2 Ways to Attain Sharper Peak Shape and Higher Sensitivity in Gas Chromatography

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There are two simple things that analysts can do in order to meet the method detection limits (MDLs) requirements. 1) The solvent focusing technique allows for greater sensitivity without sacrificing resolution. 2) Proper GC column selection is critical in order to get the best peak shape with the lowest baseline.

Introduction

Many Gas Chromatography (GC) analyses require very low detection of target analytes, such as drugs of abuse or environmental contaminants. However, GC analysts often struggle with attaining a method that is sensitive enough to meet the method detection limits (MDLs).

This technical note discusses two simple ways of attaining sharper peak shape and higher sensitivity so that you can achieve the required MDLs. The first strategy, solvent focusing, is a technique used by analysts when making on-column or splitless injections. The second strategy, proper GC column selection, is critical in order to ensure that the lowest baseline and sharpest peak is attained. Utilizing both of these methods will help provide the best results for GC methods with low MDL requirements.

Solvent Focusing

The purpose of solvent focusing is to get narrow peaks without doing a split injection. Attaining narrow peaks without splitting the sample is important because it allows for greater sensitivity without sacrificing resolution. By correctly setting the GC parameters, solvent focusing can be used to obtain narrow and symmetrical peaks for the majority of analyses.

Solvent focusing occurs via two mechanisms. The first mechanism occurs during splitless injections when a vaporized sample condenses on a cool column. Because the volume of a gas is much larger than that of a liquid, when the sample condenses, it is focused into a small area on the column. The second mechanism applies to both splitless and on-column injections and requires a ramped oven program. Unlike the first mechanism, both the solvent and the analytes must condense on the column upon injection. The condensed sample forms a “flooded zone” on the column’s surface. The flooded zone slowly decreases in size as the solvent evaporates, concentrating the analytes that are dissolved in the solvent (**Figure 1**). By the time all of the solvent has evaporated, the analytes will be focused into a tight band on the column. For both forms of solvent focusing to occur, either the analytes or both the solvent and the analytes must condense onto the column.

There are several parameters in a GC system which affect a sample’s rate of condensation (**Table 1**). These parameters include the initial oven temperature, the volatility of the solvent and analyte, and the phase ratio. In this technical note, we will be discussing how different GC parameters affect solvent focusing. We will also demonstrate how changing the initial oven temperature can easily provide improved chromatographic results with sharper peak shape.

GC Parameters Affecting Solvent Focusing

When doing solvent focusing, the initial oven temperature is the most convenient parameter to adjust. A good starting point is 50 °C below the boiling point of the earliest eluting analyte. This temperature should be held for the duration of the splitless hold time to ensure that the entire sample is focused onto the column.

The second GC parameter that affects solvent focusing is a column’s phase ratio. Unlike the initial temperature, the phase ratio can only be changed by installing a different column. The lower the phase ratio (greater film thickness), the greater the amount of solvent and analyte that can dissolve in the stationary phase. Working with a low phase ratio column may allow solvent focusing at temperatures that previously were not viable.

The third GC parameter that affects solvent focusing is the sample components. In most cases, the sample cannot be manipulated. However, when possible, solvent focusing is best achieved with a solvent that has the greatest difference between the solvent’s boiling point and the initial column temperature. For example, if the initial temperature is 30 °C, ethyl acetate (boiling point. 77.1 °C) will have a greater focusing effect than dichloromethane (boiling point 39 °C).

Figure 1.
Analyte distribution during a temperature programmed run. In Phase 1 the oven is set at a low initial temperature. In Phases 2 and 3 the oven ramps up causing the solvent to evaporate.

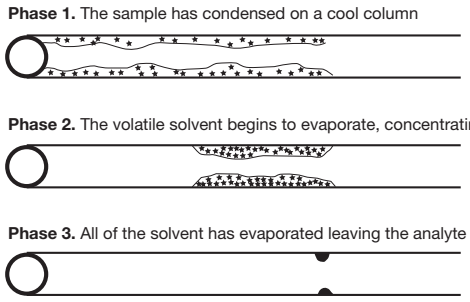


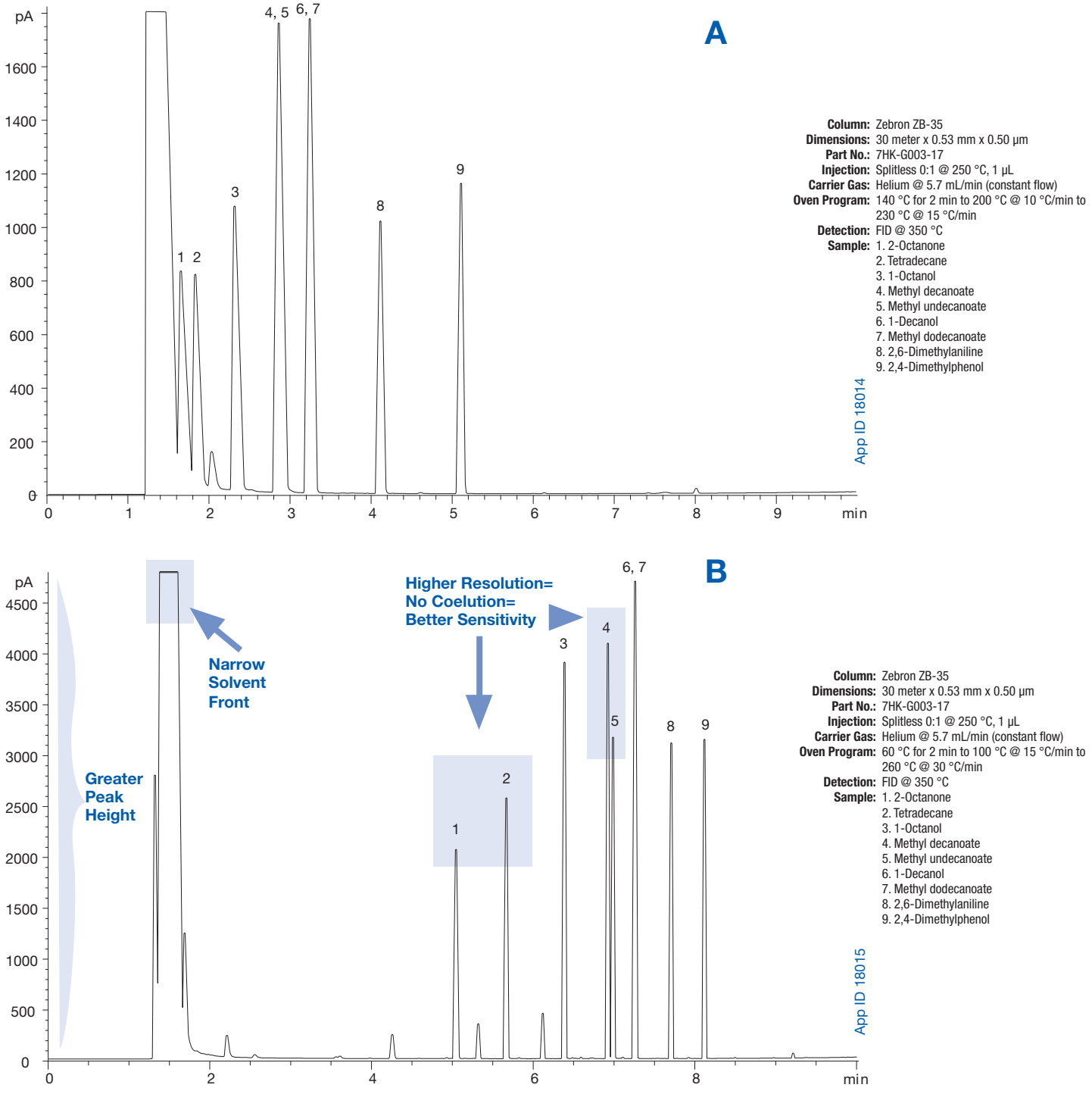
Table 1.
GC system parameters that affect solvent focusing.

GC Parameters	Affect on Solvent Focusing
Initial oven temperature	The lower the initial oven temperature, the greater the rate of sample condensation.
Phase ratio	The lower the phase ratio (greater film thickness), the greater the rate of sample condensation.
Volatility of the solvent and analyte	The higher the boiling point of the sample components, the greater the rate of condensation.

Experimental

Analysis of each sample was conducted on a HP6890 gas chromatograph (Agilent Technologies) using a Phenomenex Zebron ZB-35 GC column, 30 m x 0.53 mm ID x 0.50 µm. Additional parameters for the GC method are included in **Figure 2**.

Figure 2. Effects of changing initial temperature on solvent focusing. Solvent focusing was done on a hydrocarbon test mix using hexane as the solvent (boiling point 69 °C). A) Initial GC temperature of 140 °C. B) Initial GC temperature of 60 °C.



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Effects of Changing Initial Temperature on Solvent Focusing
Of the three parameters mentioned on the first page, changing the initial GC oven temperature is the easiest and quickest way to perform solvent focusing. **Figure 2** demonstrates solvent focusing of a hydrocarbon test mix using hexane as the solvent (boiling point 69 °C). By decreasing the initial temperature from 140 °C (**Figure 2A**) to 60 °C (**Figure 2B**), the peak width of each analyte was significantly decreased.

Proper GC Column Selection

When working on a method that requires high sensitivity and low detection limits, it is critical to select a GC column with low bleed and excellent deactivation procedures. Column bleed is the loss of lower molecular weight stationary phase pieces (MS Ions 355, 281, 207, 73) that are either the result of impurities in the starting polymer or the decomposition of the phase at elevated temperatures. It can result in an increased baseline, which can impact MDLs causing difficulties at the low calibration ranges.

Accurate analysis is also hard to attain if a column is not properly deactivated during the manufacturing process. Since the fused silica surface itself is highly active, manufacturers need to deactivate the glass in order to prevent any secondary interaction that might result in tailing or adsorption (loss of compound).

To safeguard against these potential problems, Phenomenex utilizes Engineered Self Cross-linking (ESC)™ technology in the Zebron GC line. The ESC bonding technology begins with special deactivation of the fused silica surface. Once the base polymer is carefully fractionated to eliminate low molecular weight impurities and enhances coating efficiencies, the columns are then cross-linked and surface bonded to the silica using an aggressive catalyst forming an interpenetrating network. This process provides enhanced column durability and extremely low bleeds level in MS.

Conclusions

There are two ways that GC analysts can implement to get sharper peak shape and higher sensitivity. The first, solvent focusing, is an effective method for obtaining narrow peaks. It is of particular value when low detection limits are required. Three parameters can effectively improve the technique: the initial column temperature, the column phase ratio, and the boiling points of the sample components. Of these three, changing the initial column temperature is the quickest way to effectively achieve solvent focusing and get narrower peaks and lower detection limits.

The second strategy, column selection, also plays a big factor in attaining high sensitivity and low detection limits. Selecting a column with low bleed and excellent deactivation procedures will ensure that you will get the most accurate and sensitive analysis possible. Utilizing these two methods together will help to ensure that you will get the best results for your analysis.

References

1. Grob, R.L. (1995). *Modern practice of gas chromatography*, (3rd edition): *Inlet Systems in GC* (pp.496). New York: John Wiley & Sons, Inc.

2. McNair, H.M. (1995). *Basic Gas Chromatography: Capillary Inlet Systems* (pp.99). New York: John Wiley & Sons, Inc.

Ordering Information for Zebron™ Products Referenced

Zebron ZB-35 GC Columns

ID(mm)	df(µm)	Temp. Limits °C	Part No.
30-Meter			
0.53	0.50	50 to 340/360	7HK-G003-17

Additional Zebron Products for MS Analysis

Zebron ZB-1ms GC Columns

ID(mm)	df(µm)	Temp. Limits °C	Part No.
10-Meter			
0.10	0.10	-60 to 360/370	7CB-G011-02
0.20	0.33	-60 to 360/370	7DE-G011-14
15-Meter			
0.25	0.25	-60 to 360/370	7EG-G011-11
0.32	0.25	-60 to 360/370	7EM-G011-11
20-Meter			
0.18	0.18	-60 to 360/370	7FD-G011-08
25-Meter			
0.20	0.33	-60 to 360/370	7GE-G011-14
30-Meter			
0.25	0.10	-60 to 360/370	7HG-G011-02
0.25	0.25	-60 to 360/370	7HG-G011-11
0.25	0.50	-60 to 360/370	7HG-G011-17
0.25	1.00	-60 to 360/370	7HG-G011-22
0.32	0.25	-60 to 360/370	7HM-G011-11
0.32	1.00	-60 to 360/370	7HM-G011-22
60-Meter			
0.25	0.25	-60 to 360/370	7KG-G011-11
0.25	1.00	-60 to 360/370	7KG-G011-22
0.32	1.00	-60 to 360/370	7KM-G011-22

Zebron ZB-5MSi GC Columns

ID(mm)	df(µm)	Temp. Limits °C	Part No.
15-Meter			
0.25	0.25	-60 to 360/370	7EG-G018-11
30-Meter			
0.25	0.25	-60 to 360/370	7HG-G018-11
0.25	0.50	-60 to 360/370	7HG-G018-17
0.32	0.25	-60 to 360/370	7HM-G018-11
0.32	0.50	-60 to 360/370	7HM-G018-17
60-Meter			
0.25	0.25	-60 to 360/370	7KG-G018-11

Zebron ZB-5ms GC Columns

ID(mm)	df(µm)	Temp. Limits °C	Part No.
10-Meter			
0.10	0.10	-60 to 325/350	7CB-G010-02
15-Meter			
0.25	0.25	-60 to 325/350	7EG-G010-11
20-Meter			
0.18	0.18	-60 to 325/350	7FD-G010-08
0.18	0.32	-60 to 325/350	7FD-G010-51
0.18	0.36	-60 to 325/350	7FD-G010-53
30-Meter			
0.25	0.25	-60 to 325/350	7HG-G010-11
0.25	0.50	-60 to 325/350	7HG-G010-17
0.25	1.00	-60 to 325/350	7HG-G010-22
0.32	0.25	-60 to 325/350	7HM-G010-11
0.32	0.50	-60 to 325/350	7HM-G010-17
0.32	1.00	-60 to 325/350	7HM-G010-22
60-Meter			
0.25	0.25	-60 to 325/350	7KG-G010-11
0.32	0.25	-60 to 325/350	7KM-G010-11

guarantee

If Zebron does not provide you with equivalent separations as compared to any other GC column of the same phase and comparable dimensions, return the column with your comparative data within 45 days for a FULL REFUND.

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