Theory and Instrumentation of GC

Sample Introduction

Wherever you see this symbol, it is important to access the on-line course as there is interactive material that cannot be fully shown in this reference manual.
Aims and Objectives

Aims

- To outline the various injector categories for GC
- To describe and explain the components and working principle of split and splitless injectors for GC
- To state the problems associated with each of these injection types
- To explain how these problems are overcome practically and give examples of optimising inlet parameters
- Describing choices for inlet consumables
- Describe working principles, components and optimisation for a series of common GC inlets

Objectives

At the end of this Section you should be able to:

- Describe the working principles and problems associated with split and splitless injection in GC
- Explain how these problems are overcome in a practical setting and demonstrate a working knowledge of which conditions to optimise
- Highlight various options for septa and liners used with split/splitless inlets
- Identify the major components and explain the working principles of various other inlet types for packed and capillary GC
- Demonstrate a working knowledge of the important variables and optimisation routines for these inlets
Content

<table>
<thead>
<tr>
<th>Topic</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC Inlet Systems</td>
<td>3</td>
</tr>
<tr>
<td>General requirements for GC Inlets</td>
<td>3</td>
</tr>
<tr>
<td>Split / Splitless Inlet</td>
<td>4</td>
</tr>
<tr>
<td>Split Injection</td>
<td>7</td>
</tr>
<tr>
<td>Setting the Split Ratio</td>
<td>9</td>
</tr>
<tr>
<td>Sample Discrimination</td>
<td>10</td>
</tr>
<tr>
<td>Injection Volume</td>
<td>12</td>
</tr>
<tr>
<td>Split Injection Experiments</td>
<td>14</td>
</tr>
<tr>
<td>Splitless Injection</td>
<td>14</td>
</tr>
<tr>
<td>Optimising Splitless Injection</td>
<td>15</td>
</tr>
<tr>
<td>Purging the inlet</td>
<td>15</td>
</tr>
<tr>
<td>Analyte Focussing</td>
<td>17</td>
</tr>
<tr>
<td>Solvent Choice</td>
<td>19</td>
</tr>
<tr>
<td>Splitless Injection Experiments</td>
<td>21</td>
</tr>
<tr>
<td>Choosing an Inlet Temperature</td>
<td>21</td>
</tr>
<tr>
<td>Liners for Split/Splitless Injection</td>
<td>22</td>
</tr>
<tr>
<td>Liner top</td>
<td>23</td>
</tr>
<tr>
<td>Liner geometry</td>
<td>23</td>
</tr>
<tr>
<td>Liner packing</td>
<td>23</td>
</tr>
<tr>
<td>Liner bottom options</td>
<td>24</td>
</tr>
<tr>
<td>Liner I.D.</td>
<td>25</td>
</tr>
<tr>
<td>Septa for Split / Splitless injection</td>
<td>26</td>
</tr>
<tr>
<td>Septum Problems</td>
<td>27</td>
</tr>
<tr>
<td>Troubleshooting &amp; Maintenance</td>
<td>28</td>
</tr>
<tr>
<td>Cool-on-Column Inlet</td>
<td>28</td>
</tr>
<tr>
<td>Performance</td>
<td>29</td>
</tr>
<tr>
<td>Optimising Cool-on-Column Injection</td>
<td>30</td>
</tr>
<tr>
<td>Use of Retention Gaps for Cool-on-Column Injection</td>
<td>31</td>
</tr>
<tr>
<td>Programmed Temperature Vaporisation (PTV) Inlets</td>
<td>32</td>
</tr>
<tr>
<td>PTV Sample Flow Rate</td>
<td>34</td>
</tr>
<tr>
<td>PTV Liner Type and Packing</td>
<td>35</td>
</tr>
<tr>
<td>PTV Flow and Temperature</td>
<td>36</td>
</tr>
<tr>
<td>PTV Solvent Elimination</td>
<td>36</td>
</tr>
<tr>
<td>Direct (Packed Column) Inlets</td>
<td>37</td>
</tr>
</tbody>
</table>
GC Inlet Systems

As an analytical technique it is estimated that Gas Chromatography (GC) is capable of analysing approximately 10-20% of the known compounds (of which there are around $10^7$). Providing that the compound has a significant vapour pressure below 400-450 °C it can be analysed by GC and even if the compound is thermally labile at elevated temperature, this does not entirely exclude GC as a means of analysis.

It has been famously said that ‘If the column is the heart of the chromatography, then sample introduction may, with some justification, be referred to as the Achilles heel’ [1]. This reflects the practical reality that in GC analysis, the limiting factor in terms of quality is often the sample introduction system.

The function of the sample inlet is to introduce a representative portion of the sample as a narrow band onto the chromatographic column –failure to achieve this objective will significantly reduce the separation capability of the GC column. Because most samples that are analysed by GC are liquids, an essential feature of one common GC inlet type is that the sample and solvent are vaporised prior to reaching the column. These GC inlets are known as ‘Vapourising’ injectors. Other inlet types allow the introduction of the liquid sample at room temperature –prior to controlled heating of the analyte to avoid sample degradation. Both injector types will be studied in greater detail in this section.

General requirements for GC Inlets

- Does not cause more band broadening than the analytical column
- Allows a representative sample to be injected with good accuracy and precision
- The composition of the injected sample is representative of the original sample
- No discrimination based on differences in boiling point, polarity, concentration or thermal/catalytic instability
- Applicable to trace analysis as well as to undiluted samples
Split / Splitless Inlet

The most common inlet for capillary GC is known as the Split / Splitless inlet, which, as the name suggests, can be operated in two modes, split or splitless. This inlet belongs to a broad range of inlet types known as the Vapourising Inlets, as during the injection process, the liquid sample is vapourised into the gas phase prior to transfer onto the capillary column. This process has a number of advantages and disadvantages—all of which will be discussed further in this section. The injection principle of the split / splitless inlet is outlined below.

- a syringe containing the sample is used to pierce a rubber septum
- the sample is rapidly introduced into the heated inlet
- the sample liquid rapidly volatilises to the gaseous form and is constrained within a glass liner of fixed volume
- the gaseous sample is swept onto the column by the carrier gas
- depending upon the mode of operation, some of the sample may be directed away from the column

The inlet is designed to overcome a number of limitations with capillary chromatography—principally:

- Due to the high efficiency of the capillary column separation, the sample size should be small and the analyte band introduced should be as narrow as possible
- Capillary columns contain very small amounts of stationary phase (typically <10mg for a 25m column) and are therefore easily overloaded, so analyte on-column concentrations have to be minimised
**Injection Syringe:** glass autosampler syringes can be obtained in a number of different sizes to achieve various injection volumes, 1, 5 and 10 µL syringe volumes are typical. 0.5 – 2 µL injection volumes are typical.

It is important to ensure that the syringe is deactivated with respect to the target analytes and will not adsorb sample components.

Various needle point styles are available for manual and autosampler injection, and for use with different septa styles.

**Septum:** a rubber composite disk used to isolate the inlet from atmospheric pressure (and hence maintain an increased pressure inside the inlet) whilst the injection is made. The disk shaped septum is held under torque by the septum retaining nut. As the injection is made and the septum pierced by the needle, it will deform around the needle creating a seal – allowing the sample liquid to be injected whilst maintaining an increased pressure within the inlet.

Septa will eventually ‘core’ or split, and fail to maintain a gas tight seal during the injection process.

**Inlet gas supply:** the gas flow (pressure) necessary to maintain the correct flows at the various outlets is supplied here. Typically a backpressure regulated supply, the ‘total flow’ as it is sometimes known, will supply the flows used for:

- Capillary column flow (carrier gas flow).
- Split Flow (for split injection mode).
- Septum Purge Flow.

The gas used will be the carrier gas for the GC separation, typically Hydrogen, Helium or Nitrogen.

**Septum purge gas outlet:** septa are manufactured from rubber or polymer composite materials containing plasticisers and other compounds capable of outgassing at elevated temperature. In order to reduce the number of these contaminant compounds reaching the detector or fouling the GC column as septum purge gas flows across the underside of the septum.

The septum purge gas also helps to prevent contamination of the underside of the septum by sample components which may ‘overspill’ from the inlet liner during injection. This will help to reduce ‘carryover’ from injection to injection.

The septum purge flow rate is typically regulated using a forward pressure valve or pneumatic regulator and typical flows rates are in the region 2-5 mL/min.

**Inlet liner:** to constrain the sample vapour cloud within the inlet a glass or quartz liner is used. There are many varieties of liner for particular applications and injection modes.

Primarily the liner also allows for sample vapour splitting as well as offering an inert (deactivated surface), which can help to reduce the degree of analyte degradation or adsorption in the inlet.
**Split Valve / Split Line Gas Outlet:** this arrangement is used to discard some or all of the liner contents. In split injection the valve is constantly in the open position and the gas flow is regulated to determine the fraction of the sample vapour that is discarded relative to that which reaches the capillary column.

In splitless injection the valve is closed initially to ensure all analyte is transferred to the capillary column. It is then opened after an optimised period of time in order to discard residual solvent and sample vapours.

**Heated Injector Body:** the metal body of the inlet is usually surrounded by a radiative heating material (blanked) into which a heating element is placed.

It is important to establish a homogeneous heating profile over the whole length of the injector body to ensure that cold spots are not created at which less volatile analytes or sample components may condense and foul the inlet liner or other components.

In practice most inlets will have a temperature variation along the liner, however, for practical purposes this does not affect injection performance.

**Capillary Column:** the position of the capillary column within the inlet is of vital importance. The column tip is the position at which the sample vapour splitting will occur and instrument manufacturers will optimise the inlet and liner geometry for this occur at different positions within the inlet.

Failure to properly site the column within the liner can lead to increased sample discrimination, incorrect and irreproducible split ratios.

It is recommended that you closely follow your manufacturer guidelines on column positioning within the inlet. Most manufacturers will specify the column position using a distance between the ferrule tip and the column tip.
Retaining Nut and Ferrule: the capillary column is held in the lower part of GC inlet using a fitting into which the column nut and ferrule are screwed.

It is important that the correct ferrule type is used.

- Graphite or Vespel®/graphite ferrules may be used to seal the column to the injector – both ferrule types have advantages and disadvantages. Graphite ferrules are the easiest to use, and they are leak-free, universal for most systems, and preferred by most beginning capillary chromatographers. Because graphite ferrules are soft, they easily conform to column outside diameters and different types of instrument fittings. However, they can flake or fragment upon removal, causing particles to lodge in the injector or detector sleeves, and they will not hold a seal under vacuum.

- Vespel®/graphite ferrules are hard and they must match the column and fitting dimensions closely to seal properly. In addition, because Vespel®/graphite ferrules can deform during initial heating, they need to be re-tightened or leakage will occur. Vespel®/graphite ferrules do not fragment, can be reused many times, and are preferred by mass spectroscopists since they do not contaminate the ion source with particles and maintain their seal under vacuum.

Split Injection

In this injection mode the sample is introduced into the inlet liner where rapid volatilisation occurs. The sample vapour is then mixed with and diluted by the carrier gas flowing through the centre of the liner.

The diluted sample vapour then flows at high velocity past the column entrance where a small portion of it will enter the column. However, most of the diluted sample will flow past the column entrance and out of the inlet via the split line. The ratio of column flow to split flow will determine the ratio (or volume fraction) of sample entering the column to that leaving the inlet via the split line. The split flow rate may be altered to either increase or decrease the amount of sample reaching the column.

Split injection is conventionally used for analyses where the sample concentration is high and the user wishes to reduce the amount of analyte reaching the capillary column. As capillary columns have a limited sample capacity it is important that the column is not overloaded. A typical 25m GC column may contain only 10mg of stationary phase distributed over its entire length.

Split injection ensures that the sample is rapidly volatilised and transferred to the capillary column – hence ensuring a narrow analyte band. For this reason initial column temperatures for split injection tend to be higher than the boiling point of the sample solvent.
Capillary columns have limited amounts of stationary phase onto which analyte and sample components may adsorb. Initially on injection, if the stationary phase at the head of the column becomes ‘saturated’ the analyte will flood down the column extending the band of analyte. A concentration gradient will form at the head of the column and this will reflect as a ‘fronting’ peak once the analyte band reaches the detector. This process is shown schematically below:

**Table 1. Advantages and disadvantages of Split mode**

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Disadvantages</th>
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</thead>
<tbody>
<tr>
<td>• Simple to use</td>
<td>• Not suitable for trace analysis</td>
</tr>
<tr>
<td>• Rugged</td>
<td>• Suffers from discrimination</td>
</tr>
<tr>
<td>• Narrow analyte band on column</td>
<td>• Dependent upon linear geometry</td>
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<tr>
<td>• Protects column from involatile sample components</td>
<td>• Analytes susceptible to thermal degradation</td>
</tr>
<tr>
<td>• Easy to automate</td>
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</tbody>
</table>

**Split mode**

- High analyte concentration – stationary phase saturated
- Lower analyte concentration resulting from excluded analyte flooding forward from the head of the column
The GC peaks show a typically fronting (or 'sharkfin') shape as a result of the concentration gradient formed at the head of the analytical column.

Where:

| 1. 2,2-Difluorobiphenyl | 4. 4-Nitrophenol |
| 2. 2,4-Dinitrophenol | 5. 4-Nitroaniline |
| 3. 2,4-Dinitrophenol |

Analysis conditions. (Temperature Program)

<table>
<thead>
<tr>
<th>Column</th>
<th>Equity -5, 15m×0.10mm ID, 0.10µm.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oven</td>
<td>50°C, 30°C/min to 325°C (5 min)</td>
</tr>
<tr>
<td>Inj.</td>
<td>250°C</td>
</tr>
<tr>
<td>Det</td>
<td>360°C</td>
</tr>
<tr>
<td>Carrier gas</td>
<td>Hydrogen 40cm/sec @ 50 °C</td>
</tr>
<tr>
<td>Injection</td>
<td>1µL, 50:1 split</td>
</tr>
<tr>
<td>Liner</td>
<td>4 mm ID, cup style</td>
</tr>
<tr>
<td>Sample</td>
<td>PTE-5 test mix, 250-500µg/mL</td>
</tr>
</tbody>
</table>

Setting the Split Ratio

The ‘Split Ratio’ describes the ratio of gas flows between the capillary column and the split flow line – and effectively gives a measure of the volume fraction of the sample vapour that will enter the column.

The calculation of Split Flow is shown under the ‘Equations’ button opposite. Of course the magnitude of the split ratio will depend on the concentration of the sample injected and the capacity of the capillary column used.

Typical split ratios lie in the range 1:20 to 1:400 meaning that only 1/20 to 1/400 of the sample is injected onto the analytical column.

The split ratio is usually adjusted empirically to obtain a good balance between analytical sensitivity and peak shape. If the split ratio is too low peak shape will be broad and may show the fronting behaviour associated with overloading. Of course if the split ratio is too high, too little sample will reach the column and the sensitivity of the analysis will decrease as peak areas decrease.

When using thick stationary phase film columns (>0.5 mm) or wide bore (0.533 mm i.d.) columns, the sample capacity increases and lower split ratios of 1:5 to 1:20 are typical.
With very narrow GC columns (<100 mm i.d.) split ratios can be as high as 1:1000 or more.

The split ratio is inversely proportional to analyte peak area, i.e. doubling the split ratio will halve the peak area. This is due to the increase in split ratio decreasing the amount of analyte reaching the capillary column.

**Sample Discrimination**

The phenomenon of sample discrimination leads to a non-representative sample entering the analytical column compared to the original sample. Sample discrimination is best described using the example on the ‘Data’ button opposite, which shows the detector response to an injection of n-alkanes at equal concentration. The normalised line shows the original sample composition, and the expected response for each of the n-alkanes. The more highly volatile n-alkanes show total recovery, however for C 25, only half of the analyte present in the sample is introduced into the column, and the recovery of C 37 is less than 25%.

For higher boiling (less volatile) analytes, the residence time of the syringe needle is too short. The analyte will condense on the cold inner and outer surfaces of the needle – prior to it being withdrawn from the inlet. Some less volatile analytes may never properly volatilise and the sample passes the split point (head of the capillary column) as a mixture of sample vapour and non-uniform liquid droplets. Several approaches to the problem have been postulated including:

- Optimisation of liner geometry and packing materials to promote sample mixing and volatilisation
- Optimising the injection routine (filled needle, hot needle, solvent flush, air flush, sandwich method etc.)
- Improved instrument design to reduce fluctuations in split flow

Due to low inlet needle residence time, low volatility (high boiling point) analytes recondense onto the (relatively) cold injection syringe needle and are withdrawn from the inlet.
Low volatility analyte still held in non-uniform liquid droplets—carried past the column entrance (split point) and out of the inlet. High volatility analytes in the gas phase preferentially samples into the column.

Discrimination due to differences in boiling point: Hot split injection of solution containing equal amounts of normal alkanes in hexane

In general, the least amount of discrimination is obtained if the injection is performed as rapidly as possible. For this reason, fast autosamplers generally give less discrimination than manual injection.
Injection Volume

The nature and volume of the sample solvent injected into the split/splitless inlet will have a major effect on the accuracy and reproducibility of quantitative analysis and the chromatographic peak shape. As the injection is made, the sample solvent rapidly volatilises and expands into the gas phase. To avoid quantitative problems, the total volume of the gas should be able to be constrained within the volume of the inlet liner.

If this is not the case, then the excess gas will spill over into the inlet gas supply and septum purge lines. The temperature in these lines rapidly decreases, and it is possible for the sample solvent vapour (containing the analyte), to recondense, ultimately depositing analyte onto the inner walls of the tubing.

When the next ‘overloaded’ injection is made, the sample solvent from this injection will again ‘backflash’ into the gas lines. In this instance analyte deposited during the previous injection will be ‘lapped’ back into the inlet –ultimately finding its way onto the column. This will cause ‘carry-over’ and will reduce quantitative accuracy and reproducibility.
The expansion volume of the sample solvent is governed by the inlet pressure and temperature, as well as the natural expansion coefficient of the solvent. It is possible to predict the expansion volume and hence the volume of solvent that may be safely injected into an inlet liner of known volume, under set temperature and pressure conditions.

**Ghosts peaks.**

A technique known as ‘pressure pulsed’ injection may be used, in which the inlet pressure is raised during the sample injection cycle. This constrains the expansion of the solvent within the inlet liner and allows for large volume injections.
Split Injection Experiments

One of the main advantages of Split injection techniques is the rate at which the sample vapour cloud is transferred to the GC column. This transfer speed affects the efficiency of the resulting GC peaks — faster transfer leading to narrower, more efficient peaks. The rate of gas flow through the liner effectively dictates the sample transfer speed.

Splitless Injection

Splitless injection is analogous to split injection in many ways. The hardware used for splitless injection is almost identical to the split injector and most manufacturers will use the same inlet for both split and splitless injection — hence the term split/splitless injector. Just as with split injection, the sample is introduced into a hot inlet using a sample syringe where it is rapidly injected and volatilised. The splitless injector also belongs to the family of ‘vaporising’ injectors. Post injection, there are a number of differences in the way that the splitless injector works and a typical splitless injection routine is outlined below:

- The sample is introduced into the inlet, via the septum, using a syringe
- The sample is vapourised and is mixed with and diluted by the carrier gas
- Initially the split line is turned off using a valve in the split line to prevent the escape of the sample vapour and carrier gas
- ALL OF THE SAMPLE is transferred to the capillary column by the carrier gas during this initial SPLITLESS phase of the injection
- The transfer of the sample vapour (diluted with carrier gas) from the inlet is much slower compared to split injection
- The sample vapours are trapped (condensed) on the head of the analytical column using a low initial oven temperature
- At an optimised time the split line is turned on to clear the inlet of any residual vapours
- The oven temperature is programmed to elute the analytes from the column
Advantages:
- Rugged
- Excellent for trace analysis ~(0.5ppm with FID should be easily achievable)
- Easily Automated
- Highly reproducible with optimised inlet settings

Disadvantages:
- More complex than split injection – more parameters to optimise
- Suffers from discrimination
- Analytes susceptible to thermal degradation – more so than with split injection due to longer analyte residence time in the inlet
- Column contamination possible – all sample components introduced into the column

Optimising Splitless Injection

Purging the inlet

During splitless injection, it is of vital importance that the inlet is purged of residual vapours once the analyte has been transferred to the capillary column. If this is not done, the solvent peak will show a high degree of tailing and the GC baseline signal may be noisy and rise markedly as the analysis progresses. This is due to the slow bleed of
excess solvent and sample (not analyte) components from the inlet into the capillary column.

The inlet purge is achieved by actuating the Split (Purge) Valve that allows a high split flow through the liner, which quickly purges the residual vapours from the inlet. The split flow is high as the aim is to quickly purge the inlet, split flows of 100-200 ml/min. are typical. The time from the beginning of the injection to the time at which the split line is turned on is known as the splitless time. It is vital that the splitless time is optimised for each application. Too short a splitless time will mean that analyte still resident in the liner will be discarded via the split line. This may lead to poor analytical sensitivity and reproducibility. Too long a splitless time will lead to badly tailing solvent peaks, extraneous peaks and a rising baseline, making reproducible integration difficult.
Spiltless time

Spiltless time too SHORT – loss of higher boiling analytes

Spiltless time too LONG – broad solvent peak and rising baseline

The splitless time is usually empirically optimised by monitoring the peak area of a mid-eluting peak in the chromatogram. The peak area is plotted against the splitless time and a plot of the form shown under the Data Optimisation button opposite is derived. For reproducible analysis the splitless time should be chosen just onto the plateau of the area response curve as indicated. Typical splitless times lie in the region 20 – 90 seconds.

Analyte Focussing

The analyte is slowly introduced from the inlet during the whole of the splitless time (the inlet volume may be exchanged as few as two times during this whole splitless period).

This slow sample vapour transfer would result in the analyte band entering the column over a period of 30 – 60 secs, or so depending upon the exact analytical conditions. This would entirely negate any efficiency gained through the use of capillary columns and the resulting chromatographic peaks would be unacceptably broad.

To overcome these problems Focussing techniques are used, which usually involves setting the initial oven temperature at a suitably low value ensuring that condensation and reconcentration takes place in the column.

Two discrete focussing mechanisms can be identified:
Cold trapping: higher boiling analytes are condensed in a tight band in the temperature gradient between the inlet (~250 °C) and the column oven (~40 °C)

Solvent effect: low boiling (more highly volatile) components remain dissolved in the solvent, which also condenses on the inner wall of the GC column at low initial oven temperatures. The solvent slowly evaporates to give a thin concentrated band of analyte.
For volatile analytes (whose boiling point is close to the sample solvent), a different focussing mechanism exists. If a suitably low initial oven temperature is used, the sample solvent will condense as a film on the surface of the column bonded phase. This film will contain the volatile analytes in a disperse form. The flow of the carrier reduces the vapour pressure within the column and the solvent band will evaporate slowly from the inlet end. The band will evaporate to a much lower solvent volume, containing a concentrated, narrow band of the analyte—effectively overcoming the band broadening incurred in the slow sample vapour transfer from the inlet.

This mechanism works most effectively when the initial oven temperature is at least 20°C below the boiling point of the sample solvent.

**Solvent Choice**

All of the comments regarding solvent injection volume are also true for splitless injection. The allowable solvent volume does not differ greatly between split and splitless injection and in each case the allowable volume may be calculated from a combination of the liner dimensions, solvent type and inlet head pressure. However, with splitless injection there is one further constraint on the sample solvent. The nature, or more specifically the polarity, of the sample solvent MUST match the polarity of the stationary phase used in the GC column.

The solvent effect relies on the formation of a single contiguous film coating the inside wall of the capillary column. This will only occur if the solvent polarity is matched to that of the stationary phase. If this is not the case (e.g. using methanol as the sample solvent with a methyl silicone stationary phase), the solvent will not condense as a film, rather droplets of solvent will form, each acting with an individual solvent effect. This will lead to broad, split or fronting & tailing peaks (the latter being known as the ‘Christmas Tree’ effect due to the triangular peak appearance).

These issues are generally most prevalent for earlier eluting (more volatile) analytes as later eluting analytes tend to be focussed via the cold trapping mechanism. Solvent droplet formation usually only occurs when a critical solvent injection volume is exceeded (usually between 1 and 2 mL). If a mismatch between the sample solvent and column stationary phase is required (due to sample solubility characteristics), then a retention gap may be used. This is a short piece of capillary column (0.5 – 3m) which is coated using a phase which matches the sample solvent polarity.
Injection of a solution of n-alkanes in methanol on a dimethylsiloxane (non-polar) stationary phase.

- Poor early eluting peak shape due to polarity mismatch between sample solvent and stationary phase polarity.
- Peak focussed predominantly by the Cold Trapping effect.
Splitless Injection Experiments

In this section we have discussed several critical parameters that are important for good splitless injection. You need to establish a splitless injection method for low concentrations of your analyte of interest.

**IMPORTANT:** Refer to the online material to access to the highly interactive experiment.

Choosing an Inlet Temperature

Selection of the correct inlet temperature for Split or Splitless inlet operation is vital. It is necessary to have a high enough temperature to ensure efficient and complete volatilisation of all sample components –this will ensure that temperature (inlet) discrimination of the higher boiling (less volatile) components is minimised.

However, it is also important to avoid thermal degradation (decomposition) of analytes and sample components, which will lead to poor quantitative reproducibility and/or fouling of the inlet liner.

The accepted arbitrary inlet temperature to begin method development for new analytes and applications is 250°C –and this temperature can be used as a general guide in all cases except where a higher or lower temperature is known to be required (i.e. where particularly thermally labile analytes are involved or where the sample is particularly high boiling).

With the inlet set to 250°C a ‘scouting’ temperature gradient may be employed which will elute analytes and sample components across a wide range of boiling point values. In this way, the elution temperature of the highest boiling component of the sample may be estimated. The inlet temperature should then be set to at least 50°C above this temperature to ensure efficient sample volatilisation.

There are a number of chromatographic symptoms that may indicate problems with analyte thermal degradation –mostly involving a plateau at the front of rear of the chromatographic peak. If this symptom is seen the inlet temperature may be reduced in 20 °C steps until the problem is resolved.

![Scouting Temperature Gradient](image)
Typical peak shape encountered with inlet thermal degradation of analytes – notice the plateau appearance of the baseline either before or after the peak.

Typical peak shape of analyte Inlet thermal degradation

Optimisation of inlet temperature can be carried out by decreasing the inlet temperature in 20°C steps – once peak shape problems are overcome the reproducibility of the peak area should be confirmed with at least 6 injections giving a satisfactory relative standard deviation (RSD) (<1%). Both of these checks are required to indicate thermal stability (it can never be truly ruled out!).

Liners for Split/Splitless Injection

The selection and correct use of liners is of critical importance in Split and Splitless GC injection. The liner has many functions which include:

- To constrain the volatilised components of the sample
- To allow the sample to be split through excess sample and carrier escaping from the liner outlet (some instrument designs)
- To cause mixing of the sample vapours with the carrier (split injection)
- To prevent involatile material from fouling the GC column
- To avoid analyte thermal degradation
- To decrease the potential for inlet discrimination

All of the above are achieved through a number of features of the liner design. These features are often poorly understood and the interactive diagram opposite outlines the main features of typical GC inlet liners for a better understanding. The main variables in GC liners selection are:

- Liner internal diameter
- Packed / Unpacked
- Packing Position
- Liner internal geometry and features including:
  - Upper Gooseneck
  - Lower Gooseneck
  - Inverted (Jennings) Cup
  - Baffled
  - Deactivation Type
**Liner top**

**Wide bore (~4mm) open top:** Gives good access for the sample syringe needle and ensure high carrier gas velocity through the liner. Does not prevent backflash via the top of the liner.

**Single gooseneck restriction:** Essentially exists to prevent backflash from the liner top when injecting large volume samples and carrier over is a problem. Care required to match gooseneck ID with needle ID to prevent breakage. Some users report a reduction in sample mixing with this feature.

**Liner geometry**

**Fritted liners:** can also be used when glass wool is found to be too active towards the analytes of interest. These are usually used for split injection.

**Baffled liners:** are designed to promote improved sample vapour mixing and reduce mass discrimination as well as providing a large surface area for large volume and programmed thermal vaporisation injections.

**Liner packing**

**No packing material:** Allows for very rapid transfer of sample vapours onto the column. Does not encourage mixing therefore more prone to mass discrimination and does not protect the column from non-volatile material. Will reduce sample adsorption (irreversible) with compounds such as pesticides – and this may improve quantitation.

**Small amount of packing material:** for split injection. Positioned in order to ‘wipe’ the exterior of the needle to help prevent high mass discrimination, as well as providing good sample vapour mixing. The large surface area encourages volatilisation of higher boiling material, again preventing discrimination and prevent the liquid analyte from contacting the metallic bottom seal of the inlet. The packing will also prevent non-volatile material (septa shards, sample components etc.) from fouling the lower liner, liner internal surfaces and inlet. Packing is usually quartz or glass wool folded into a 1cm plug and deactivated to reduce activity towards active compounds which may affect quantitative reproducibility or cause peak tailing.
Increased volume of glass wool packing for trace splitless analysis: All of the reasons for using packing with split injection are applicable here –however for trace analyses the positioning of the wool must be optimised and the volume is usually increased slightly. It is important to emphasise that the wool will prevent the analyte from contacting the lower metallic part of the inlet when a straight through liner is used.

Liner bottom options

Wide bore unrestricted liner: Works well for splitless injection as much of the mixing is carried out at the bottom of the liner. May cause problems with thermally labile analytes as they are able contact the metal injector seal before entering the column.

Single gooseneck restriction: Mainly used for splitless injection –the gooseneck restriction ‘focuses’ the analytes onto the column. Prevents thermal decomposition by protecting analytes from the metallic inlet base seal. May give low boiling analyte discrimination.

Inverted (Jennings) cup: Used mostly with split injection. Provides improved mixing of the sample vapours with the carrier through turbulence and direction change of flow. Aim is to decrease discrimination in the liner when the sample and carrier linear velocity is high.

Inverted (Jennings) cup with packing material: The packing material serves too further improve mixing of sample vapours, acts as a high surface area to allow less material to condense and re-volatile and to trap particulate material.

Inverted cup (Laminar) liner: Again improves mixing of sample vapours in split injection –only works well with high molecular weight compounds.
**Single gooseneck restriction (bottom)/Packing material (Bottom):** Mainly used for splitless injection—the gooseneck restriction ‘focuses’ the analytes onto the column. The glass wool prevents debris and particulate material from reaching the column, as well as providing a large surface area to encourage volatilisation of high boiling point compounds (minimising high mass discrimination).

**Liner I.D.**

**Narrow I.D. liners:** Can also be packed for trace splitless analyses, the reasons for use are the same as with split or splitless inlets.

**Effect of liner I.D. – Improving peak shape in splitless injection**

![Graph showing peak shapes with different liner I.D.]

**Liner (packing) deactivation using trimethylsilyl chloride reagents**

Deactivation is more critical during splitless injection than split injection. In splitless injection, the split vent is usually closed for about 1 minute and these results in a low liner gas flow. A low gas flow will result in slow transfer and the residence time of analytes within the liner is increased. Thus for thermally labile and heat sensitive compounds, the interaction time of the analyte with the inner surface of the glass liner is increased and this enhances breakdown. The effect is not nearly as pronounced in split injection because the residence time in the liner is very short.

![Chemical reaction diagram]

Deactivation is usually carried out by end capping the silanol (hydroxyl) groups on the quartz surface and replacing them with a non-polar trimethylsilyl groups (see the reaction scheme).
Septa for Split / Splitless injection

The septum isolates the inlet from atmospheric pressure—allowing the inlet to be pressurised. The septum is pierced with the injection syringe needle to allow the sample to be injected.

The septum is ‘plastic’ in nature (i.e. it is deformable), and is held under mechanical pressure with a retaining nut—allowing it to seal around the injection syringe needle and maintain inlet pressure during the injection phase.

Care is required with the torque applied to the septum nut. Over tightening the nut will compress that septum and may promote splitting. Under tightening the nut may cause leaks and pressure failures. Many instrument manufacturers recommend having the septum nut ‘fingertight’. There are many types of septa available, which vary in characteristic—some of these are explained below. Care should be taken to use the correct septum and syringe combination for optimum performance.

**Temperature limit:** Each septum is manufactured using different materials to give an upper temperature limit—beyond which bleed will be unacceptable. Upper temperature limits will usually range between 300°C and 450°C.

**Size:** The diameter and depth of the septum is important to achieve a good fit in the upper part of the inlet as well as the correct compression characteristics when the septum nut is tightened. Check with your manufacturer for the correct size.

**Material:** The material of construction generally dictates the temperature profile and the level of bleed seen from the septum in routine use.

**Sandwich:** Some septa are manufactured using ‘layers’ of different material, usually a softer upper layer to minimise ‘coring’ and a temperature optimised lower layer to give less bleed. Septa may have up to three discrete layers.

**PTFE Faced:** The upper and/or lower face of the septum may be covered with a thin layer of PTFE. This performs two functions—on the upper surface it will help to reduce septum coring. On the lower face it will also help to reduce septum bleed.

**Pre-drilled:** Many septa are available with a counter-sun k pre-drilled hole through to act as a needle guide and to extend the number of injections possible with the septum.
Septum Problems

The materials used to plasticise the septum bleed continuously (phthalates etc.). In capillary GC the bleed products may give rise to discrete noise peaks and may also result in a rising baseline as shown opposite. The septum purge flow of the inlet helps to reduce these effects, however correct septum choice with regards to inlet temperature is important.

Septa eventually wear through continuous piercing, and will ‘core’ to deposit fragments into the liner. Eventually the liner will split or core so badly that a pressure seal is no longer maintained during the injection phase. Peak shape will suffer when this occurs and the baseline shifts are diagnostic of a worn septum.

The figure below shows a typical chromatographic symptom created by a leak at the septum during injection. The baseline position changes after the elution of a large (usually solvent), peak. This indicates that the septum is not sealing correctly around the needle will therefore leak during injection and for a short time afterwards. This symptom is exacerbated if the diameter of the needle used is too large.

A very badly cored or split septum will leak and fail to maintain system (head) pressure. Without a proper maintenance regime, this may be the first time you realise that the septum is causing the problems!

**Column:** 30m x 0.53mm (1.0µm) (100% PDMS)
**Oven temp:** 40°C to 300°C @ 15°C/min.
**Inj. / Det. Temp:** 330°C.
**Carrier gas:** Hydrogen.
**Linear vel:** 40cm/sec.
**Atten:** $4 \times 10^{-11}$ AUFS.

**Column:** 30m x 0.25mm (0.25µm) (100% PDMS).
**Oven temp:** 100°C.
**Inj. Temp:** 260°C.
**Carrier gas:** Hydrogen.
**Linear vel:** 50cm/sec.

**Baseline Shifts Encountered with Leaking Septum**

- Normal
- Problem (flow increase)
- Problem (flow decrease)
Troubleshooting & Maintenance

Following a basic preventative schedule can prevent most septum related problems

1. Change septa regularly (users will become familiar with expected lifetimes of septa for particular applications), but daily and with column change for instruments that are used constantly.

2. Use the correct injection syringe, selecting the correct diameter needle for your particular autosampler and use a cone tip rather than a pointed needle to prevent excessive coring.

3. Condition septa. Septa may be conditioned by holding at 50°C above the expected operating temperature for an hour or so. Cycle through the oven temperature program to be used at least twice after conditioning to clear any residues from the system.

4. Ensure that the septum nut is tight. Studies have shown, that septa in an HP5890 GC gave up to 400 more injections at 12 in/lbs torque on the septum nut than at 4 in/lbs. Syringe needles on modern autosampler systems can easily pierce septa under 20 or more in/lbs of torque before bending.

5. Ensure that the injection liner is clean. Shards of septa that fall into the injection liner will constantly produce volatile species, giving rise to baseline instability and increased background during the whole analysis cycle. Inspect the liner (and clean if necessary), every time you change the septum.

Cool-on-Column Inlet

In cool-on-column (COC) injection, the sample solvent is injected directly into the column using a small diameter needle –this inlet type does not use flash vaporisation.

After injection the oven temperature is raised and during the temperature program analytes will begin to chromatograph according to their boiling point and affinity for the stationary phase.

By using an inlet and oven temperature appreciably below the boiling point of the sample solvent (and analytes), unfavourable processes such as discrimination and analyte thermal decomposition are eliminated. This makes COC injection particularly suited to mixtures containing high and low volatility analytes and for trace analysis where quantitative reproducibility is important.

The COC inlet hardware (opposite) is reasonably straight forward. As usual a syringe is used to introduce the sample via a septum (with purge gas). The syringe enters the column and once the sample solvent is expelled, it forms a long contiguous film on the inside wall of the column. The film length is appreciably longer than seen with splitless injection, as there is no negative temperature gradient to halt the film formation process. For example a 1 mL injection may wet as much as 50cm of a capillary column.

Without refocusing (reconcentration) this may lead to very broad analytical peaks – therefore several optimisation processes may be employed to improve peak shape.
Most manufacturers encourage the use of a ‘megabore’ (0.53mm i.d.) column or retention gas so that stainless steel needles may be used to increase robustness of the injection technique.

**Table 2. Needles for COC**

<table>
<thead>
<tr>
<th>Column Internal Diameter (i.d.) (mm)</th>
<th>Needle Type</th>
<th>Needle Outside Diameter (o.d.) (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.20 – 0.25</td>
<td>Fused Silica</td>
<td>0.17</td>
</tr>
<tr>
<td>0.32</td>
<td>Fused Silica</td>
<td>0.23</td>
</tr>
<tr>
<td>0.53</td>
<td>Stainless Steel</td>
<td>0.26 / 0.23 dual diam.</td>
</tr>
</tbody>
</table>

**Performance**

On-column injection allows accurately known volumes to be very reproducibly introduced directly onto the column. This greatly reduces the chance for discrimination and thermal degradation. On-column injection is very suitable for high boiling components because no vaporization takes place during the injection period. The figure shows the peak areas obtained in the analysis of a sample containing equal amounts of n-alkanes as a function of the carbon number. Since no intermediate vaporization step is present during on-column injection, the composition of the COC data show a much better agreement with the actual amounts of the various n-alkanes compared to the splitless injection data.
There are three important aspects in optimising Cool-on-column (COC) injection:

- Initial oven & inlet temperature.
- Nature of the sample solvent.
- Use of a retention gap.

The initial oven & inlet temperatures will ultimately govern the peak width in the chromatogram. It is essential that the long film of sample solvent created on injection is reduced in order to reconcentrate the dissolved analytes. It is generally accepted that temperatures between 20 °C below and 10 °C above the sample solvent boiling point work best. At higher temperatures selective vaporisation may occur from the syringe, leading to discrimination.

When using low boiling solvents, the inlet has to be cooled to prevent selective vaporization. Some COC inlets are equipped with a “secondary” cooling system that cools the front portion of the column to an appropriate temperature. This allows on-column injection to be performed without the need to cool the entire GC oven and acts as an aid in cooling the inlet after it has been heated during the GC run.

As in splitless injection, the formation of a homogeneous solvent film at the front of the column is of utmost importance. When polar solvents are introduced onto non-polar stationary phases, the incompatibility of solvent and stationary phase will again result in droplet formation in the column, giving rise to broadened peaks and peak splitting. In contrast to splitless injection, this no longer occurs only for the low boiling components, but will be observed throughout the entire chromatogram.
Use of Retention Gaps for Cool-on-Column Injection

A retention gap is a few meters of deactivated, non-coated fused silica capillary column connected in front of the analytical column.

The use of long retention gaps (1-5m) allow increased injection volumes (2-5 mL) by providing a large surface area for solvent film formation. The retention gap is non-retentive towards the analyte (hence the name), and this causes the analytes to migrate towards the analytical column, which is connected to the retention gap, at the start of the temperature program. This results in the analyte collecting (or focussing), on the stationary phase at the beginning of the analytical column.

In cool-on-column injection the entire sample is introduced directly into the column, the retention gap also acts to protect the analytical column from involatile sample components. The retention gap may be replaced at less expense than the analytical column when it becomes fouled.

Regardless of the analytical column internal diameter, a wide bore (0.53mm) retention gap may be used, which will allow the use of stainless steel needles for direct sample
injection. This increases the robustness of the technique and will allow automation using an auto-sampler device.

Programmed Temperature Vaporisation (PTV) Inlets

Programmed temperature sample introduction was first described by Vogt in 1979. Programmed temperature vaporising injectors (PTV) closely resemble split/splitless inlets but differ in two very important ways:

- The inlet is kept cool during sample introduction—allowing the analyte to condense inside the liner, whilst the solvent is vented via the split line.
- The inlet has a very low thermal mass, which allows rapid heating to transfer the analyte to the GC column after solvent venting has taken place.

These two important differences in inlet design give the PTV several important advantages:

- Large volumes may be injected at controlled speeds into the inlet allowing the introduction of very large sample volumes (>100 mL is possible)
- Discrimination due to differences in analyte boiling point can be eliminated

The PTV inlet can be used in several modes, including ‘cold split’ and ‘cold splitless’. Perhaps the most useful mode is ‘solvent vent’, where the sample is introduced at a controlled rate either ‘at once’ or using a series of smaller injection aliquots. The principle is the same in both cases, the analyte condenses onto the liner walls or packing, whilst the solvent is vented via the inlet split line. Once the majority of the solvent is eliminated,
the split valve is closed and the analyte transferred to the column in splitless mode. Very low detection limits may be achieved in this mode.

Application Example – Analysis of an automated extract of water.

1.2 L of water was extracted with 0.3 L of ethyl acetate. 250 mL of the ethyl acetate extract was injected at an injection rate of 25 mL per minute. This ‘at-once’ injection using a controlled speed is the alternative to injecting several smaller aliquots of sample with a pause between each injection. Injection speed is a key variable.

The detection limits observed were in the low ppb range. From this example it is clear that the PTV inlet is an excellent system for the introduction of large sample volumes in capillary GC. Using this technique detection limits can be routinely improved by more than a factor of 100.
PTV Sample Flow Rate

Using PTV Inlets in Solvent Vent mode requires a degree of skill and knowledge of the key variables for sample introduction – some of these key variables are explained here.

When using multiple small injections the time interval should be sufficiently long to allow almost all the solvent to evaporate. Too short an evaporation time may result in loss of components due to flooding of the liner, and loss of liquid sample via the split vent. This phenomenon will also occur if the injection rate is too fast when using ‘at once’ injection.

If, on the other hand, too long a time interval is used, excessive loss of the more volatile sample constituents may occur. The optimum interval time is generally in the range of 2 to 20 seconds, and depends on a number of parameters such as the liner temperature, the pressure in the liner, the split flow, and the vapour pressure of the solvent.

Due to the lack of controlled speed programming during injection, this technique is limited to a maximum injection volume of 25 mL and to determining compounds with lower vapour pressures (higher boiling points).

The following table lists injection speeds for a number of solvents where the initial PVT temperature is 20°C and the split flow is 200mL/min.

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Injection speed (μL/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Toluene</td>
<td>26</td>
</tr>
<tr>
<td>Hexane</td>
<td>82</td>
</tr>
<tr>
<td>Acetone</td>
<td>60</td>
</tr>
<tr>
<td>Methanol</td>
<td>17</td>
</tr>
<tr>
<td>Carbon Disulfide</td>
<td>81</td>
</tr>
<tr>
<td>Acetonitrile</td>
<td>17</td>
</tr>
<tr>
<td>Ethanol</td>
<td>12</td>
</tr>
<tr>
<td>Ethyl Acetate</td>
<td>32</td>
</tr>
<tr>
<td>Pentane</td>
<td>225</td>
</tr>
<tr>
<td>Isooctane</td>
<td>30</td>
</tr>
<tr>
<td>Diethyl Ether</td>
<td>210</td>
</tr>
</tbody>
</table>

The injection speed for ‘at once’ injection can be estimated empirically or calculated [1].
PTV Liner Type and Packing

If the injection volume is 10 μL or less, an unpacked baffled liner is usually effective in trapping the analytes for large volume injection.

For larger volumes, glass wool or beads help both solvent evaporation and analyte trapping. A straight liner, packed so that the syringe needle just touches the packing, should be used. It is important to use clean and deactivated materials. Injection of large volumes can cause non-volatiles to build up in the liner. The liner should be checked frequently when developing new methods to determine an acceptable period of use.

By using liners packed with a selective adsorption material, such as Tenax®, Carbotrap® or Chromosorb®, the range of components that can be trapped in the liner can be significantly extended towards the more volatile components. With liners packed with these materials, even relatively volatile species (e.g. n-C4) can be trapped quantitatively at liner temperatures around or slightly below room temperature. With the addition of sub-ambient cooling, components down to n-C2 can be trapped.

The analysis of high boiling components with packed liners is not possible because of the extremely high temperatures that would be required for thermal desorption leading to thermal degradation of the components or the adsorbent.

Table 4. Selected liners

<table>
<thead>
<tr>
<th>Liner</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image" alt="PTV liner, single baffle, 2 mm I.D., glass wool deactivated, 180μL volume. For large volume injectors, not for extremely active compounds." /></td>
<td>For large volume injectors, not for extremely active compounds.</td>
</tr>
<tr>
<td><img src="image" alt="PTV liner, single baffle, 2mm I.D., glass wool deactivated, 200μL volume. For general purpose PTV applications." /></td>
<td>For general purpose PTV applications.</td>
</tr>
<tr>
<td><img src="image" alt="PTV liner, multi baffle, 1.5 mm I.D., glass wool deactivated, 150μL volume. For active compounds, drugs, pesticides." /></td>
<td>For active compounds, drugs, pesticides.</td>
</tr>
<tr>
<td><img src="image" alt="PTV liner, fritted glass, 1.5 mm I.D., glass wool deactivated, 150μL volume. For large volume injectors, all but extremely active compounds." /></td>
<td>For large volume injectors, all but extremely active compounds.</td>
</tr>
</tbody>
</table>

Large volume injection of a 30 ppm solution of normal alkanes

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PTV Flow and Temperature

The best results are obtained when the boiling point difference between the solvent and the analytes is at least 150°C. A smaller difference will result in some loss of analytes but, due to the design of the system, this loss is reproducible and will not affect quantitation. Lower boiling solvents (b.p.< 120°C) work best. An initial inlet temperature set at 30°C below the boiling point of the solvent is a good starting point. When working with volatile solvents and analytes, cryogenic cooling (either liquid CO₂ or N₂) can be beneficial.

A carrier gas flow that provides optimum separation should be used. Normal split ratios are best (50:1 ~ 200:1). The split ratio can be increased if needed during large volume injection method development. In general lower temperatures with higher flows are more desirable than high temperatures with low flows.

PTV Solvent Elimination

Solvent venting injection techniques can also be used to remove small quantities of a solvent prior to introduction. This could be advantageous for the introduction of polar solvents on non-polar columns or for removing solvents that are not compatible with the detector being used.

Also, in many cases, removing most of the solvent for a typical sample injection volume of 1-2 μL will greatly improve the chromatography.
Typical examples of solvent/detector systems that are not compatible are: chlorine-containing solvents with nitrogen/phosphorous detectors and halogen-containing solvents with electron capture detectors.

If vulnerable non-chemically bonded stationary phases are used, solvent elimination can have a favorable effect on the lifetime of the chromatographic column.

The figure opposite illustrates the effects of solvent venting on peak shapes observed when a polar solvent is introduced onto an a-polar column, with and without solvent elimination. The injection with solvent venting clearly gives better peak shapes.

Plasticizers in LC-extract (acetonitrile) 60m DB5 0.25mm×0.25μm. Introduction of 2μL of acetonitrile containing 1-chlorodecane on an a-polar column, without and with solvent venting.

Direct (Packed Column) Inlets

As their name suggests, packed column inlets are used for sample introduction into packed GC columns and are the original type of vaporising injector. Many different varieties of this injector exist ranging from a simple device containing a septum and carrier gas flow controller, to more sophisticated electronic pressure control devices with septum purge. In all cases the fundamental premise of the vaporising injector applies, in that the liquid sample is introduced into the inlet where the sample and solvent are vaporised and the resulting gas phase mixture passes into the column containing the stationary phase. The most basic design of packed column inlet allows the use of syringe to directly introduce the sample into a void at the top of the packed column—which acts as liner and the gaseous components pass through a glass wool plug into the column packing. More sophisticated designs contain a separate (narrow internal diameter) liner, which constrains the expansion of the sample and results in narrower chromatographic peaks. More complex inlets also allow the use of septum purge to protect from sample carry-over and improve both qualitative and quantitative analysis.
Packed GC columns are wider in diameter than capillary columns (2-4mm i.d. compared to 0.1 – 0.5mm) are manufactured from either glass or stainless steel and contain an inert pellicular support onto which the stationary phase is bonded. As packed columns have a much higher capacity than capillary columns, it is not usual to split the sample and so all of the sample and solvent vapour enters the column. The efficiency of packed column GC separations is inherently lower than obtained using capillary columns, therefore voids created through the use of wide bore liners do not impact the separation quality so drastically.
In this simple packed column inlet the GC column itself provides the void space in which sample volatilisation occurs. The vaporised analyte and solvent passes through a plug of glass wool directly into the stationary phase. As the separation is inherently less efficient than with capillary columns – the impact of the large void on separation quality is not so drastic.

Care needs to be taken with analytes which adsorb strongly to the glass wool and the wool needs to be replaced regularly as it becomes fouled with non-volatile sample residues.
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