Application Note No. 001

Large volume sample introduction in Capillary Gas Chromatography

Principles, Instrumentation and Applications

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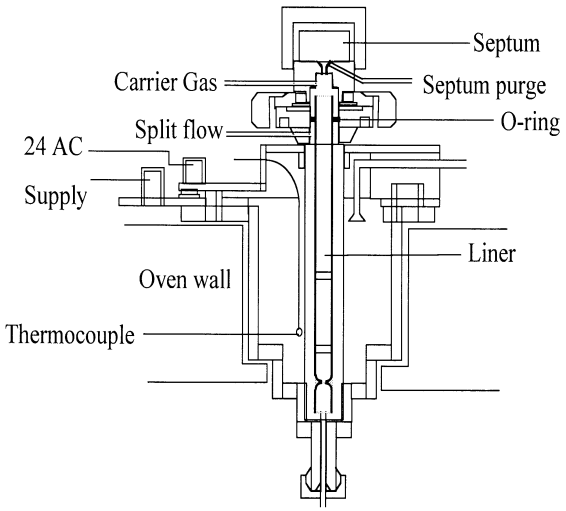
Introduction

Many samples ion analytical chemistry consist of trace components in a complex and interfering matrix. Identification and quantification of these components often requires the use of sample pretreatment. Moreover, preconcentration is often necessary in order to meet the required detection limits. The introduction of large sample volumes is an attractive means of improving the detection limits in capillary gas chromatography. In comparison with conventional preconcentration techniques, large volume sampling is generally more time and cost effective. The subject of this contribution is large volume sampling using programmed temperature injectors.
Programmed Temperature Vaporizer (PTV) injectors were developed by Vogt in the late seventies. A schematic drawing of the OPTIC PTV injector is given in figure 1.
The primary difference between conventional split/splitless injectors on the one hand and PTV injectors on the other is the temperature control. In PTV injectors the vaporization chamber can be heated or cooled rapidly. Bu introducing the sample at a low initial liner temperature many of the disadvantages of the conventional split-, splitless and on-column injection can be circumvented. For example, discrimination due to differences in boiling point can be minimized by introducing the sample in the liquid state into a cold liner and subsequently raise the temperature of the liner to the normal
temperature of a conventional hot injector.

Figure 1. Schematic diagram of the OPTIC injector



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Large volume sampling using PTV injectors

PTV injectors have proven to be easy to use and flexible inlet devices for capillary GC. Besides being used for hot and cold split and splitless injection, PTV injectors can also be used for on-line sample preconcentration by selective elimination of the solvent. In this mode of operation, a large volume of sample is introduced into the liner at a controlled speed. The initial inlet conditions are arranged so that the solvent is vented via the split line while the components are trapped and preconcentrated in the liner. At the end of the sample introduction process the split vent is closed and the liner is heated. In this way very large sample volumes up to 1 millilitre (!) have been introduced onto 320 µm i.d. capillary columns. Typical injection speeds are generally between 100 and 250 µl/min.
An example of a large volume sample introduction is given in figure 2. This figure shows the analysis of 250 µl of a hexane extract of water from Baikal Lake. 100ml of water was extracted with 2ml of hexane. Of this hexane sample 250 µl was injected at an injection rate of 80 µl per minute. The detection limits observed were in the low ppt range. From this example it is clear that the PTV injector is an excellent system for the introduction of large sample volumes in capillary GC. Due to the introduction of the large volume the detection limits could be improved more than 100 times.

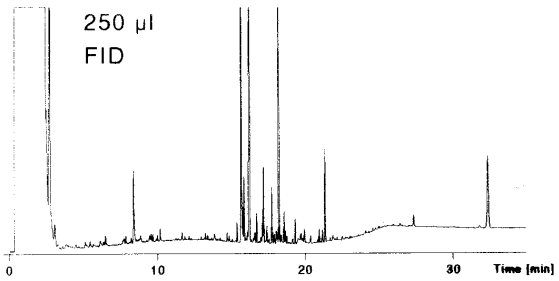

Figure 2. Large volume sampling: Analysis of 250µl of a hexane extract (1:50) of Baikal Lake water.

Two other examples of large volume sampling are given in Figures 3 and 4. Figure 3 shows the analysis of very low concentrations of PAH's in water. Also here the water was first extracted with hexane. 250µl of the extract was introduced into the GC at a rate of 80µl/min. The initial liner temperature was 0°C. The recoveries of fluorene, phenanthrene and pyrene were 100%. The recovery for the relatively volatile β -methyl-naphthalene was only 78% due to incomplete trapping of this component in the PTV liner.

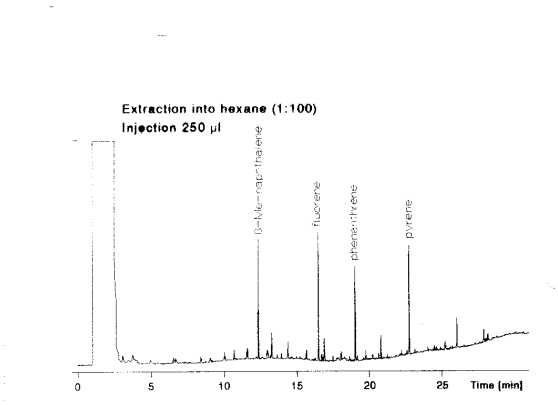

Figure 3. Extraction and large volume sampling of PAH's Extraction from water with hexane. Concentration level: 0.2 ppb in water. Sample volume: 250µl

Figure 4 shows the large volume sampling of a number of relatively volatile priority pollutants. Here a liner packed with Tenax was used to minimize the loss of volatiles. In this way quantitative recovery was obtained for all components.

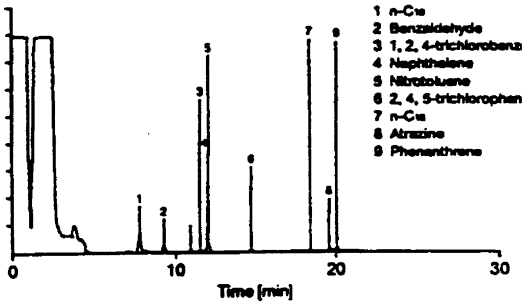

Figure 4. Large volume injection of a 20 ppb solution of priority pollutants in hexane. Injector: 30°C (2.7 min), 12°C/s, 330°C. Injection rate: 100µl/min. *Adsorption material: Tenax*

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Conclusions

Programmed temperature injectors are very flexible sample introduction systems for capillary gas chromatography. PTV injectors hold a remarkable promise for large volume sample introduction. Sample volumes up to 1000µl can be introduced with an injection speed up to 250µl per minute. Large volume sampling is an effective means of improving the detection limits in capillary gas chromatography. Moreover, it can help to improve the reliability of quantitative analysis and to reduce the total analysis time.

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