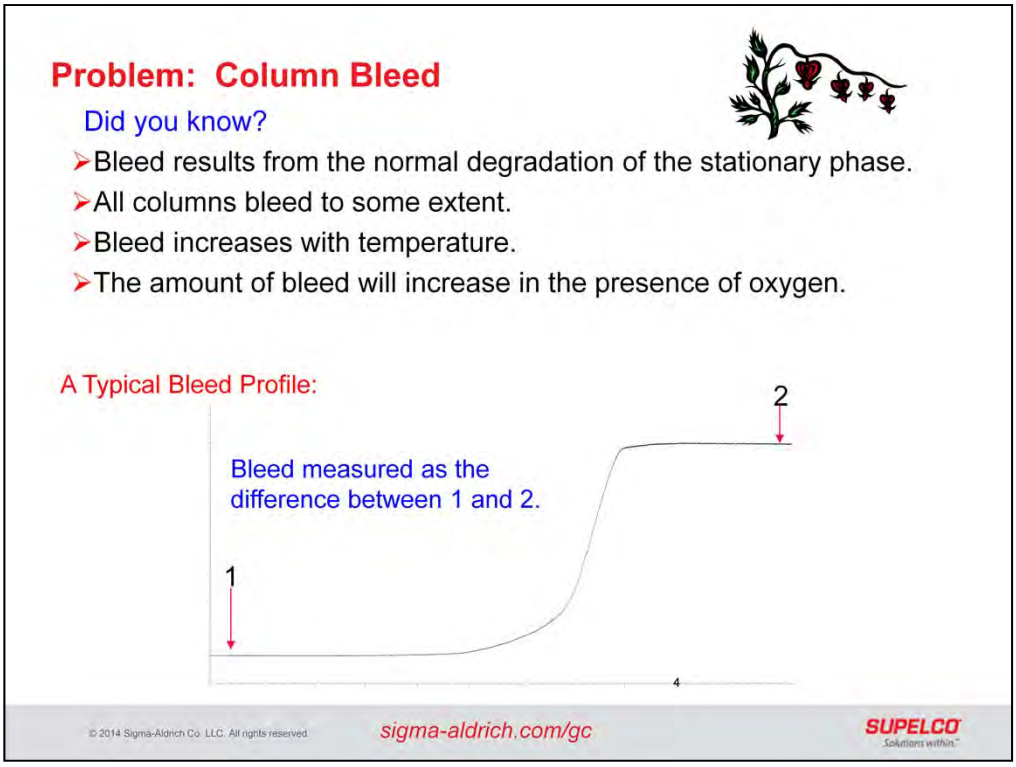


This shows the effect of reducing column ID on resolution. Of course, you have tried everything else first that does not involve changing the column and it has not worked at this point. Reducing column ID adds more efficiency because the smaller the ID, the more plates per meter the column has. If we look at theses sets of peaks [CLICK], resolution increases as the ID is reduced from 0.53 to 0.10 mm. The 0.10 mm ID column has so much more efficiency that a shorter column can be used.



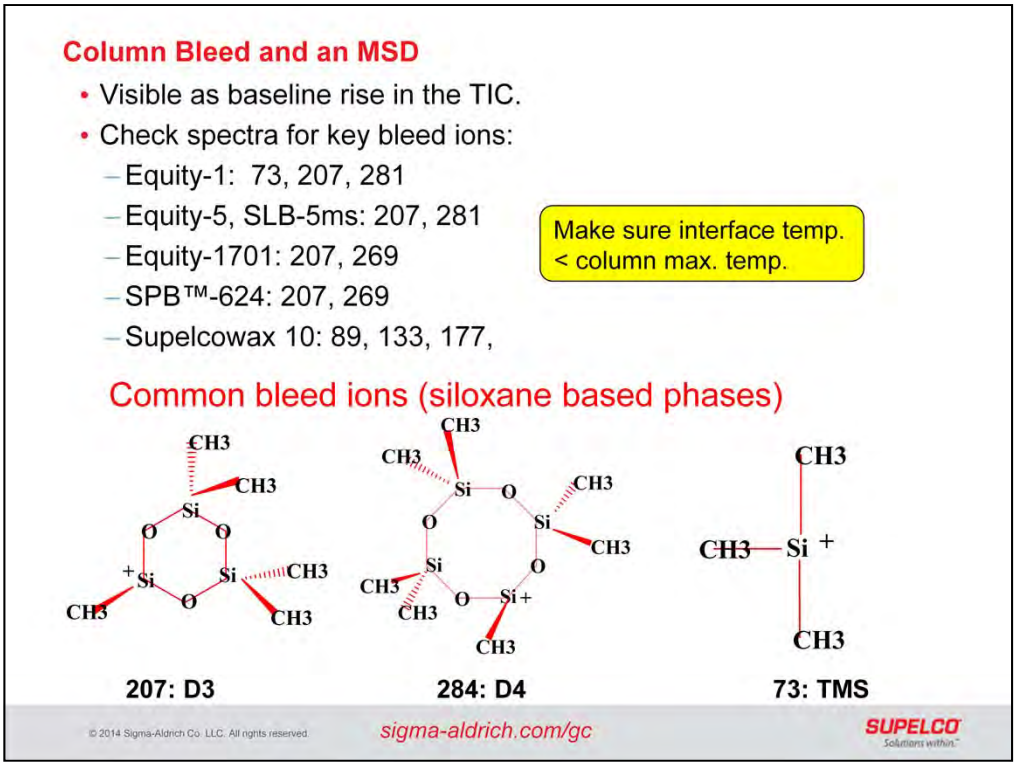
In this example, the effect of stationary phase film thickness is shown on early eluting peaks. This sample was run on the same length, ID and stationary phase, just with differing phase thickness. On this column (top cgram), the methylene chloride solvent coelutes with the first analyte peak, which is pyridine. Doubling the film thickness [CLICK] provides more retention and more phase for focusing the analytes. This has the greatest effect on the early elutes. We can now see the pyridine peak resolved from the solvent front. [CLICK] a 1 um film thickness provides even greater separation between the pyridine and methylene chloride.

One downside to increasing film thickness, is that it can adversely effect resolution for later eluters. We can see that here with the last cluster of peaks. We start with 7 peaks on the 0.25 um column, and end up with 5-6 peaks on the 1 um.



A common common-related complaint is bleed. What exactly is column bleed?

- It results from normal degradation of the stationary phase as it is exposed to heat, samples, etc.
- All columns bleed to some extent, even those designed to be “low bleed”…they just do not produce as much
- Bleed will increase with temperature; that is why the baseline rises with the oven ramp.
- If oxygen is present in the system, for example impure carrier gas, bleed will increase. This is oxygen acts to accelerate the degradation reaction that takes place to produce bleed.
- This is what a typical bleed profile looks like…as manufacturers, we quantitate column bleed by measuring the difference between the baseline at the upper and starting temperatures of the oven ramp.

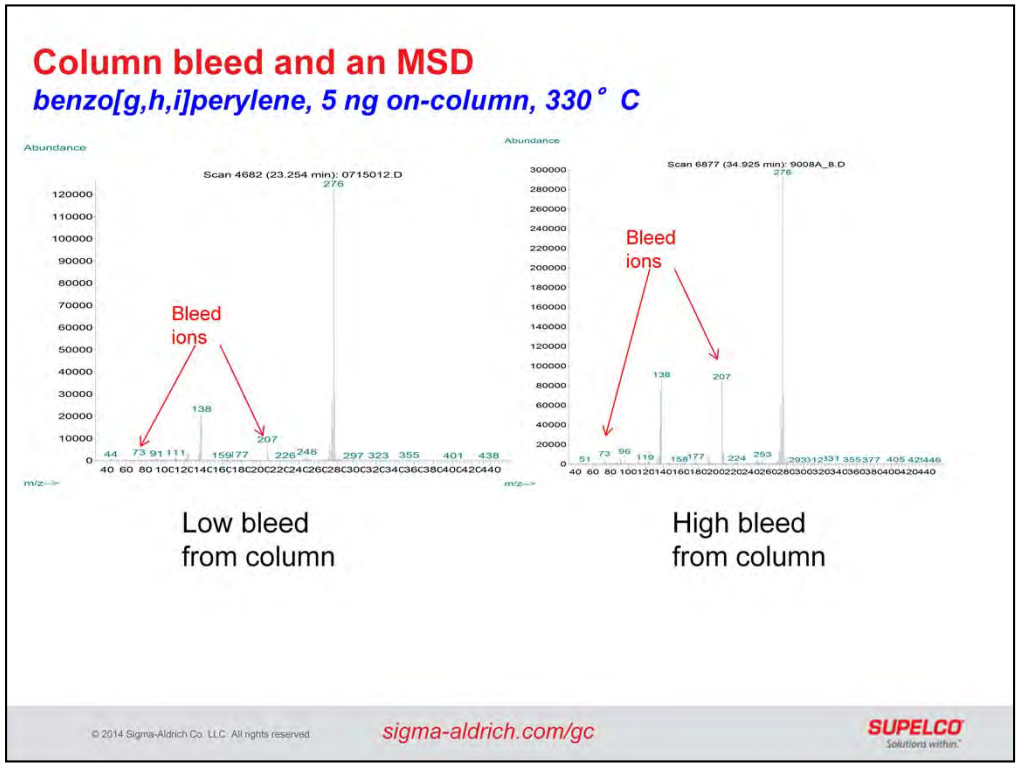


Analyses using mass spectral detectors (MSDs) are especially sensitive to bleed. This is because excessive column bleed can not only dirty the source, but also interfere with spectral identification.

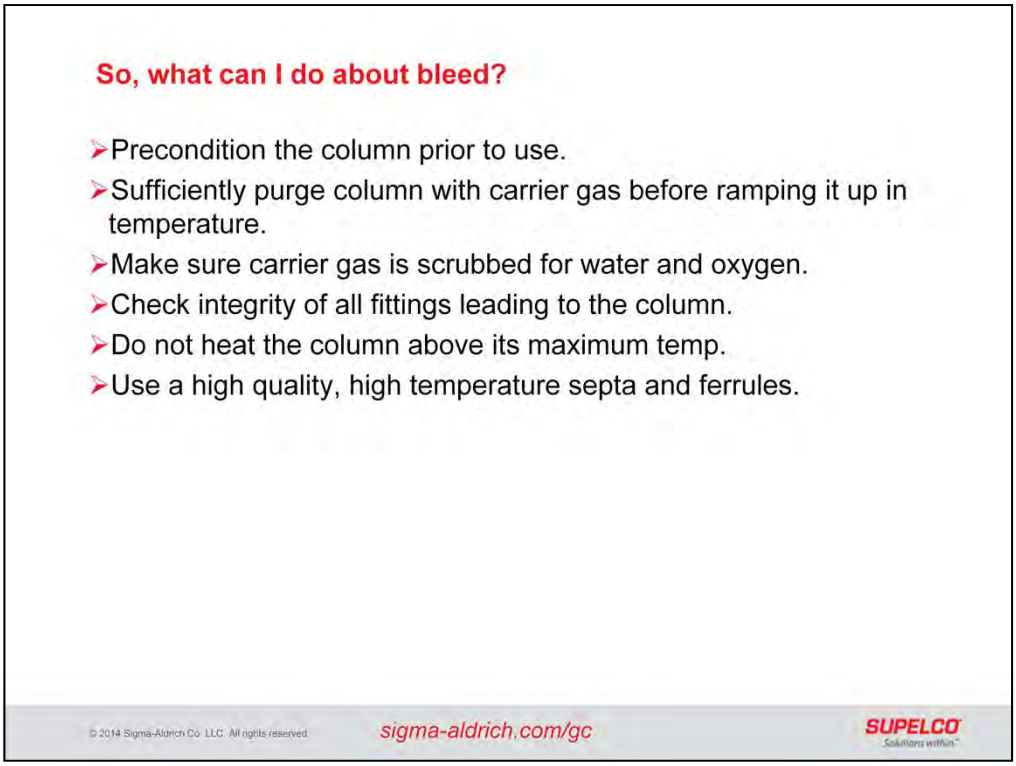
MSD users have the unique advantage of being able to tell if baseline rise is actually being caused by column bleed or something else.

These are some common bleed ions along with the structures attributable to them. If an unacceptable rise is observed in the TIC, spectra should be taken at various points to determine if the column is the source.

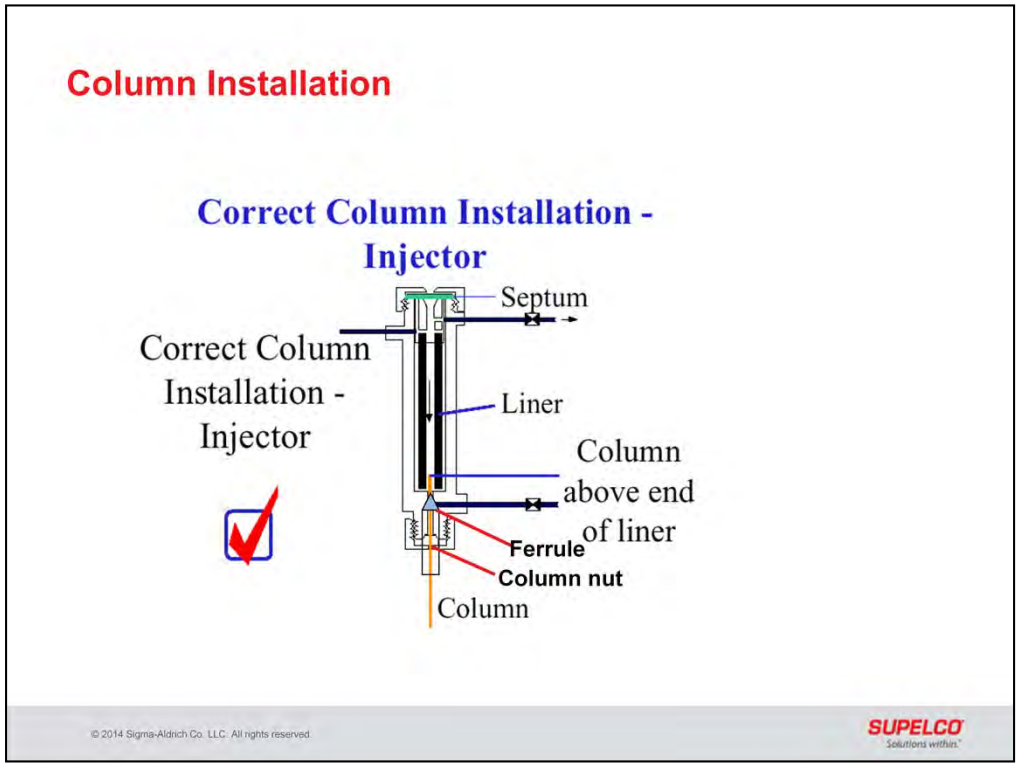
A high interface temp. (i.e. the transfer line) will not cause a classic bleed profile as we saw before, rather it will cause an increase in the baseline noise throughout the run.



This shows MS spectra of a compound that elutes at 330 ° C on a 5% phenylmethyl siloxane column. On a low bleed column, the spectrum shows levels of bleed ions 73 and 207 which are well below the key ions for identification and quantitation of this compound (138 and 276). On a high bleed column, the 207 ion is quite a bit higher in abundance, almost as high as the secondary ion (138) in the analyte spectrum. This can cause problems when trying to do library searching to match unknown spectra. The bleed ions can interfere with proper identification.

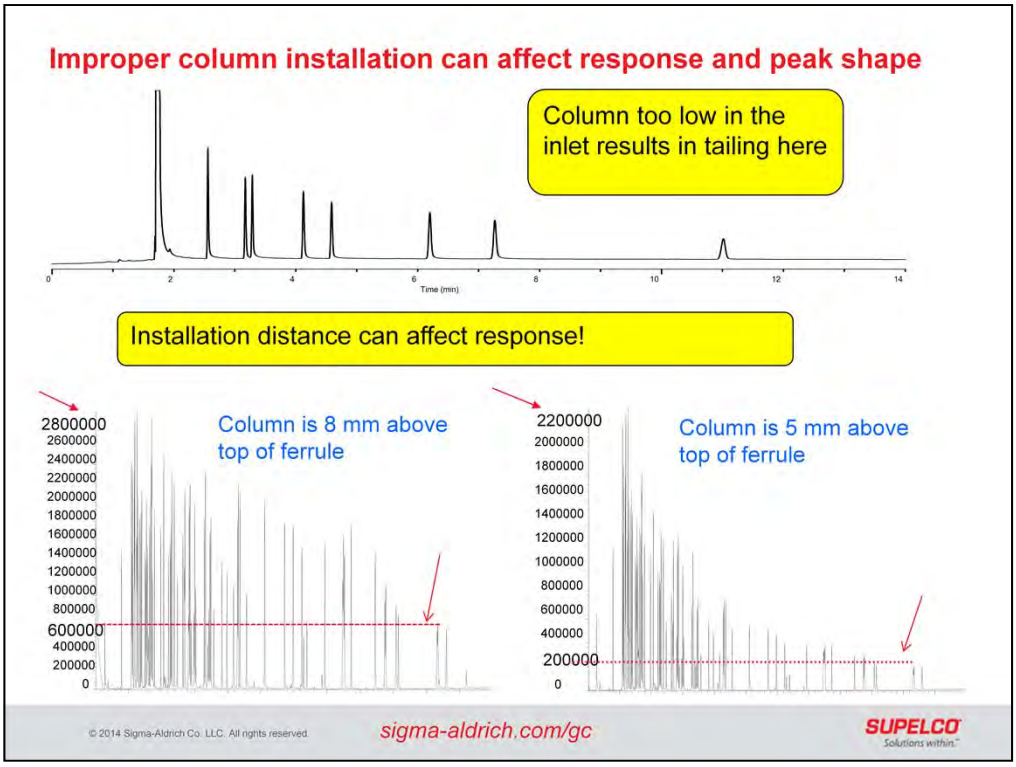


- Columns should be preconditioned before installing for the first time. Follow the instructions included with the column for starters, and modify for your specific application if necessary. If columns are being reinstalled after sitting idle, they should be run through some sort of preconditioning prior to reuse. If you can avoid it, do NOT precondition columns into an MSD or an ECD. They could contaminate these detectors.
- XXX…. And , the longer the column, the longer it will take to purge.
- XXX…Polar phases are especially susceptible to damage if this not done properly
- XXX…Do not use soap-based solutions to check for leaks. Use an electronic leak detector.
- XXX
- And finally, XXX



Correct column installation is also important to peak shape and response. Make sure you are installing your columns according to the instructions furnished by the GC manufacturer. For example, in this particular GC, the column is supposed to be 3-6 mm above the top of the ferrule after installation.

What happens if this is not done correctly? [NEXT SLIDE]



Installing the column too low in the inlet can lead to peak tailing, as we see here (top cgram).

In these chromatograms (bottom), we see the difference in response from having the column at different heights above the top of the ferrule. We get better response for the heavier compounds with a higher position.

Column maintenance

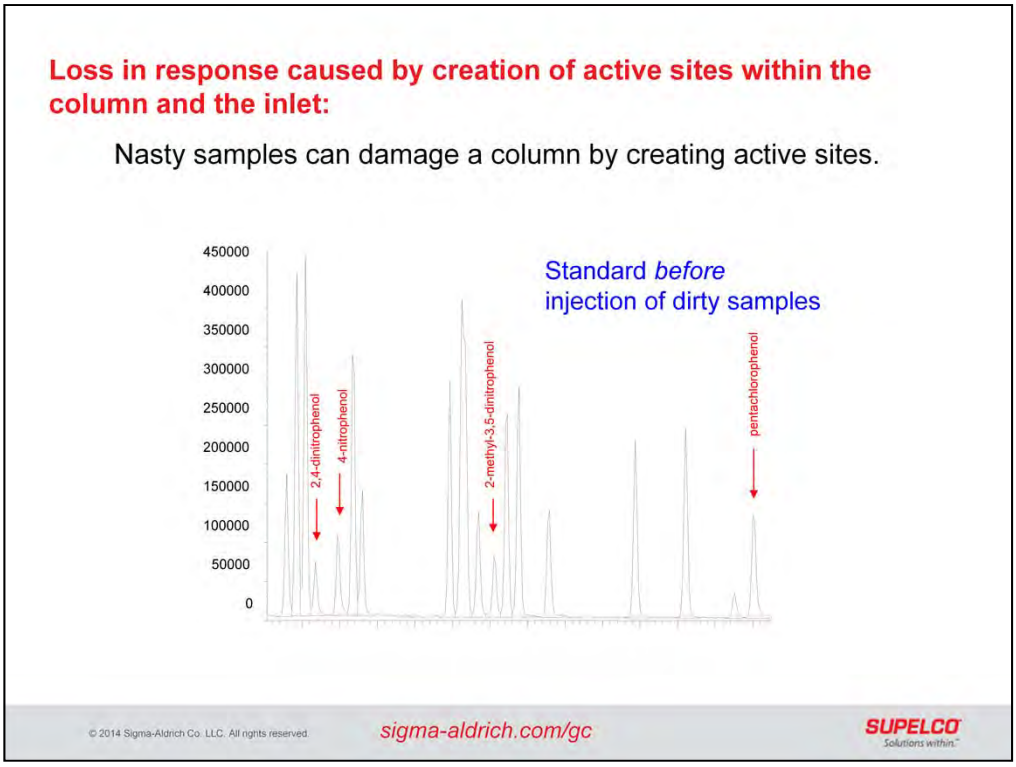
- GC columns can be clipped
- Done to restore analyte response when contamination is suspected
- Remove 2-6” of column (5 – 15 cm)
- In extreme cases of contamination, remove 1 or more column loops
- Install with a new ferrule

Sapphire fused silica capillary column cutter

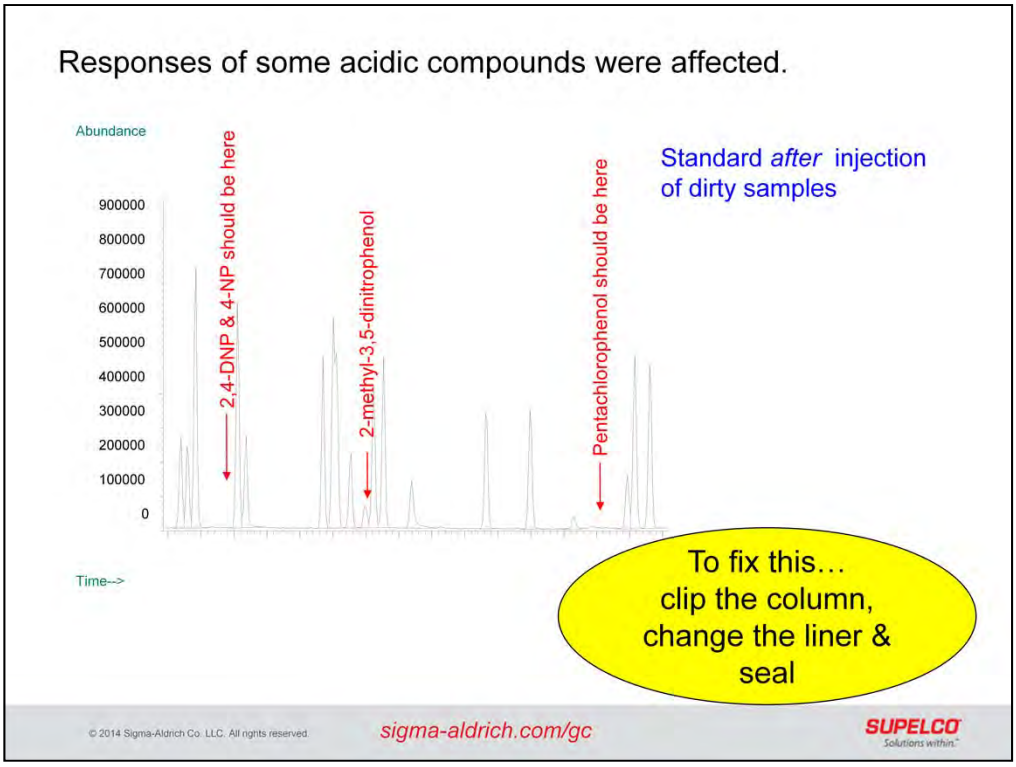
sigma-aldrich.com/gc
SUPELCO Solutions within.

Clipping a GC column is done on the premise that contamination has built up on the inlet side of the column that is causing adsorption. By removing this portion of the column, response is improved. Usually, 2-6” or 5-15 cm is removed. You can remove 1 or more loops if extreme contamination is suspected. Keep in mind that removing loops can affect the resolution of close eluting pairs, especially for shorter columns.

Shown here is a cutter I use for clipping columns. It is a sapphire blade cutter. Many people use ceramic wafers to do this. That works too, but these dull much faster. If you have to “saw” to cut the column, replace your cutter.



Let's take a look at an example of how active sites in the column can cause response problems. Nasty samples can quickly damage any column. This is a chromatogram of a semivolatile standard before injecting a particularly nasty sample. If we focus on these phenolic compounds, which are sensitive to active sites… [next slide]

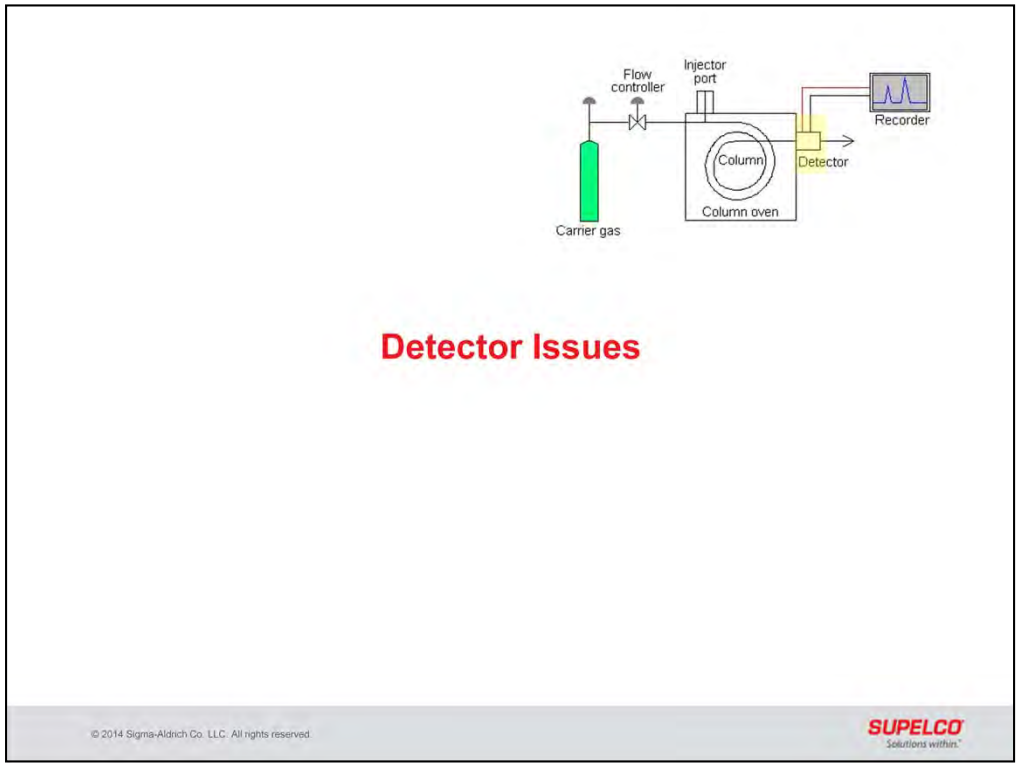


This is the same column from the last slide after making approximately three injections from a sample with a nasty matrix.

Notice the loss in response of the phenolic compounds we were focusing on in the last slide:

- In the case of 2,4-dinitrophenol and 4-nitrophenol, no response was seen.
- The response of 2-methyl-3,5-dinitrophenol is reduced significantly
- Pentachlorophenol is barely visible.

- The first things to try and fix this, would be to clip the column and change the liner & seal.



Moving on to detector related issues; GC probably has the widest array of detectors available of any chromatographic technique. It would be impossible to talk about all these detector types in our time today, so I will focus on some more general problems.

GC Detector Maintenance

Flame Ionization Detector

- Noises spikes often indicate a dirty detector
- Dismantle to clean
  - Fine grit sandpaper, Small wire brushes
- Replace jets as needed (noise, will not light)

Electron Capture Detector

- Bake out – follow manufacturer's procedure
- Return to manufacturer for replacement or rebuilding if necessary

MSD – when to clean

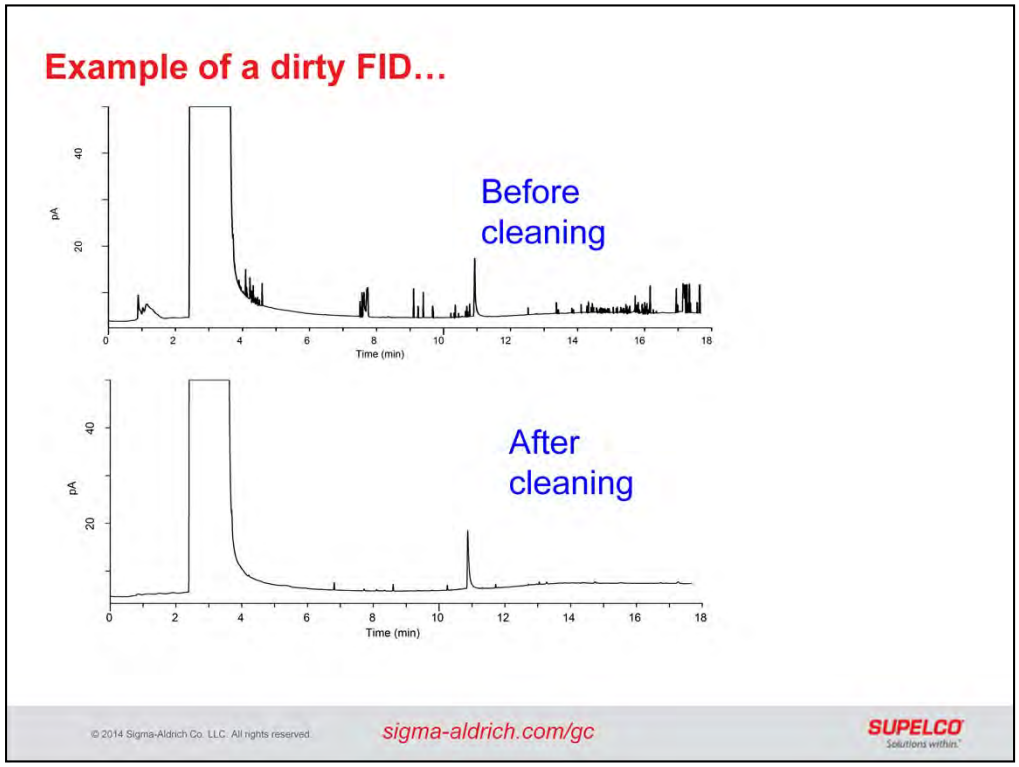
- Poor reproducibility
- Will not tune (extreme case)
- Loss of response; evident in sample runs and tune file
- Indications when tuning
  - Loss in response; especially for heavier mass (m/z=502)
  - Increase in EM voltage
  - Increase in repeller and/or ion focus voltages
  - High number of peaks in tune
- Clean per manufacturer's instructions

sigma-aldrich.com/gc

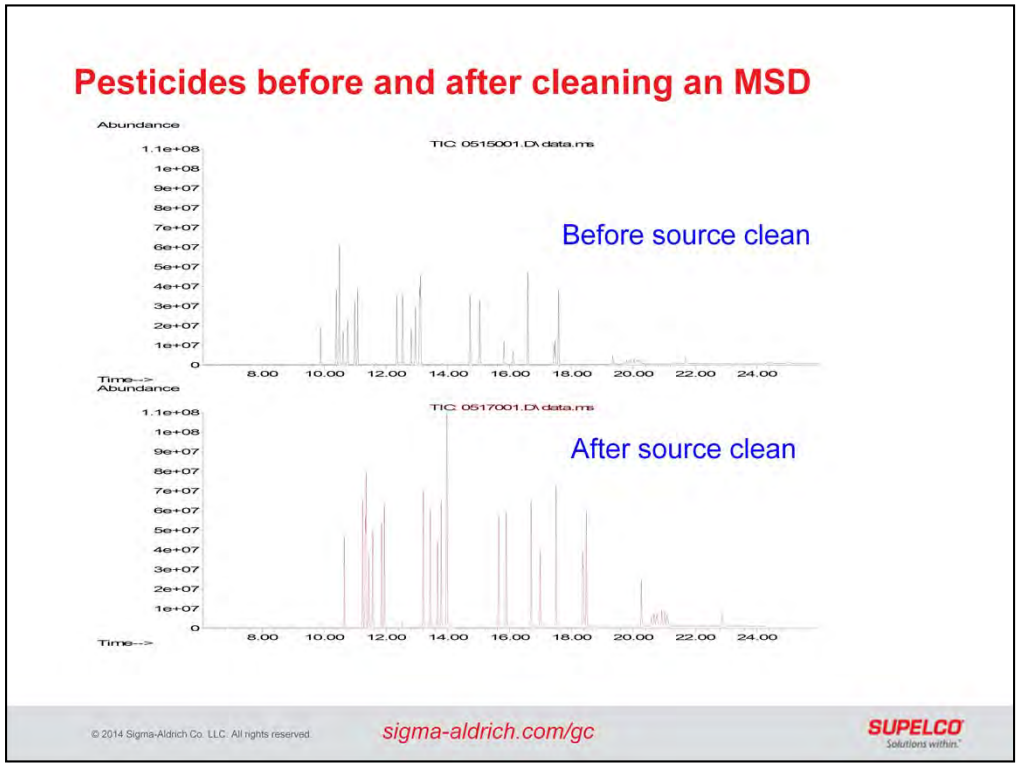
SUPELCO Solutions within.

The three most commonly used GC detectors are flame ionization, known as FID, electron capture, ECD, and mass spectral or MSD. All of these need to be maintained by use of clean carrier and makeup gases, and cleaned from time to time.

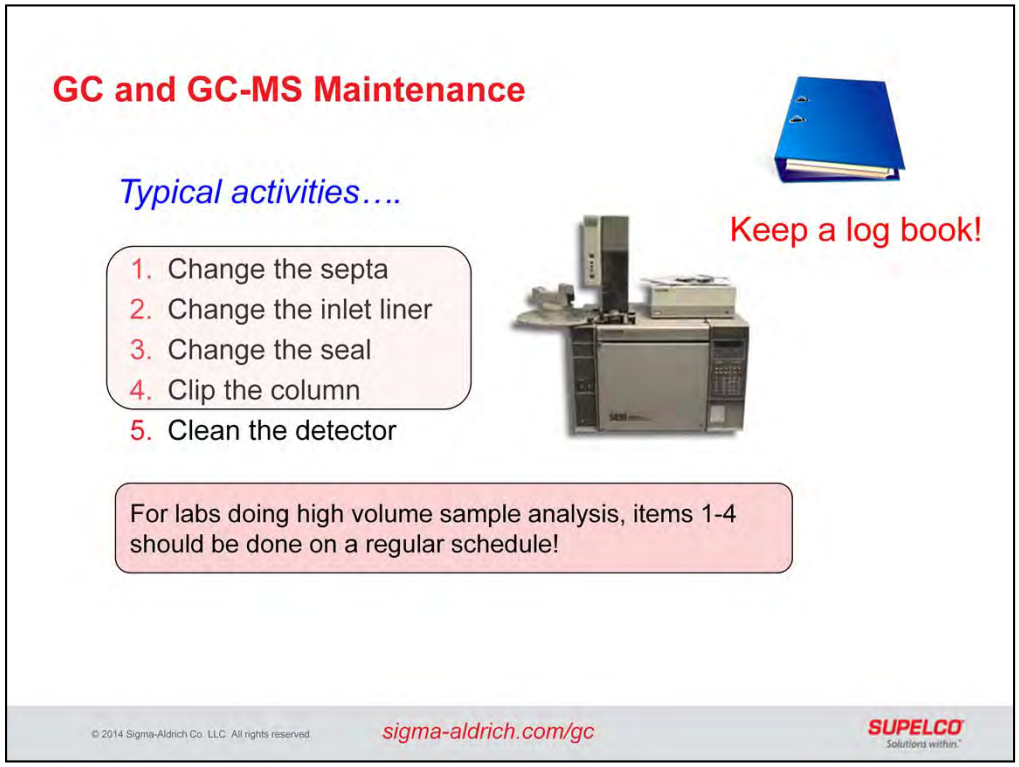
GO THROUGH LIST ON SLIDE



This is an example of what you will often see when an FID is dirty. Noise spikes appear in chromatograms. These spikes looks very different than peaks, in that it they are straight up and down, and not a classic gaussian shape.



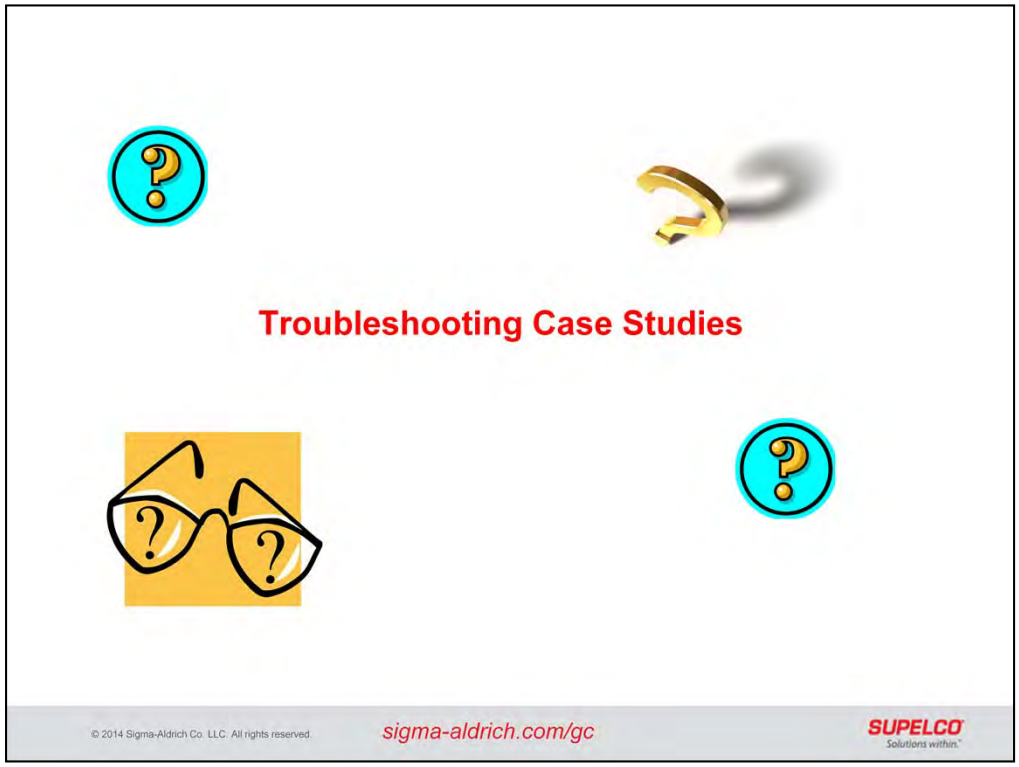
This shows the benefit of a clean MS source. The top chromatogram is a pesticide standard injected on a dirty MS. Just prior to running this standard, we had run several hundred injections of avocado extract. The chromatography is still pretty good, but response was lower than what it had been. The source was cleaned. As you can see in the bottom cgram, response has increased significantly for some pesticides.



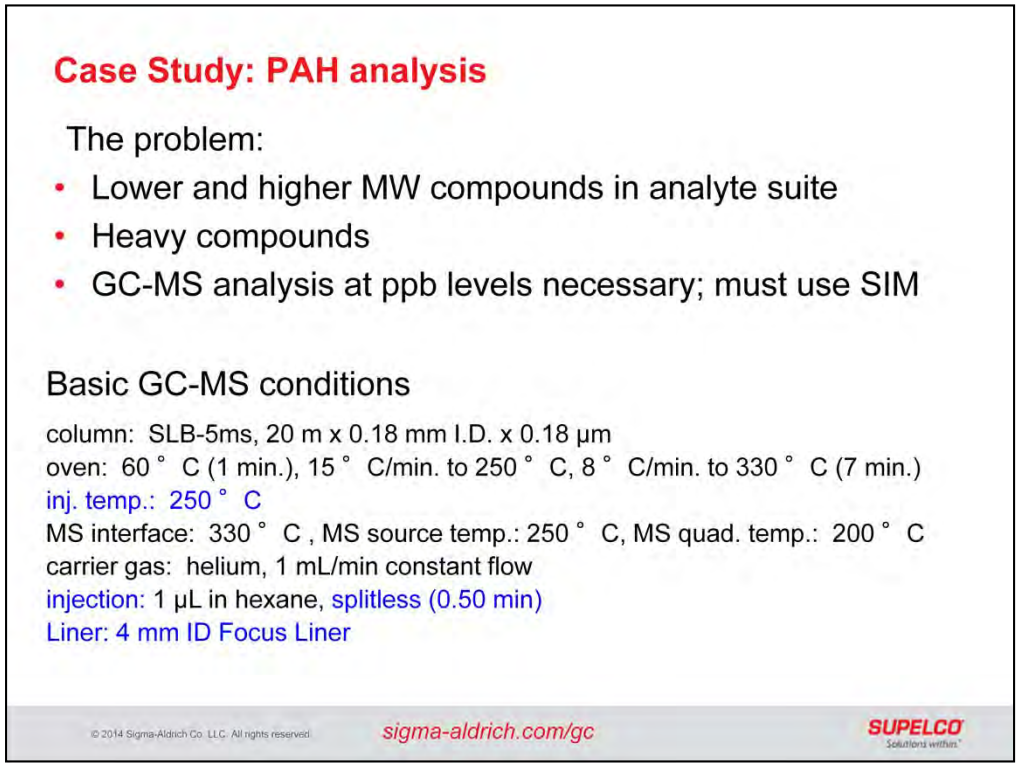
Finally…I cannot stress enough the importance of routine maintenance to prevent problems from even occurring. Maintenance activities are things such as changing the septa, liner, seal, and clipping the column. Detectors are usually cleaned on an “as needed” basis.

For labs doing high volume sample analysis [CLICK] these items should be done routinely.

Finally, keep a maintenance log. A record of what was done to correct a problem is invaluable if that same problem crops up again at a later time.

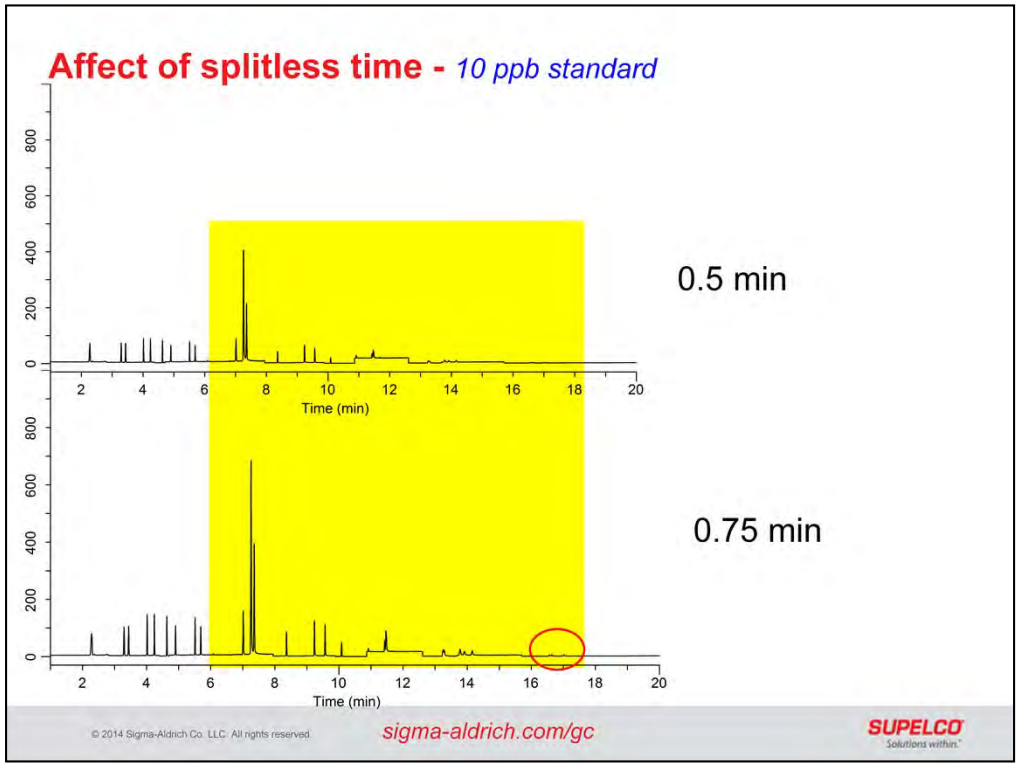


I would now like to share with you some case studies of things we have seen in our lab and how they were corrected.



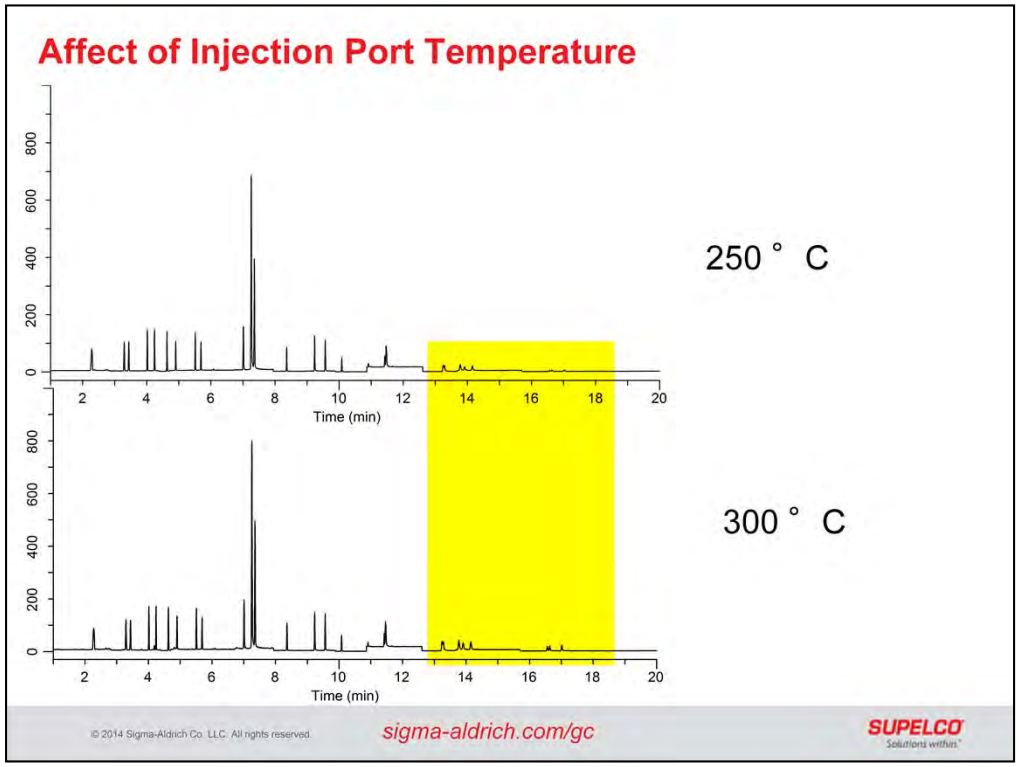
This application involved the analysis of PAHs at very low ppb levels out of food samples. We were expecting some background, so the use of MS for specificity was necessary. Before even attempting to do samples, it was necessary to optimize the GC-MS conditions.

Here are the basic conditions we started with. Pretty standard; inj. Temp. of 250C, splitless injection with a splitless time of 0.5 min., and a 4 mm ID liner.



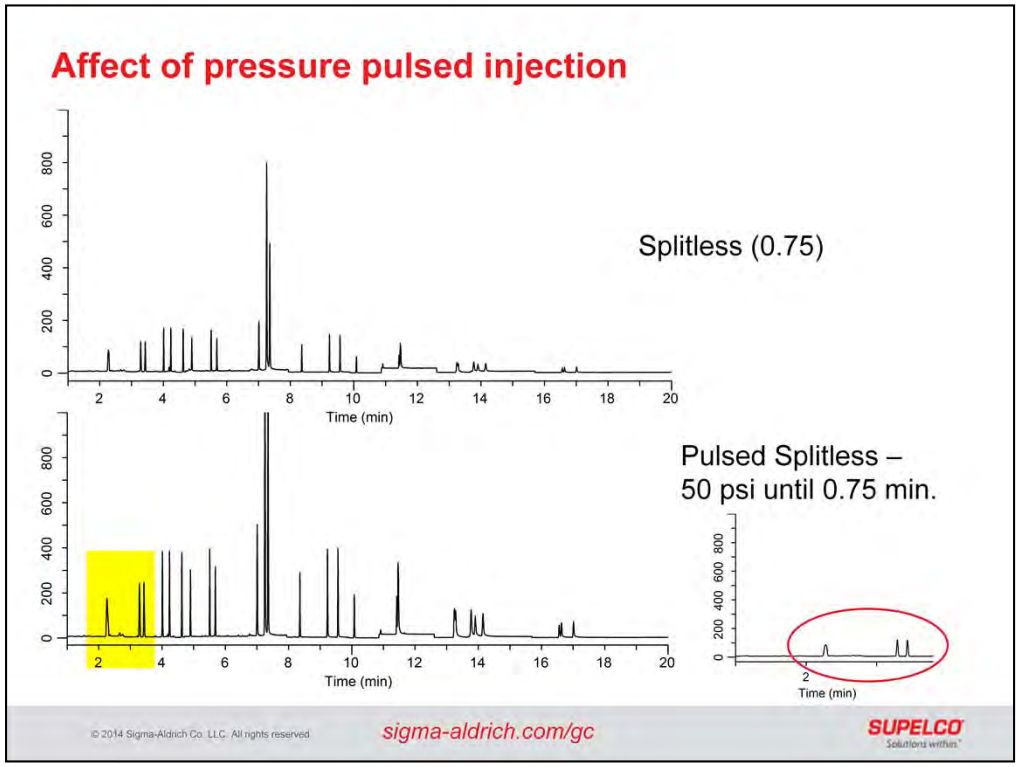
With the conditions from the last slide, response was pretty poor in a 10 ppb standard – the last few analytes were barely detectable.

[CLICK] Increasing the splitless time from 0.5 to 0.75 min increased response overall, and [CLICK] especially in the last half of the chromatogram. If we look at the last 3 analytes [CLICK] they are now visible. By increasing the splitless time, we are providing more time for the heavier compounds to reach the GC column.

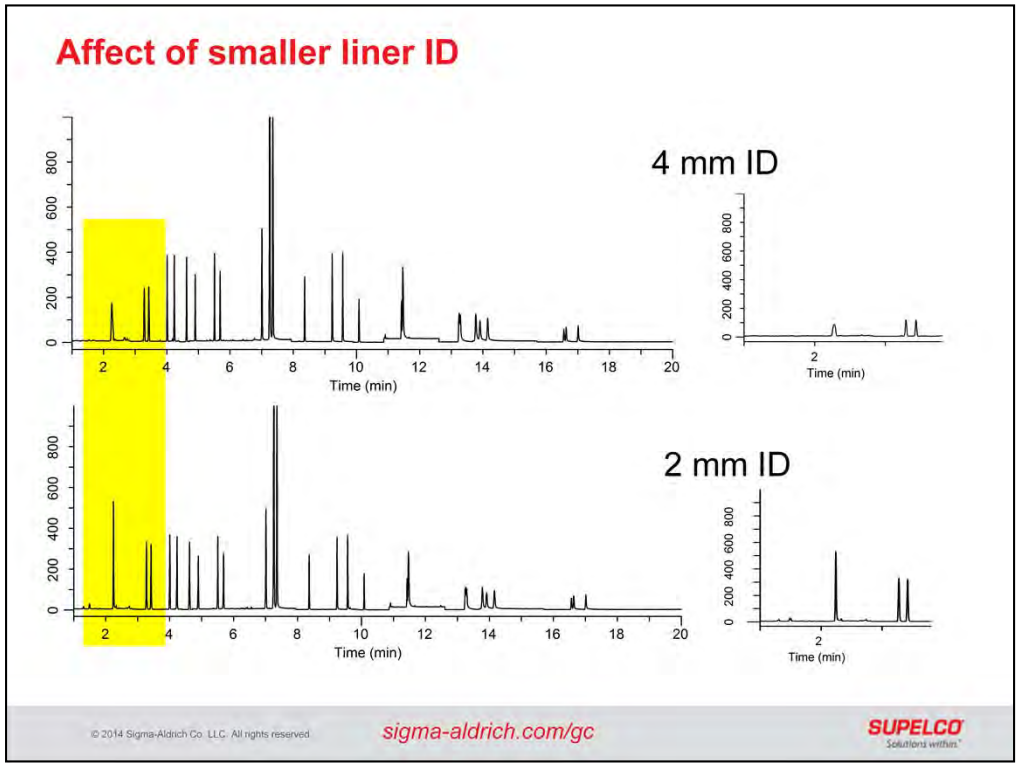


Next, we took a look at injection port temperature

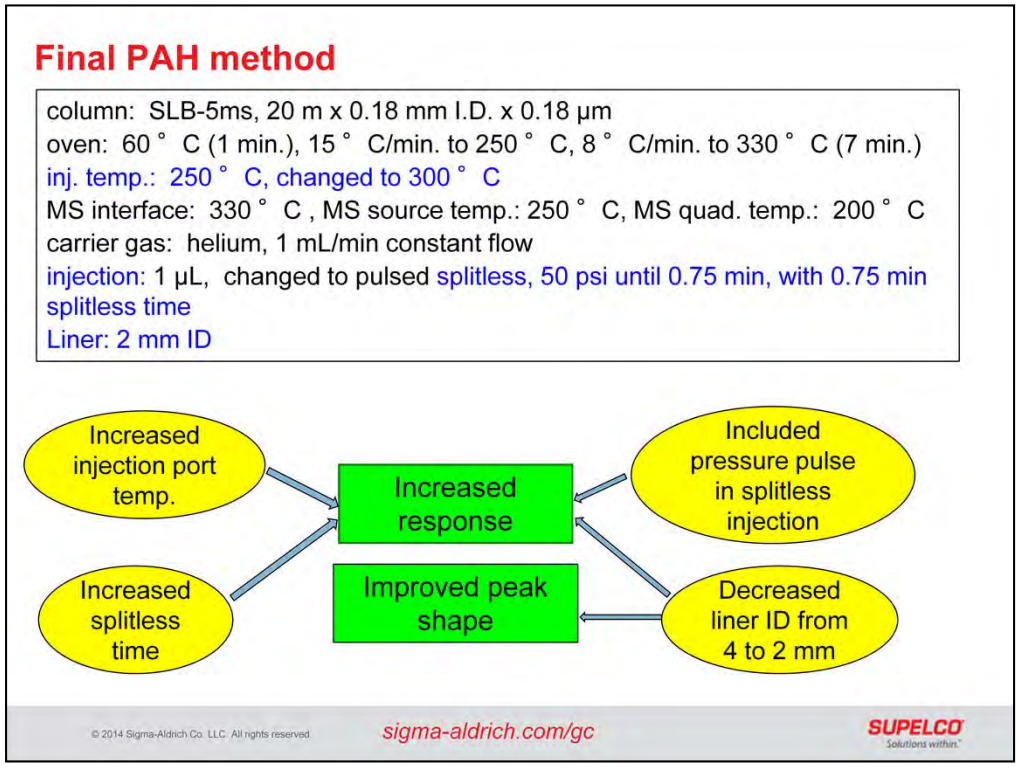
Since PAHs are heavy and some have extremely low volatility, it will require more energy to get good vaporization in the inlet, which is essential to getting more sample onto the column in a hot splitless injection. Increasing the injection port temperature from 250 to 300 ° C [CLICK] had a major impact on response for the heavies as we can see. Keep in mind though that a higher temp. will also increase the volume of the vapor cloud formed. This can affect focusing for the early eluters.



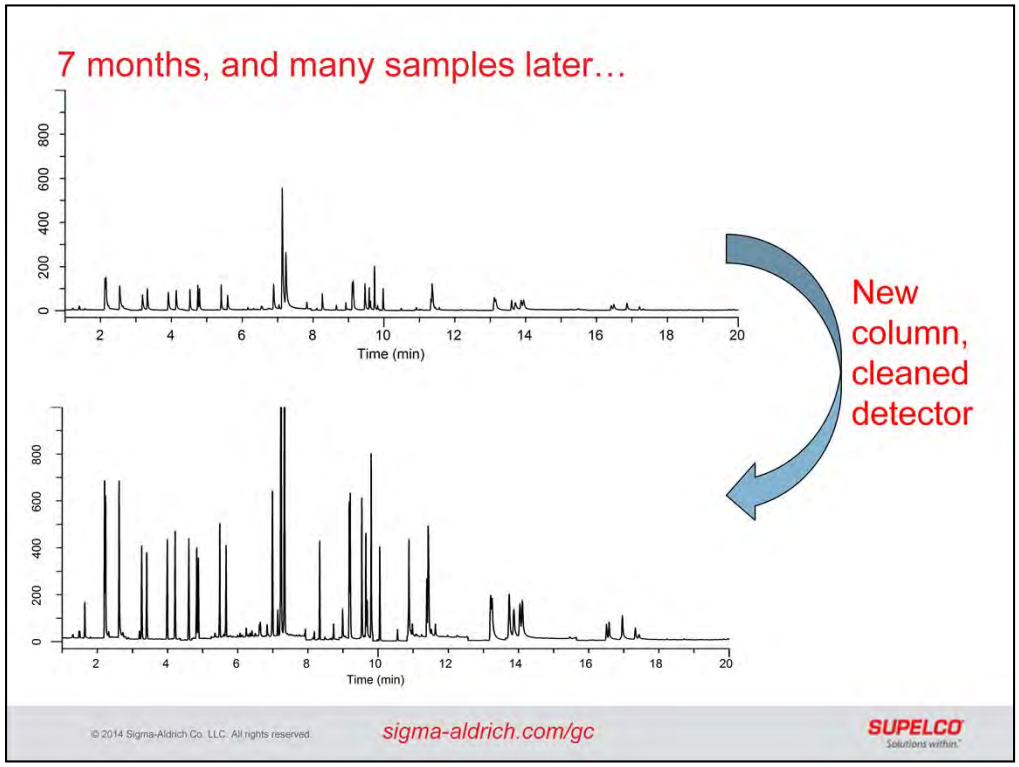
Next, to increase response we tried speeding up the focusing of analytes onto to the column…i.e. getting them through the inlet faster through use of a pressure pulse. Up to this point the injection has just been straight splitless. [CLICK] Using a pulse of 50 psi until the splitter opened really increased response, BUT [CLICK] at the same time had a negative effect on peak shape for the early eluters due to focusing issues. Naphthalene is now a broad peak. How do we fix this?



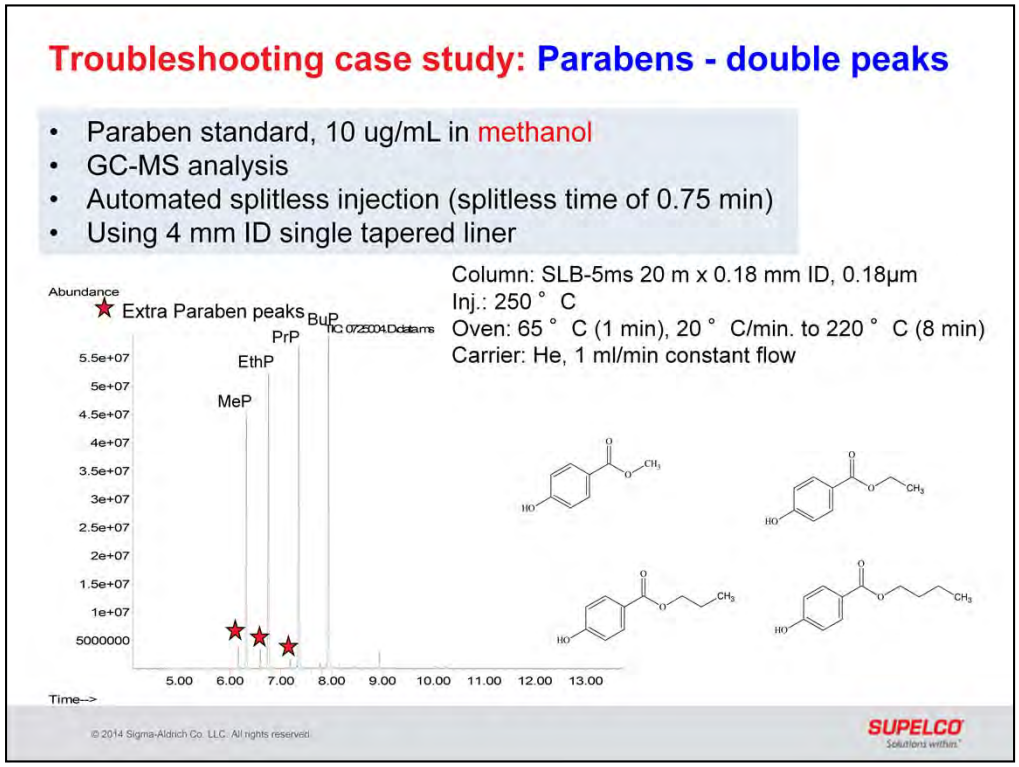
We had been using a 4 mm ID liner to this point…as it has sufficient volume to contain the hexane vapor cloud. Now that we are using a pressure pulsed injection, we can reduce liner ID and still contain the vapor cloud. A smaller liner ID will benefit peak shape for early eluters by making focusing more efficient. We can see here a great improvement in peak shape and response for naphthalene.



Incorporating all of these changes, this is the final GC method. So in summary, by increasing the splitless time [CLICK] and injection port temperature [CLICK], using a pressure pulse on injection [CLICK], and decreasing the liner ID [CLICK] [CLICK] we increased response for all the PAHs. The decrease in liner ID [CLICK] also had the benefit of improving peak shape for the early eluters.



How we have a good method…so after running many samples of different types on this system: olive oil, butter, fishoil….it almost looks like we are back where we started! Don't despair…remember routine maintenance! By replacing the column and cleaning the MS detector, response is back up to where it should be.



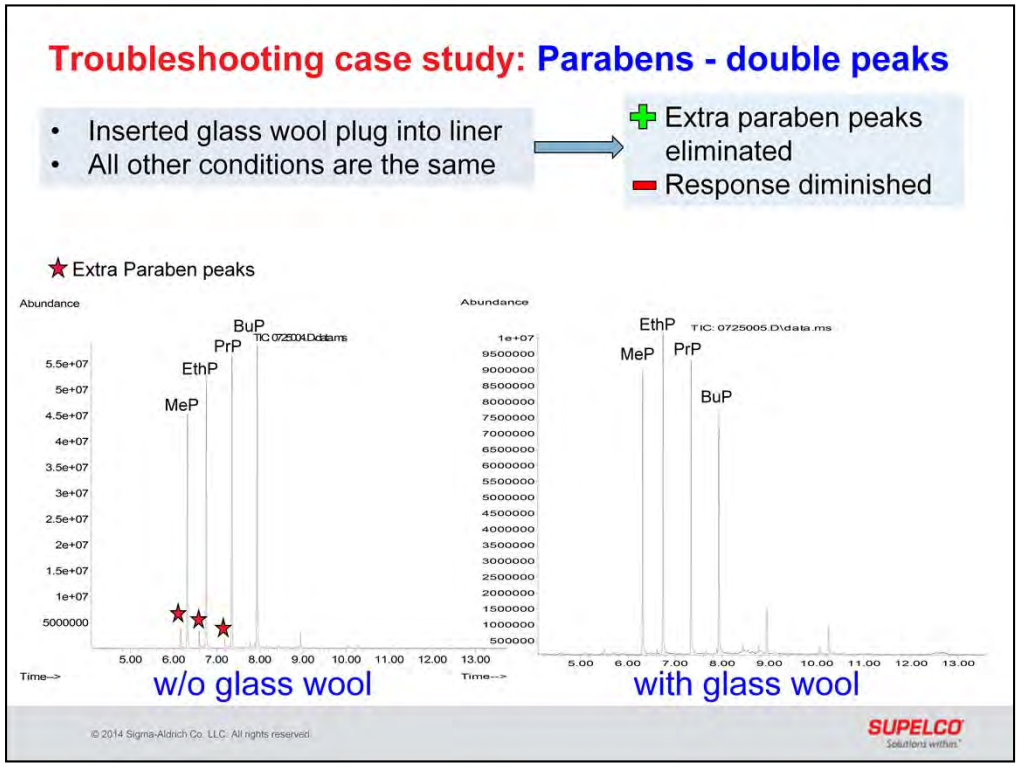
In this application, we are analyzing parabens, which are commonly used as preservatives in personal care and some food and pharmaceutical products. As you can see from the structures, they have some polarity. They are highly soluble in methanol, so this was chosen as the injection solvent. Using the GC conditions listed and a single tapered liner, we got two peaks each [CLICK] for methyl, ethyl, and propyl paraben. Since this is a full scan GC-MS analysis, the identities of these peaks could be confirmed as being the same as the parent peaks.

A couple of things could be happening here….

The first is the injection solvent of methanol. It is polar, and the column being used in nonpolar. So, it is not going to wet the column very well. This will effect focusing of the analytes on the column. Also, methanol is harder to flash vaporize than less polar solvents. This can result in vaporization and subsequent condensing of analytes on the column in stages, leading to two sets of chromatographic peaks. These peaks have a fairly short elution time, so there is not enough time for at least the first three parabens to reform into single bands while moving through the column.

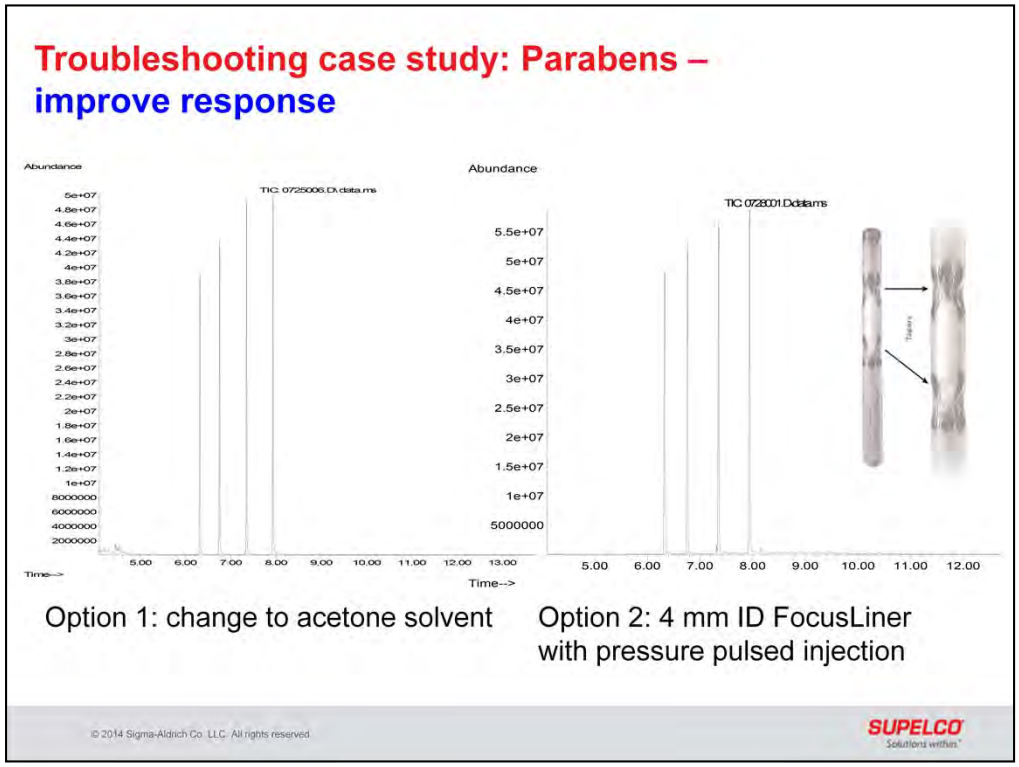
The second thing is the liner….it is unpacked, so the only surface area available for vaporization of the solvent is the glass itself. This is often OK, but in this case with what appears to be inefficient vaporization of the methanol, more surface area would be better.

So, what to do about it?



One thing is to increase the surface area within the liner – this will aid in vaporization. To do this [CLICK], a plug of glass wool can be used. Here I inserted deactivated glass wool into the single taper liner and injected the same standard again (all other conditions the same) [CLICK]. The extra paraben peaks are now gone, which is good. The other extra peaks visible in the chromatogram were identified as artifacts introduced by the wool, probably from my handling of it while packing the liner. However, notice that now the response is diminished. This probably due to adsorption onto the glass wool plug that was inserted into the liner.

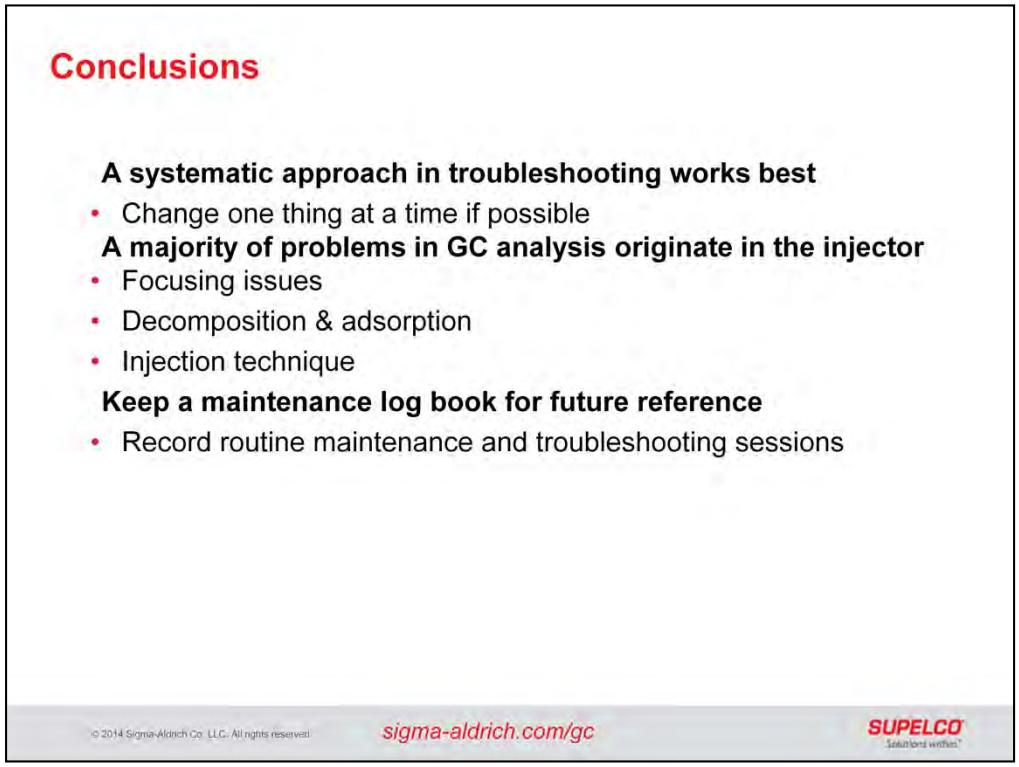
We fixed one problem, the extra peaks, but created another. How can we fix that?



Two options:

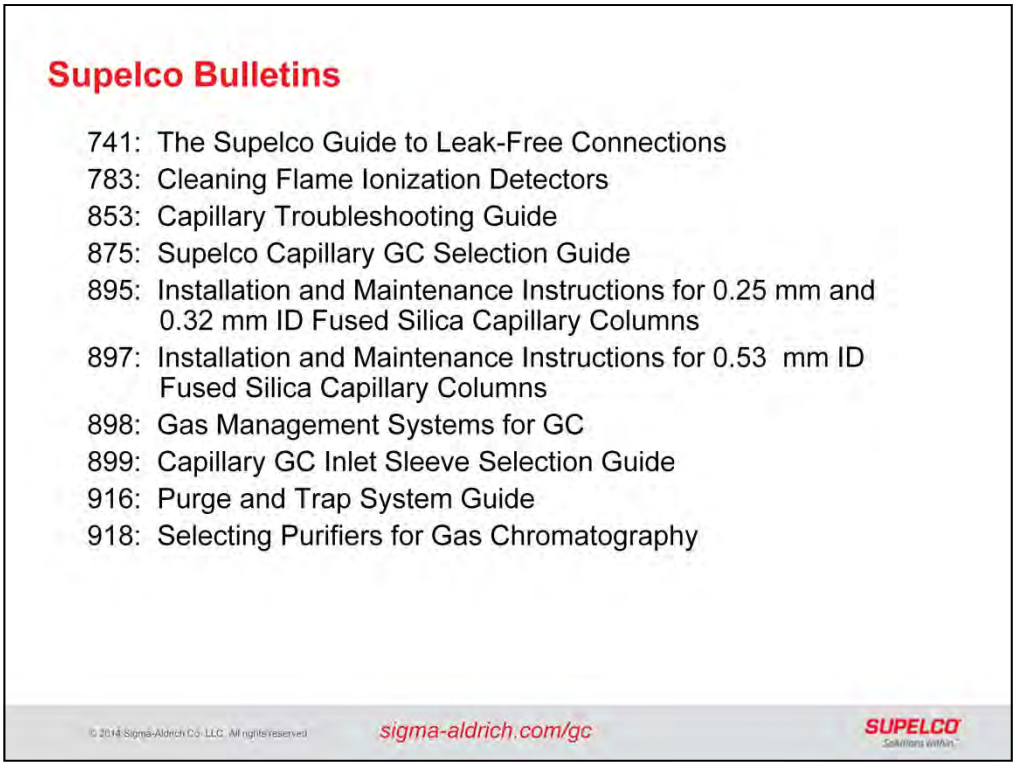
[CLICK] The first is to go back to the liner without the wool, and change to a less polar, more volatile solvent in which the parabens are still soluble. This is a 10 ppm paraben standard made in acetone. The response is similar to that from methanol, and the extra paraben peaks are not present. Acetone vaporizes much more easily than methanol, so the extra surface area provide by the wool is not necessary for quick and efficient vaporization.

The second option [CLICK] is to continue with methanol injection solvent and a wool containing liner, but to use a pressure pulsed injection to get the analytes through the inlet faster and decrease contact time with the wool. For this, I chose a 4 mm ID FocusLiner with quartz wool. The internal “puckers” in the glass of the FocusLiner hold the wool in place, so I do not have to be concerned that it will be dislodged by the pressure pulse.



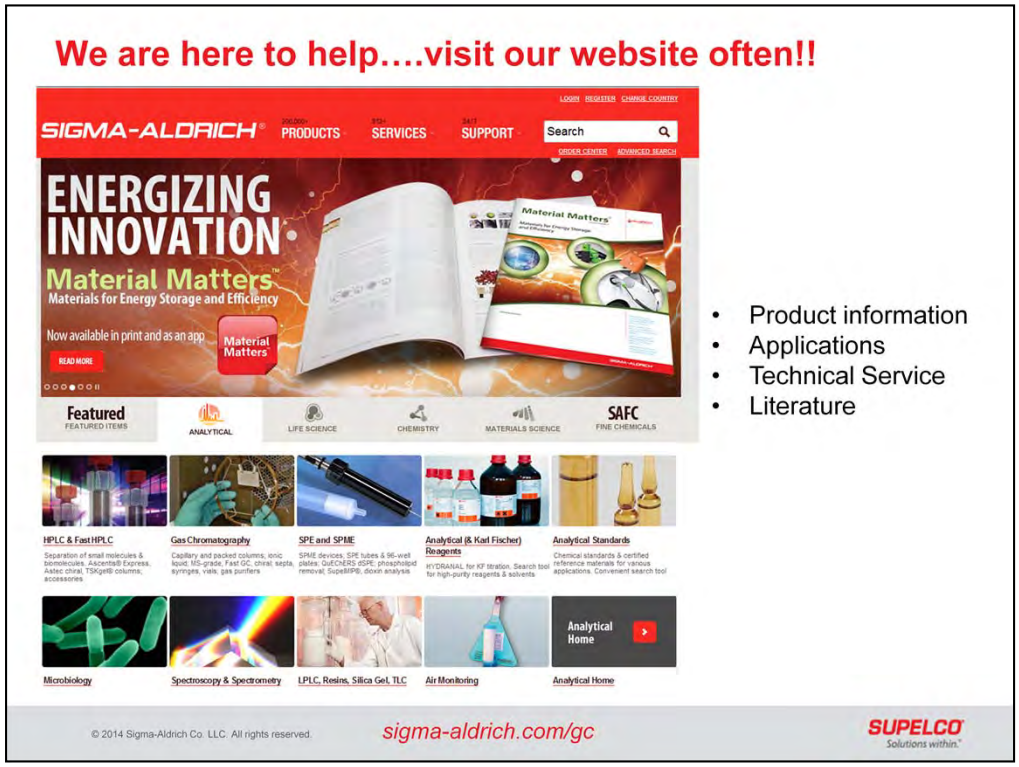
So in conclusion, when a problem occurs with your GC analysis,

- Consider using a systematic approach first. That is, rather than the “shot-gun” approach of changing multiple things at one time, try to isolate the problem by changing one thing at a time. That way, the experience will provide learnings on the specific cause for a problem.
- Remember that many GC problems orginate in the injector. There are a lot of processes taking place during an injection, so there is more source here for error than other places in the system.
- Finally, keep a maintenance log for future reference, for the benefit of yourself and your colleagues.



If you are interested in more details on troubleshooting and maintenance, this is some suggested reading materials available from Supelco which can help.

All of these bulletins are available on our website.



Speaking of our website, in addition to the literature listed on the last slide, you can find product and application information as well as get technical assistance, so be sure to visit it often.

Finally, thank you for your attention today. If you have any questions regarding this presentation, please contact me at the above email address.