OPTIMIZING SENSITIVITY IN SPLITLESS INJECTIONS

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INTRODUCTION

Splitless injections are used for many applications in gas chromatography and are the preferred way to increase the sensitivity of an analysis because there are no modifications that need to be made to the instrument to use the technique. The problem with splitless injections is the associated increase in the width of the sample band, which can have an adverse affect on the quality of the chromatography.

WHAT IS SAMPLE BANDWIDTH?

In gas chromatography, bandwidth is a term used to describe the amount of time that it takes for an entire peak to be detected and is measured as the time between the beginning and end of the peak. In splitless injections, the width of solvent front is a good indication of the initial sample bandwidth (the initial width of each peak as it enters the capillary column). The shorter the solvent front, the narrower the bandwidth. As a rule, small initial bandwidths mean better peak shapes.

GOOD PEAK SHAPE IN SPLITLESS INJECTIONS

The length of time that the sample spends in the liner is critical. The longer it takes to transfer the sample to the capillary column, the larger the initial sample bandwidth will be. An approximate value for transfer time during splitless injections can be calculated by the following formula:

$$\text{Transfer Time (min)} = \frac{\text{Internal Volume of the Liner (mL)}}{\text{Column Flow (mL/min)}}$$

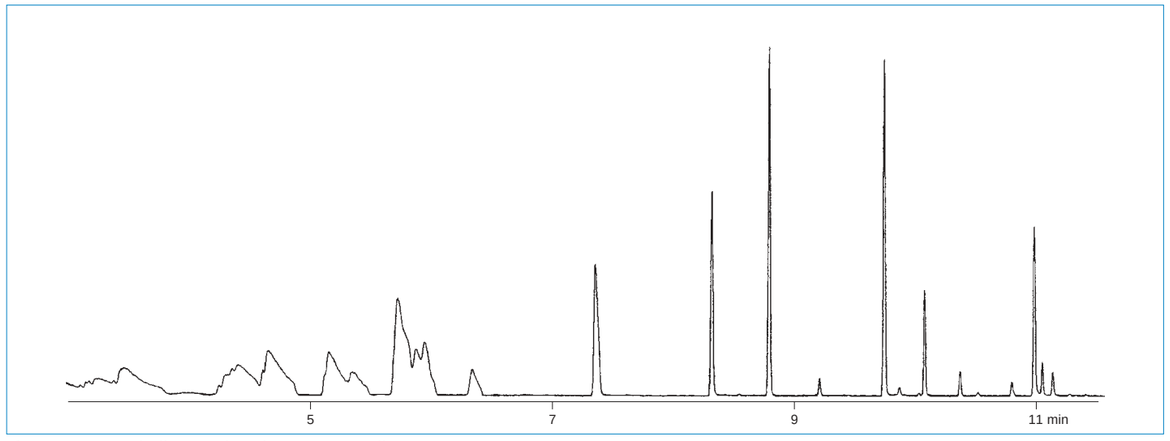

Figure 1. A volatile sample using a 4mm ID liner.

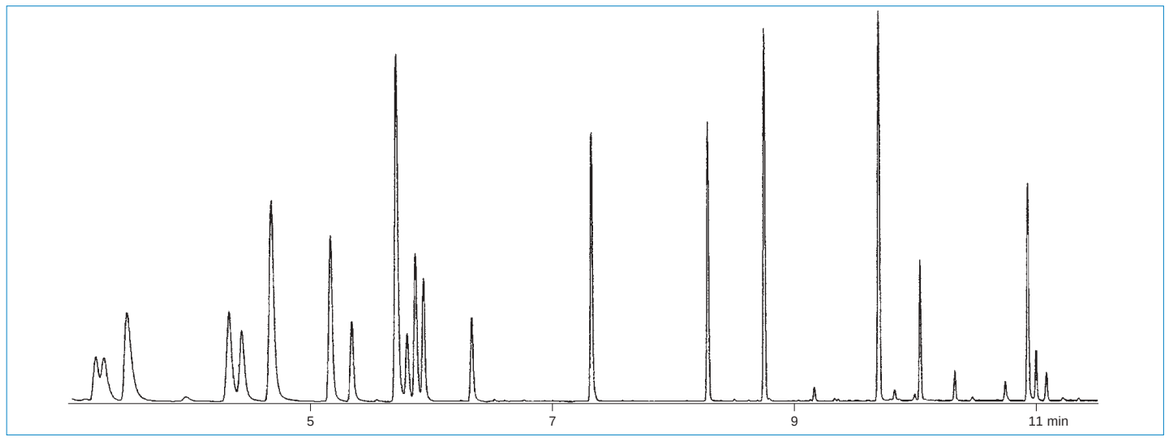

Figure 2. A volatile sample using a 2.3mm ID liner.

Figures 1 and **2** show the same mix of volatile compounds, but the resolution in **Figure 1** is very poor. The difference between the two chromatograms was the volume of the inlet liner and therefore the transfer time.

In **Figure 1** the liner volume was 800µL and the column flow was 1.8mL/min, this gives an approximate transfer time of 27 seconds. In **Figure 2** the liner volume was only 210µL. With the same column flow the transfer time is only 7 seconds, which, is almost 4 times faster than the transfer time in **Figure 1**. Difference in the quality of the chromatogram is obvious. The smaller 2.3mm ID liner causes a smaller initial bandwidth.

The capillary column also plays an important role in bandwidth minimization. If low initial oven temperatures are used the column will compress the initial sample band and minimize the band broadening caused by the injection port. When solvents are used to dissolve the sample, initial oven temperatures should be at least 30°C below the boiling point of the solvent. This will reduce the band broadening considerably. Using a thick film column will also have the similar effect.

CONCLUSION

The vaporization process that occurs in the injection port always causes band broadening of the sample. Minimizing the band broadening that occurs can go a long way to ensuring accurate and reproducible results. The approximate transfer rate of the sample to the column should be calculated whenever there is a problem, as reducing the transfer time often solves resolution problems. SGE has a range of liners with low internal volumes, such as the 2mm ID parallel and the 2.3mm ID FocusLiner™, that are designed to improve the peak shape of early eluting components in splitless injections.

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