SWAFER TECHNOLOGY COOKBOOK FOR GC APPLICATIONS





2 . Swafer™ Technology Cookbook

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Any comments about the documentation for this product should be addressed to:

PerkinElmer 710 Bridgeport Avenue Shelton, Connecticut 06484-4794 U.S.A.

Or emailed to: info@perkinelmer.com

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Introduction .5





Introduction .7



Introduction

The Swafer[™] system uses small circular metal disks into which micro-channels have been laser fabricated to provide flow switching and splitting capabilities on the PerkinElmer Clarus 500 and 600 GCs.

In this document we summarized, for quick reference, the technology, the chromatographic techniques for which it can be used and the various ways it can be configured to support these techniques.

Technology

There are currently two SWAFER devices available as shown in Table 1.

Device	Description
D-SWAFER	A wafer designed to switch the direction of capillary column gas flows based on the Deans' principle.
S-SWAFER	A wafer designed to split capillary column gas flows (rather than switching).

Table 1. The SWAFER Devices

Figure 1 shows the D-Swafer and S-Swafer devices next to each other. They are both the same size and use a common holder to mount them and the same fittings to connect to external tubes and columns. The Swafers are 20mm in diameter and about 3mm in depth.



Figure 1 Photograph of the D-Swafer and S-Swafer

Figure 2 shows how a Swafer is inserted into the holder with a threaded back plate to secure the Swafer in place during operation. Figure 3 shows how the assembly is mounted on a key-hole bracket in a Clarus 600 GC oven and how external tubes and columns are connected.



Figure 2 Swafer assembled in holder



Figure 3 Assembly mounted on key-hole bracket in Clarus 600 GC oven and external connections being made.

The internal channels are created by a laser fabrication technique and are 80μ m in height and can be made in a range of widths down to 50μ m. Figure 4 shows a schematic diagram of the internal channels of the D-Swafer and Figure 5 provides a corresponding diagram for the S-Swafer.



Figure 4 D-Swafer internal channels



Figure 5 S-Swafer internal channels

Functionality

The D-Swafer is designed as a flow switching device and the S-Swafer is intended for flow splitting. As such they are complementary to each other and the user can interchange the type of Swafer in the Clarus GC depending on whether there is a need to switch or split chromatographic sample streams.

The D-Swafer may be used to either switch a sample stream between two inputs or between two outputs. The S-Swafer can split a sample stream between up to 4 outputs or combine up to 4 input streams.

Figure 6 summarizes the functionality of the two Swafers in a simplistic form for clarification. Each input or output in all these scenarios can be fitted either a GC column or a length of fused silica restrictor tubing. The combinations are almost limitless.



Figure 6 Summary of main functions of D-Swafer and S-Swafer

If the sheer flexibility in configuring the inputs and outputs was not enough, another function available on all the Swafer designs and configurations is the ability to reverse the flow direction of the gas stream at their inlets. This enables the same backflushing and zone isolation techniques that have been previously established with the PerkinElmer PreVent technology family. In fact there is nothing that the PreVent system does that cannot be done with a Swafer system.

The Swafers will, however, do far more not only than the PreVent system but much more than any other flow switching technology available.

There are currently a total of 15 different hardware arrangements in which the two Swafers can be configured. These configurations enable a total of 18 different analytical techniques. More configurations and techniques are being discovered.

Table 2 provides a matrix of the various configurations and the techniques. Each of these is discussed in some detail in the remainder of this document. You will find active hyperlinks to aid navigation (in underlined blue text) – mouse-click on these will take you to the indicated link within the document.

In the configuration diagrams, there is a button labeled 'Run Calculator'. If the Swafer Utility Software has been installed and this document is being read from installation directory, these buttons will automatically load and run that particular calculator from the software.

Technique	<u>D1</u>	<u>D2</u>	<u>D3</u>	<u>D4</u>	<u>D5</u>	<u>D6</u>	<u>D7</u>	<u>D8</u>	<u>D9</u>	<u>S1</u>	<u>S2</u>	<u>S3</u>	<u>S4</u>	<u>S5</u>	<u>S6</u>
1.Solvent venting	•														
2.Column conditioning	•														
3.Pre-Column Venting						•									
4.Detector switching		•													
5.Column switching			•												
6.Column selection							•								
7.Inlet selection								•							
8.Column backflushing	•	•					•		•	•				•	
9.2-column backflushing				•	•										•
10.Column isolation	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
11.MS isolation	•	•					•			•				•	
12.Heartcutting				•	•										
13.Polarity tuning	•			•	•				•						•
14.Peak attenuation									•						
15.Carrier gas swapping						•							•		
16.Column splitting											•				
17.Detector splitting										•					
18.Column combining												•			

Table 2. Matrix of Swafer Configurations(Dx columns are for the D-Swafer and Sx are for the S-Swafer) and Analytical
Techniques.

• = Supported

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Techniques .17



1. Solvent Venting

Configurations

<u>D1</u>

Description

Not everything that passes through a GC column is good for the detector. Some detectors are damaged or contaminated by large amounts of solvent or other high concentration material that may find its way into a GC column.

Solvent venting is a technique which can divert column effluent away from the detector and out through a vent. This solvent venting process is controlled by valves operated through timed events. The user can decide when to start and end the venting by entering valve operations into a timed events table.

- When using a <u>MS</u>, it is standard practice to enter a solvent delay into the method to ensure that the ionizer filament remains off during the sample solvent elution. Without the solvent delay, the hot filament may become corroded as the relatively large amounts of solvent vapor pass over it. While the solvent delay is effective, complete elimination of the solvent from the chromatographic system prior to reaching the detector is a much better way of protecting the detector.
- Chlorinated solvents are often used in the preparation of sample extracts. In environmental applications, an electron capture detector is commonly used to monitor the presence of chlorinated pesticides in such extracts. The introduction of a chlorinated solvent into this detector will cause the signal to go off-scale for many hours. The solvent purging technique enables these solvents to be used with an <u>ECD</u>.

2. Column Conditioning

Configurations

<u>D1</u>

Description

This is essentially the same technique as the Solvent Venting described in <u>Section 1</u>. When a column is conditioned or reconditioned, silicones and other materials become vaporized and will pass into the detector where they may condense or react causing silica depots inside the detector. The solvent venting technique directs the column effluent containing this bleed material away from the detector.

- Any detector would benefit from this but this would be especially useful with detectors like <u>MS</u> and <u>ECD</u> where bleed deposits would contaminate and poison a detector significantly impairing performance.
- Because the conditioning mode is initiated by timed events, a conditioning run; may be used as part of an analytical sequence to bake out and re-condition a column after a series of particularly dirty samples.

3. Pre-Column Venting

Configurations

<u>D6</u>

Description

Not everything that passes through a GC injector is good for the column or the detector. Large amounts of solvent or other volatile material may overload the column and degrade the chromatography. Further, some detectors are damaged or contaminated by large amounts of solvent.

Pre-column venting is a technique which can divert solvent away from the column and out through a vent. This solvent venting process is controlled by valves operated through timed events. The user can decide when to start and end the venting by entering valve operations into a timed events table.

This technique is very similar to the large volume injection (\underline{LVI}) technique developed for the <u>PSS</u> injector. In that instance, the ability to rapidly change the injector liner temperature enables the solvent to be evaporated and purged out through the split vent at a low temperature leaving the less volatile analytes behind. The injector can then be programmed to a higher temperature to vaporize the analytes which are then transferred to the column with the split vent shut.

In this Swafer technique, instead of using a <u>PSS</u>, the <u>LVI</u> sample is introduced into a retention gap typically made from a few meters of 0.53mm i.d. deactivated fused silica tubing. Once the solvent is vented by the Swafer, the outlet is switched to the column to allow the analytes to enter the column as the temperature program proceeds.

The main disadvantage of this technique, unlike the Solvent Venting technique described earlier, is that it is only suitable for those samples where the solvent is much more volatile than the analytes. It does protect the column from the solvent and it can be used with any injector.

Applications

• Probably the main utility for this technique would be to support <u>LVI</u> applications with cold on-column injection (<u>POC</u> or <u>PSS</u>). Applications have been published with long retention gaps that are capable of accepting over 100µL of solvent-based samples injected by cold on-column injection. The Swafer technology is effective at removing most of this solvent before it reached the GC column. The vapor from the solvent would otherwise take over an hour to elute before any chromatography can take place.

4. Detector Switching

<u>Configurations</u>

<u>D2</u>

Description

This technique allows you to choose which detector to direct the column effluent. The switch is made by simple timed event controls embedded in standard GC methods and so the detector can be switched in between runs or within a run itself.

It could be used for similar applications to the Detector Splitting described in Section 16. The big disadvantage of <u>splitting</u> is that not all the material exiting the column will reach the detector and so detection limits may be greatly affected.

- One key application for this technique would be when the sample contains a mixture of analytes of different chemistries and would need a choice of detectors available to provide the best performance. For instance, looking at chlorinated and organophosphorus pesticides in the same chromatogram with an <u>ECD</u> and <u>FPD</u> detector and switching the individual peaks to the appropriate detector.
- Another possibility is to run the same sample twice choosing a different detector for each run. This gives maximum sensitivity and the whole chromatogram is seen on each detector.

5. Column Switching

<u>Configurations</u>

<u>D3</u>

Description

In this technique you can choose which column and detector to chromatograph a sample coming from the same injector.

The only real limitation is that the columns must be flow balanced for the switching to work.

- This can be applied to the same sample for confirmatory analyses perhaps with a <u>MS</u> detector or for completely different analyses using an external sampler such as a <u>headspace</u> <u>sampler</u> or <u>thermal desorption system</u>.
- This technique is applicable to any system where the same injector is used to run samples on different columns and the user wants to switch between these columns automatically.

6. Column Selection

<u>Configurations</u>

<u>D7</u>

Description

This technique differs from the Column Switching technique described in <u>Section 5</u> in that the user chooses which column will be connected to a single detector. In this case the two columns do not need to be geometrically matched and there is no need to flow balance them.

- This will be particularly useful in instances where two applications use the same detector which is either expensive or physically large or both. For instance, a <u>headspace sampler</u> can be used to perform an analysis on one column attached by the Swafer to a <u>MS</u> detector and then the system can be automatically switched to a liquid injector with an autosampler to perform a completely different analysis.
- Another possibility is to run the same sample on different columns but sharing the same detector. One could be a short column for screening samples. Samples showing a positive result could be re-injected onto a longer column for confirmatory analysis. The ability of the Clarus autosampler to automatically switch between injections is critical to this operation.

7. Inlet Selection

<u>Configurations</u>

<u>D8</u>

Description

This is a powerful configuration it is similar to the Column Selection technique given in Section 6. The main difference is that the two inlet systems share the same column and detector. Again it would be particularly useful with a MS detector. The <u>restrictors</u> or <u>transfer lines</u> between the two inlet systems and the column do not have to be matched.

- One of the key applications of this technique is to use it with an external sampling system such as a <u>headspace sampler</u> or <u>thermal desorption system</u> and use a liquid injector for the other inlet system. The liquid injector can now be used to inject liquid standards to calibrate or troubleshoot the system or determine recovery values for the sampling system.
- Another application would be where the same analytes are to be determined in different types of sample (solid, liquid or gaseous). This technique would allow the easy change between different types of inlet to the same chromatographic system.

8. Column Backflushing

Configurations

<u>D1, D2, D7, D9, S1, S5</u>

Description

Backflushing is perhaps the single most effective technique for reducing analysis time. Many analyses do not require chromatography of the whole sample – there are often target analytes or compounds groups to be determined and the rest of the sample can be comfortably ignored. If this unwanted sample material is less volatile than the analytes, the system will still need to elute it from the GC column before proceeding with the next analysis. This may required extended temperature programming to remove the last traces of the sample residue with occupies significant time and the high temperatures involved may have a detrimental effect on the column.

Backflushing is a highly efficient technique in removing unwanted heavier sample material from a GC column once the peaks of interest has eluted. It works through the simple expedient of reversing the direction of flow through the GC column. The heavier the compound the less far it will have traveled down the column and the less far it will then need to be driven backwards to remove it from the column.

Many analyses can be performed under isothermal conditions with backflushing whereas otherwise extended temperature programming was required.

- Any sample where the primary interest is in the volatile components in a wide volatility range sample is a prime candidate for backflushing. Examples include samples normally analyzed by a <u>headspace sampler</u> or <u>thermal desorption system</u>.
- Looking a fuel contamination in heating or lubrication oil is an application that would greatly benefit from backflushing.
- The determination of solvents in food or natural materials is another application.

9. 2-Column Backflushing

Configurations

<u>D4, D5, S6</u>

Description

This is very similar to the single-column backflushing technique described in <u>Section 8</u>, the main difference being that there are now two columns involved. This has two main benefits. The first being that the backflush process now occurs *during* the chromatography on the second column. For instance if the column used in single column backflushing was divided in half and each piece was used for the two columns in the 2-column backflushing system, exactly the same chromatography would be obtained and in the same time but the backflushing would occur during the analysis. With single column backflushing, the backflushing process must be performed *after* the analysis has finished.

The second benefit of 2-column backflushing is that the two columns can have dissimilar stationary phases enabling some possibilities for multidimensional separations.

The main downside of the 2-column backflushing system is the inability to monitor the elution of the sample from the first column. Whereas as this was not supported by the ProTect system, configurations $\underline{D4}$ and $\underline{S6}$ have provision for a mid-point detector to monitor chromatographic activity at the mid-point.

Applications

• Any analysis that benefits from single column backflushing will be further improved with 2column backflushing.

10. Column Isolation

Configurations

<u>D1, D2, D3, D4, D5, D6, D7, D8, D9, S1, S2, S3, S4, S5, S6</u> (i.e. All configurations)

Description

Column isolation is possible when backflushing a column as described in <u>Sections 8</u> and <u>9</u> or a length of fused silica restrictor. While the column or tubing is being <u>backflushed</u>, the Swafer and the column downstream of it become effectively isolated from the injector. This means that actions can take place at the injector that will not have any effect on what is connected to it. This enables injector servicing and injection techniques like <u>ELVI</u>.

With column downstream of the Swafer in the configurations <u>D3</u>, <u>D6</u>, <u>D8</u>, <u>S2</u> and <u>S4</u> it is even possible to run chromatography while the injector is being disturbed. Systems configured with the column upstream of the Swafer (<u>D1</u>, <u>D2</u>, <u>D4</u>, <u>D5</u>, <u>D7</u>, <u>D9</u>, <u>S1</u>, <u>S3</u>, <u>S5</u> and <u>S6</u>) will also allow injector maintenance and enhanced large volume injection while the column is being <u>backflushed</u>.

- An injector may be serviced while the system is in column isolation mode. Normally disruptive procedures like septum replacement and liner replacement are possible without the need to cool columns and detectors.
- The other key application for column isolation mode is for enhanced large volume injection (<u>ELVI</u>). Originally developed for the <u>PreVent</u> system with a <u>PSS</u> injector, exactly the same technique can be used on the Swafer system. With the <u>PSS</u> liner set to a low temperature, a large volume sample (e.g. 50µL) is injected with the split vent open. The solvent evaporates and is vented out through the injector splitter. While this solvent purge is occurring, the Column Isolation mode prevents any solvent vapor from entering the chromatographic system. Once the solvent has been removed, the split vent is shut and the analytes remaining in the cold liner are heated, vaporized and transferred to the GC column via the Swafer device, for analysis. This technique is particularly suited for large volume injections of sample containing solvents that would otherwise grossly overload the detector for instance the determination of chlorinated pesticides in a chlorinated solvent on an <u>ECD</u>.

11. MS Isolation

Configurations

<u>D1, D2, D7, S1, S3, S5</u>

Description

This technique is used in <u>MS</u> installations to enable column exchange while the detector is still active. When a column is disconnected, the mid-point <u>PPC</u> gas supply will vent out through the open Swafer port. This venting gas will serve to keep ambient air out of the Swafer and out of the restrictor going to the <u>MS</u>. Although this technique may represent some carrier gas wastage, if the new column is immediately installed, this will be kept to a minimum.

- Any system with a <u>MS</u> detector will benefit from this technique.
- A further benefit would be that if the GC column were to break, the MS Isolation technique will ensure that no air is drawn into the <u>MS</u> detector through the broken column.

12. Heartcutting

Configurations

<u>D4, D5</u>

Description

The heartcutting technique perhaps the ultimate in GC separating power and can often succeed when other GC options are failing. The chance of separating a peak of interest from all the others in a chromatogram is often just a matter of statistics. The more peaks there are then the higher the chance of co-elution with another peak. Changing the column stationary phase only means that now different peaks may now co-elute. Increasing the length or decreasing the diameter of the column improves matters in some instances but success is still a matter of chance or the very judicious selection of the column and conditions.

With heartcutting, the sample is separated on a single column and a narrow band of vapor eluting around the peak of interest is transferred (or 'heartcut') to a second column with a different stationary phase. Thus only a very small fraction of the original sample injected is chromatographed on the second column and so the statistical probability of separating a peak of interest is greatly enhanced.

This technique is really only suitable for the separation of a limited number of peaks but such separations may be obtained with hugely complex or concentrated samples. It really is the GC equivalent of finding 'a needle in a haystack'.

Because the heartcut systems also support 2-column backflushing as described in <u>Section 9</u>, considerable savings in analysis time are also possible.

Heartcut systems normally use two columns (as in D4) but three column systems are possible (as in D5). Although the three column configuration is potentially the more powerful, the availability of the midpoint detector to monitor peak elution from the first column makes the system far easier to setup and use.

- Separating a limited number of peaks from a highly complex sample matrix:
 - Petrochemical samples (DHA, MTBE, TAME)
 - Biodiesel (FAMES)
 - Environmental samples (pesticides in plant extracts)
 - Food (off-flavors, additives, contaminants)
 - Solvent impurities
- A further use would be, as applied in the Ozone Precursor System, to separate the injected sample between a strongly retentive and weakly retentive column to eliminate the need for oven cryogenic cooling in order to chromatograph a very wide volatility range sample.

13. Polarity Tuning

Configurations

<u>D4, D5, D9, S6</u>

Description

This technique is similar to the heartcutting technique described in <u>Section 12</u> but normally the whole sample flows from the first column into the second column of different polarity. Under a given set of conditions, a sample component will spend a finite time in the first column and finite time in the second. The column it spends most of the time in (assuming it's moving) will dictate the main character of the separation (e.g. polar or non-polar). By changing the midpoint pressure the relative time spent in each column will be directly affected and will change the chromatographic selectivity.

This technique will be most useful in chromatography where the separation between small clusters of peaks needs to be 'tweaked' to get an acceptable separation. The alternatives might be to try a series of different columns or analytical conditions.

- This tweaking or polarity tuning would be highly effective in many instances where a good GC separation has almost been achieved but is difficult to finally get the desired result:
 - The PerkinElmer Arnel DHA system uses manual polarity tuning to achieve critical separations

14. Peak Attenuation

Configurations

<u>D9</u>

Description

This technique allows the effluent from the primary column to enter a secondary column directly or to be mixed with carrier gas and be diluted before it enters the secondary column. This peak dilution effect can be applied by timed events to individual peaks.

The benefit of this technique is that it would effectively extend the dynamic range of the detection system by enabling low level concentration and high level concentration analytes to be monitored.

Applications

• The <u>FPD</u> is a good example of a detector that has a limited dynamic range (in the sulfur mode). In practice, this may be less than 200x. The peak attenuation feature could extend this by a further factor of up to ~50x

Another application could be to reduce the size of solvent and other peaks reaching a sensitive detector like a \underline{MS}

15. Carrier Gas Swapping

Configurations

<u>D3, D6, D8, S2, S4</u>

Description

For some applications it is desirable to use a different carrier gas within the GC column than that used in the inlet system. One example might be to use hydrogen for the chromatography on a headspace system that uses nitrogen for sampling for safety reasons.

Simply dropping the gas pressure at the inlet and/or raising the pressure at the Swafer after the injection has been made, will ensure that the only carrier gas entering the GC column will come from the mid-point supply.

Applications

• The technique of swapping the carrier gas would be particularly useful with external sampling systems where there are safety concerns in using hydrogen or with the expense of using helium.

16. Column Splitting

Configurations

<u>52</u>

Description

Column splitting is the simple technique of diverting fractions of the sample vapor eluting from the inlet to more than one column and associated detector. The carrier gas flow rate through each column and into each detector is defined by the length and diameter of each column and the applied common mid-point pressure. In most instances it is convenient to use columns of the same length and internal diameter so that optimum flow rates are easy to establish for each column. If a <u>MS</u> detector (at vacuum) is being used or columns of different geometry are required, then an in-line fused silica <u>restrictor</u> must be connected between one of the columns and either the Swafer or the detector.

Column splitting enables simultaneous chromatograms to be produced from the same sample but on columns of different selectivity is particularly suited to confirmatory analyses.

Applications

• One classic example is the blood alcohol application where two different columns are used to simultaneously chromatograph the same sample to produce different separations. Confidence in the identification of the peaks is significantly enhanced without the need for <u>MS</u> detection.

17. Detector Splitting

<u>Configurations</u>

<u>51</u>

Description

Detector splitting is the simple technique of diverting fractions of the column effluent to more than one detector. It is similar to column splitting except there is only one column and that is now connected between the injector and the Swafer. The flow rate to each detector is set by the length and diameter of a fused silica restrictor tube connecting the Swafer to that detector. The relative amount (or <u>split ratio</u>) of the sample reaching a detector is proportional to the relative flow rate down the restrictor. A mid-point carrier gas supply enables the range of <u>split ratios</u> to be greatly extended.

Detector splitting enables simultaneous chromatograms to be produced from the same column and injection and is particularly suited to complex samples containing analytes with functional groups that can be selectively monitored by different detectors.

- One classic example is the TO-14 air toxics monitoring method where an <u>FID</u> is used to monitor the hydrocarbon content in air samples and an <u>ECD</u> is used to monitor halide-containing compounds.
- Organophosphorus and organochlorine pesticides in environmental samples using an <u>FPD</u> or <u>NPD</u> and an <u>ECD</u>.
- Simultaneous detailed hydrocarbon analysis and sulfur compound analysis.

18. Column Combining

Configurations

<u>S3</u>

Description

Column combining is the simple technique of connecting multiple columns to the same detector. Its main benefit is just convenience. During operation bleed and any other contamination from all columns will continue to elute into the detector. In most instances the <u>Column Switching</u> technique would be a better choice.

Applications

• Any analysis where a convenient way of connecting columns to the same detector is sought and the analytes are at sufficiently high concentration to make issues with column bleed minimal.


Configurations .37



D1. Column Venting



Techniques Supported

- Solvent venting
- <u>Column conditioning</u>
- <u>Column backflushing</u>
- <u>Column isolation</u>
- MS isolation

Comments

The configuration uses the D-Swafer configured as a classic <u>Deans switch</u>. The two <u>restrictors</u> must be flow balanced to enable the mid-point gas supply to direct the column effluent between them. One good application for this configuration is to direct chlorinated solvents away from an <u>ECD</u>. Another example would be to direct solvents away from a <u>MS</u> detector.

D2. Detector Switching



Techniques supported

- <u>Detector switching</u>
- <u>Column backflushing</u>
- <u>Column isolation</u>
- MS isolation

Comments

This is essentially the same as the <u>D1</u> configuration except a second detector is fitted to the vent <u>restrictor</u>. Switching the state of the solenoid valve will change which detector the column effluent is directed to. This is very useful is situations when differing selective detectors are being used to monitor the same chromatography. For instance an <u>ECD</u> could monitor organochlorine pesticides and pesticides containing phosphorus could be directed to an <u>FPD</u>.

D3. Column Switching



Techniques supported

- <u>Column switching</u>
- Column isolation
- <u>Carrier gas swapping</u>

Comments

This configuration allows the injected sample to enter either of two columns enabling two separate analyses to share the same injector. This may prove most useful with external sampling systems like <u>headspace</u> or <u>thermal desorption</u> – perhaps use an <u>FID</u> with one column for presscreening and a <u>MS</u> on the other. The two columns must be flow balanced in order for the switch to work. For normal detectors this means that the columns should be of the same length and diameter. If one of the detectors is an <u>MS</u>, then an inline restrictor should be used between the column and <u>MS</u> in order to balance the vacuum. Configurations <u>D7</u> and <u>S2</u> may be better options because of there is less need to balance the two columns.

D4. Classic Heartcutting



Techniques supported

- <u>2-Column backflushing</u>
- Column isolation
- <u>Heartcutting</u>
- Polarity tuning

Comments

This is perhaps the most powerful of the D-Swafer configurations as it can significantly improve the separating power of the chromatographic system. The D-Swafer is used to 'cut' sections from the chromatography eluting from the first column and send these to the second column. Thus, the second column only sees a small fraction of the total sample and normally uses a different stationary phase to achieve a full separation of the cut sample fraction. This technique is very effective in situations where it is necessary to separate and quantify a small number of peaks in a very complex matrix - for instance, pesticides in food extracts or specific compounds in petrochemical samples.

D5. Three-Column Heartcutting



Techniques supported

- <u>2-Column backflushing</u>
- Column isolation
- Heartcutting
- Polarity tuning

Comments

This configuration opens up new opportunities for multidimensional separations. It's more difficult to use than configuration $\underline{D4}$ as peak elution from the first column cannot be monitored. The two outlet columns must be carefully matched to give the same gas flow rate through them. This configuration could be used to cut volatile compounds onto one column and less volatiles on to the other and then <u>backflush</u> unwanted heavy sample residue from the first column during the run (to save time).





Techniques supported

- Pre-column venting
- Column isolation
- Carrier gas swapping

Comments

This configuration may be considered as being similar to the <u>D1</u> configuration. The main difference is that it allows the solvent to be vented *before* it enters the GC column. Normally <u>D1</u> would give better results but there may be times when there's too much solvent (as in large volume injection) or a solvent that may degrade the column. This technique will only work for those samples where the solvent is much more volatile than the analytes. The <u>restrictor</u> and the column must be carefully balanced in order to allow the solvent vent switching to work. The pressures must be carefully adjusted to prevent solvent vapor from passing up into the midpoint <u>PPC</u> regulator. A good application for this technique would be to vent the solvent from a retention gap when a large volume cold on-column injection is being used.

D7. Column Selection



Techniques supported

- Column backflushing
- <u>Column isolation</u>
- MS isolation
- <u>Column selection</u>

Comments

Sample may be injected into either of two injectors - each with their own column. The D-Swafer is able to select which column has effluent sent to the detector. The pressure of the unused injector is reduced so that its column becomes backflushed. Any combination of injectors (including headspace samplers and thermal desorption systems) may be used. The main use of this configuration would be to allow two applications to be set up in a single GC that share a common (expensive) detector such as a <u>MS</u>. Switching between the applications is just a matter of changing the two injector pressures and switching the solenoid valve position. Changing detector mid-run is not supported.

D8. Inlet Selection



Techniques supported

- <u>Column isolation</u>
- Inlet selection
- <u>Carrier gas swapping</u>

Comments

This is similar to configuration <u>D7</u> the difference being that both injectors share the same column here. This configuration would be particularly useful in systems where a <u>headspace sampler</u> or <u>thermal desorption system</u> is present and it is necessary to be able to inject liquid samples on to the same column for calibration, troubleshooting or because of a different sample matrix.

D9. Peak Attenuation



Techniques supported

- Column backflushing
- <u>Column isolation</u>
- Polarity tuning
- Peak attenuation

Comments

This allows individual peaks to be selectively split and attenuated. During normal operation, the solenoid valve directs the switching gas to the restrictor side of the D-Swafer. This has the effect of directing all the effluent from the primary column into the secondary column. When the solenoid valve is switched, the switching gas mixes with the effluent from the primary column and some of this mixture will go into the secondary column but most will be vented out through the restrictor and thus splitting the sample peak. This technique will be useful to extend the effective dynamic range of the secondary column and the attached detector. Thus very large and very small peaks may be monitored in a single chromatogram.



Techniques supported

- Column backflushing
- <u>Column isolation</u>
- MS isolation
- Detector splitting

Comments

This simple configuration allows up to 4 detectors to be connected to the outlet of a single column. Fused silica restrictor tubing is used to make these connections and the way the column effluent is split can be set by choosing <u>restrictors</u> of appropriate geometry. The S-Swafer is designed so that any unused outlets can be capped with low dead-volume plugs so that performance is maintained when using only two detectors. A make-up gas supply enables higher flow rates to be used in the split lines so that high <u>split ratios</u> (>1000:1) can be used. The make-up gas supply also enables the other techniques listed to be used.

S2. Column Splitting



Techniques supported

- <u>Column splitting</u>
- Column isolation
- <u>Carrier gas swapping</u>

Comments

In this configuration, the sample vapor eluting from the injector is split between 2 and 4 columns. The relative split ratios will depend on the geometry of the columns. Each column will have a common inlet pressure set by the mid-point <u>PPC</u> regulator and so the columns will normally be chosen to have the same geometry. When columns of different geometry are required or a <u>MS</u> is being used, fused silica <u>restrictor</u> tubing may need to be connected in series with one or more of the columns.

This configuration is particularly useful where confirmatory independent separations of the same sample are required, for instance in blood alcohol analysis with a headspace sampler used as the 'injector' and the transfer line as the 'restrictor'.

S3. Column Combining



Techniques supported

- <u>Column combining</u>
- <u>Column isolation</u>
- MS isolation

Comments

In this configuration, the effluents from two or more columns are combined to provide a single output to the detector. All columns remain active throughout operation so bleed from all columns and contamination from injectors will continue to flow into the detector even though no sample has been injected into that column. Also the flow rate into the detector will be at least the sum of all the column flow rates – this may be too high for some detectors (e.g. <u>MS</u>). The <u>D7</u> configuration would normally be a much better alternative.

S4. PreVent Injector Mode



Techniques supported

- <u>Carrier gas swapping</u>
- <u>Column isolation</u>

Comments

This configuration is the same as $\underline{S2}$ but with only one column.

This S4 configuration is essentially the same as that for a <u>PreVent</u> system installed in an injector and so should be able to handle any of the applications developed using that system.

The other possibility is that the carrier gas at the injector and the S-Swafer could be different. With the injector pressure high, injected sample vapor will be swept into the column. If the midpoint pressure is raised or the injector pressure is dropped, the carrier gas flowing through the column will now come from the midpoint <u>PPC</u> supply. This technique would enable, say, nitrogen to be used on a headspace sampler for safety reasons and then use hydrogen in the GC column for fast chromatography.

S5. PreVent Detector Mode



Techniques supported

- <u>Column isolation</u>
- MS isolation
- <u>Column backflushing</u>

Comments

This configuration is identical to $\underline{S1}$ but with only one detector.

This S3 configuration is essentially the same as that for a <u>PreVent</u> system installed in an detector and so should be able to handle any of the applications developed using that system.

S6. ProTect Mode



Techniques supported

- <u>Column isolation</u>
- <u>2-column backflushing</u>
- Polarity tuning

Comments

This configuration is essentially the same as the <u>ProTect</u> option that is part of the <u>PreVent</u> family of products. It enables two columns of possibly different geometry and selectivity to be connected in series. Once the peaks of interest have passed from the first to the second column, the injector pressure can be dropped or the midpoint pressure is raised (or both) to backflush residual sample from the first column while the chromatography in the second column proceeds.

There are two large advantages over <u>ProTect</u> in that there is complete flexibility in the choice of the first column (in <u>ProTect</u>, a special miniature column was used) and that an optional detector can be connected to the S-Swafer to monitor peak elution from the first column.

In most instances, the $\underline{D4}$ configuration would provide a better platform for backflushing with a 2-column system, however, this S6 configuration would be easier to set up and use as there is not need to install a restrictor with critical dimensions for pressure balancing.

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Backflushing – A chromatographic technique in which the direction of carrier gas flow is reversed. This has the effect of eliminating heavy sample material residing near the column inlet without the need for extensive temperature programming and so saves time and reduces stress on the column and detector. It can also be used to isolate the injector from the column to enable **ELVI** and injector maintenance.

ECD (Electron Capture Detector) – A highly sensitive GC detector that uses a radioactive source to ionize the carrier gas and allow a standing current to be maintained. Electronegative molecules such as organo-halides passing into the detector from the column collect electrons and the reduction in the standing current produces the output signal.

Deans' Switch – Named after David Deans of ICI Chemicals, UK. This is a means of changing the direction of gas flow by changing the pressures across the flow path. This can be used as a basis for <u>heartcutting</u> and <u>backflushing</u> devices.

ELVI (Enhanced Large Volume Injection) – This is the same as <u>LVI</u> but with ability to backflush the column or restrictor connected to the injector, all the solvent may be eliminated from the column during the solvent purge step

FID (Flame Ionization Detector) – A GC detector in which organic compounds are burnt in a flame to produce ions which are collected by a polarizing voltage. This is a popular detector in GC that is sensitive to most organic compounds.

FPD (Flame Photometric Detector) – A GC detector that monitors light emissions of selected wavelengths from a flame into which the column effluent passes. It is usually used to monitor sulfur or phosphorus containing compounds.

Headspace Sampler – A sample introduction system that extracts and injects volatile components from heavier sample matrices.

Heartcutting – A process in which a switching device can direct the effluent from a column to one of two outlets. Normally one of these outlets is connected directly to a detector to enable peak elution from this column; the second outlet is normally connected to a second column of a

different selectivity. By transferring small time slices (heartcuts) of the effluent from the first column to the second, it becomes possible to get complete separation of specific analytes from other components in complex samples.

LVI (Large Volume Injection) – This is a technique in which a large volume (e.g. 50µL) of a liquid sample is injected and chromatographed to improve detection limits. The sample is injected into a cold <u>PSS</u> injector liner. The liner is held at this low temperature for a minute or two while carrier gas is flushed through the liner and out through the split vent. This has the effect of evaporating the solvent held within the liner and venting it out of the split vent (some of the solvent vapor will also enter the column at this time). At the end of the solvent purge, the split vent is closed and the temperature of the liner is raised to vaporize the sample residue and introduce it into the GC column for chromatography. Also see <u>ELVI</u> for details on how to further eliminate the solvent from the column.

MS (Mass Spectrometer) – A detector adapted for GC use that uses molecular fragmentation to isolate, identify and quantify amounts of specific compounds eluting from a GC column. A very powerful detector that is becoming increasing popular as it is able to establish or confirm peak identities.

MS Vent – A device from the <u>PreVent</u> family of products that passes carrier gas across the MS transfer line inlet while the column is disconnected to prevent ingress of air. It enables column exchange without the need to cool and vent the detector and so saves significant time. It is also called <u>MS Isolation</u>. This functionality is many supported by many of the Swafer configurations.

NPD (Nitrogen Phosphorus Detector) – A GC detector in which reactions in a plasma flame around a heated alkali bead induce ions selectively from nitrogen and phosphorus containing compounds that are collected by a polarizing voltage.

POC (Programmable On-Column) Injector – An injector similar to the PSS except that the column is introduced right into the cooled injector and its inlet is located close to the septum. A needle guide is used to insert a syringe needle directly into the internal bore of the column and make an injection. The injector is heated after the needle is withdrawn and chromatography commences. This injection technique, although not the easiest to use, produces the best results – the hot sample does not make contact with any surface except the inside of the column and so it is particularly useful for labile compounds and wide volatility range samples where mass discrimination needs to be minimized.

Polarity Tuning – A technique in which two columns of differing selectivity are connected in series. A mid-point pressure regulator is used to change the relative gas velocities in each column and the time each sample component spends in each column. A low mid-point pressure would decrease the time spent in the first column and increase the time spend in the second. A high mid-point pressure would reverse this effect. In this way, the effective selectivity of the combined column system can be manipulated and tuned to enable separation of critical peaks from their neighbors.

PPC (Programmable Pneumatic Controls) – pressure regulators and flow controllers that may be programmed electronically by GC methods and timed events (rather than knobs that must be manually rotated to make changes in conventional mechanical controllers). This capability is essential for operation of the Swafer system as programmed pressure changes are responsible for the system functionality.

PreVent – A modular system that connects directly to an injector or detector port to insert a short fused silica restrictor into that port. A midpoint carrier gas supply provides the ability to

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reverse the flow of gas through the column or restrictor connected to the injector. This enables techniques like **ELVI**, <u>column backflushing</u>, <u>column isolation</u> to be performed. All these functions are now supported by the Swafer platform.

ProTect – Part of the PreVent family of products. ProTect was essentially a short, narrow bore column (5m x 0.120mm) that was wound onto a 25mm mandrel assembly that replaced the <u>PreVent</u> injector restrictor. It enables <u>2-column backflushing</u> that is now also possible with the Swafer technology.

PSS (programmable Split/Splitless) Injector – A capillary liquid injection system in which the liner is normally cold during sample injection and then temperature programmed after the syringe needle has been withdrawn. Sample vaporization is controlled and analytes are swept from the liner as soon as they vaporize and so are not exposed to extremes in temperature. The technique minimizes analyte thermolysis and improves performance with respect to mass discrimination is wide boiling range samples.

Restrictor – In this context, it is a piece of narrow-bore fused silica tubing of prescribed length and internal diameter.

Splitting – The process of directing a sample stream between two or more outlets. The relative amount of sample passing to each of the outlets is controlled by the relative flow rate in each outlet. In this context, each outlet flow rate is controlled by a <u>restrictor</u>.

Split Ratio – The ratio of the amount of sample exiting from one splitter outlet to that of another.

Thermal Desorption System – A sampling device that vaporizes volatile components collected in tubes packed with adsorbents and injects them into a GC.

Transfer Line – A length of (normally) fused silica capillary tubing used to connect a column to an injection system or detector.



PerkinElmer Life and Analytical Sciences 710 Bridgeport Avenue Shelton, CT 06484-4794, U.S.A.

Internet: http://www.perkinelmer.com email: info@perkinelmer.com

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