

Split Injections in Gas Chromatography: How to Reduce Inlet Discrimination By Using a Liner with Glass Wool

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Introduction

Inlet discrimination is a problem encountered by GC analysts when making a split injection. It is characterized by smaller than expected peak response for less-volatile compounds that gradually gets worse as retention time increases. The cause of this problem stems from both the sample and the injection technique. Upon injection of a sample with multiple analytes into a heated inlet, different compounds enter the gas phase at different rates. This means that a disproportionate amount of sample will be purged from the split vent.

This article describes how the use of a liner containing glass wool can help reduce inlet discrimination and ensure more accurate analysis. The article presents comparative results from two experiments using different types of liners.

Experiment

An Agilent 6890 gas chromatograph coupled to an Agilent 5973 MSD was used for the experiment.

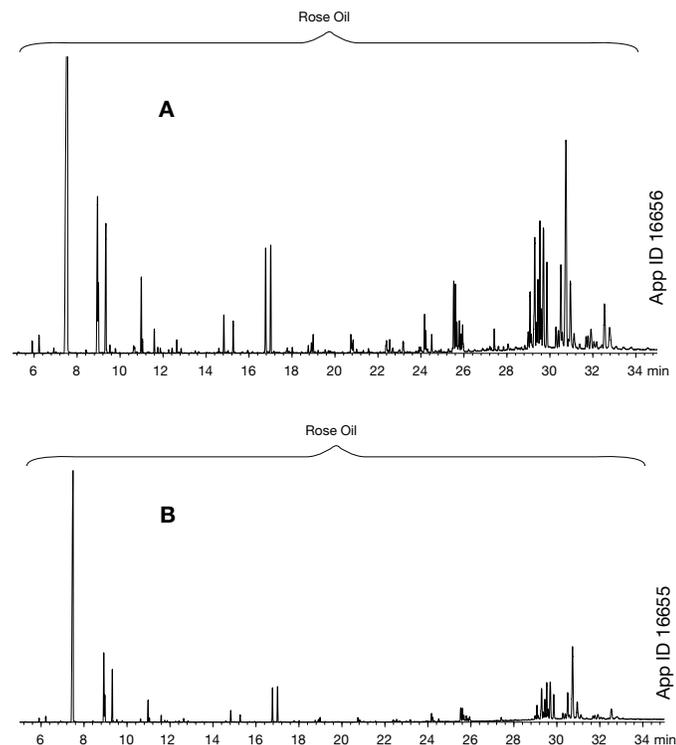


Figure 1 Response for the rose oil injection using a liner containing glass wool versus a liner that does not contain glass wool. The rose oil response using the liner with glass wool (A) is significantly larger than that of the liner without glass wool (B).

Running conditions:

Column: Phenomenex® Zebron™ ZB-XLB
Dimensions: 30 meter x 0.25 mm x 0.25 μ m
Part No.: 7HG-G019-11
Injection: Split 25:1 @ 250 °C, 1 μ L
Oven Program: 50 °C to 340 °C @ 10 °C/min for 10 min
Carrier Gas: Helium @ 1.2 mL/min (constant flow)
Detector: MSD @ 230 °C, 45-450 amu
Sample: 1 % Rose oil in ethyl acetate

Two injections of rose oil are made, each using a different style of liner. One injection is made on a single taper liner without glass wool (Part No.: AG0-7516) and the other on a single taper liner with glass wool (Part No.: AG0-8175). All other GC parameters remain constant.

Results and Discussion

Inlet discrimination occurs when making a split injection of a mixture containing a wide range of molecular weights because the higher molecular weight compounds are slower to volatilize and enter the gas phase nearer to the split vent. This means that a greater proportion of these slow to volatilize compounds are swept out of the split vent than the lighter, more volatile compounds. However, this problem can sometimes be reduced by selecting a different style of liner for the analysis.

A liner without glass wool has increased sample discrimination because the location of volatilization is uncontrolled (Figure 1B). When making a liquid injection, the entire sample does not fully volatilize. Upon injection, the portion of the sample containing the more volatile components vaporizes quickly and enters the column. The remaining liquid makes contact with the bottom of the injection port liner, splatters, and then vaporizes. This non-homogenous volatilization causes the less-volatile components to be swept out the split vent increasing discrimination.

Better response is achieved in Figure 1A because of the deactivated glass wool near the top of the liner. Aside from forming a barrier to stop any solid particulates from getting onto the column, the glass wool has another important function: it provides additional surface area for volatilization. This feature improves vaporization for the less-volatile compounds giving a more consistent response for all analytes.

Both of the liners used in the experiment have a single taper at the bottom of the liner (Figure 2). One reason for the taper is to help focus the sample onto the column and reduce interaction with the base of the injection port. This extends the life of the gold seal and decreases inlet activity. However, the taper does not stop inlet discrimination, as volatilization of the high-boiling compounds

remains in close proximity to the split vent. Consequently, peak area and the signal-to-noise ratio are affected.

The chromatogram in Figure 1A shows an overall increase in response with the liner containing glass wool for all retention times. However as expected, the response of the later eluting compounds improves even more considerably. This is because later eluting compounds are less volatile and they are more impacted by inlet discrimination. The earlier eluting compounds also show an increase in response, but the increase in response is not as dramatic. This is because earlier eluting compounds are low-boiling and do not require glass wool to volatilize. Therefore, it can be concluded that using a liner containing glass wool yields better results for samples containing a wide range of boiling points.

Conclusion

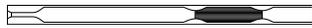
Injecting semi-volatile compounds with a split flow leads to inlet discrimination. As demonstrated, the negative effects from inlet discrimination can be greatly reduced by switching from a standard single taper liner to a single taper liner containing glass wool. Inherently, the liner containing glass wool stops solid particulates from entering the column and forces all semi-volatile compounds to volatilize early in the inlet. Thus, the single taper liner with glass wool is a more reliable choice for analysis of semi-volatile compounds.

Technical Tip

Did you know that many GC problems are related to improper column installation? To ensure the best chromatography, use the Cool-Lock™ Nut to get the proper installation depth. This patent pending tool will also help you to avoid burning your fingers while installing your column accurately in record breaking time. For more information, call your local Phenomenex GC Specialist.

Figure 2

Single Taper Liner w/Wool

Part No.	Description
AG0-8175	

Single Taper Liner

Part No.	Description
AG0-7516	

ORDERING INFORMATION

Single Taper Liner w/Wool

Part No.	Description	Unit
AG0-8175	GC Liner for Agilent (HP) Systems, 4 mm ID x 78.5 mm L x 6.3 mm OD	5/pk

Single Taper Liner

Part No.	Description	Unit
AG0-7516	GC Liner for Agilent (HP) Systems, 4 mm ID x 78.5 mm L x 6.3 mm OD	5/pk

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