Improved Detection from Sample Concentration

Enrichment and Desorption Unit Sample Concentrator with the 490-GC Micro-GC

Advantage Statement: Sample detection limits can be markedly improved if analytes are concentrated before they are injected into the Varian 490-GC Micro GC. Using the Enrichment and Desorption Unit (EDU) Sample Concentrator, the sample is adsorbed onto a porous medium. These trapped analytes are then desorbed and transferred to the Micro-GC for separation and analysis. Using this approach, enrichment can be increased by more than 230 times.

Introduction

In general, the typical detection limit of the 490-GC is ~ 1-10 ppm and is compound dependent. Detection limits can be improved by concentrating the sample prior to introduction to the Micro-GC by the method outlined above.

Instrumentation

Figure 1 EDU-Varian Sample Concentrator and 490-GC Micro-GC.

490-GC, single channel (CP-Sil 5 CB GC column, 4 m)
EDU-Varian Sample Concentrator
Adsorption Material: Tenax®
Software: Galaxie™ Chromatography Software for GC control and data handling. EDU software for control of the sample concentrator.

Principle of Operation

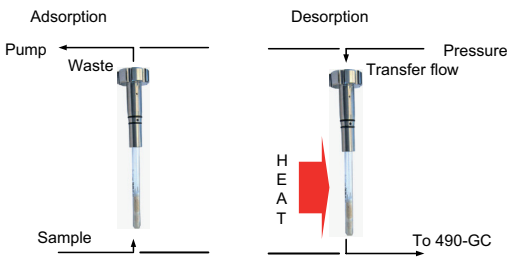

Figure 2 Principle of operation of the VarianEnrichment and Desorption Unit.

Sampling Phase
The sample is pumped from its container (Tedlar® bag) through a heated sample line onto the adsorption tube.

Desorption Phase
The adsorption tube is heated in "stop" flow.

Injection Phase
The adsorption tube is set in "back flush" mode and the "desorbed" sample components flow through a heated transfer line to the Micro-GC.

Materials and Reagents

BTEX Gas Sample Containing (balance nitrogen)

Benzene	1.42 ppm
Toluene	1.46 ppm
Ethylbenzene	1.59 ppm
o-Xylene	1.63 ppm
m-Xylene	1.53 ppm
p-Xylene	1.72 ppm
Propylbenzene	1.38 ppm
i-Butylbenzene	2.35 ppm
Butylbenzene	2.79 ppm

Conditions

490-GC Micro-GC

Carrier Gas:	Helium, 150 kPa
Column Temp:	100 °C
Inj Time:	255 ms
Flow Mode:	Continuous
GC Sampling Time:	Optimized

EDU-Varian Sample Concentrator

Transfer Gas:	Helium, 85 kPa
Sampling:	60 s, 30 °C
Desorption Phase:	120 s, 180 °C
Inj Phase:	30 s, 180 °C
Clean Phase:	90 s, 220 °C
Cool Phase:	100 s

Results and Discussion

Using the conditions listed above, the chromatogram in Figure 3 was obtained.

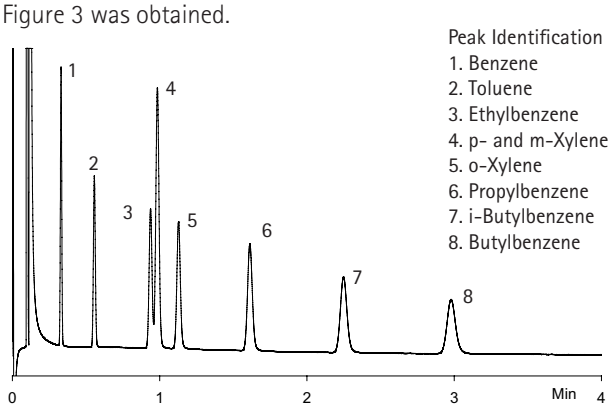

Figure 3 BTEX analysis using the Varian sample concentrator and 490-GC.

For each application the system requires some method optimization, particularly the transport delay of sample from EDU-Varian to the 490-GC Micro-GC, and the adsorption trap material and capacity.

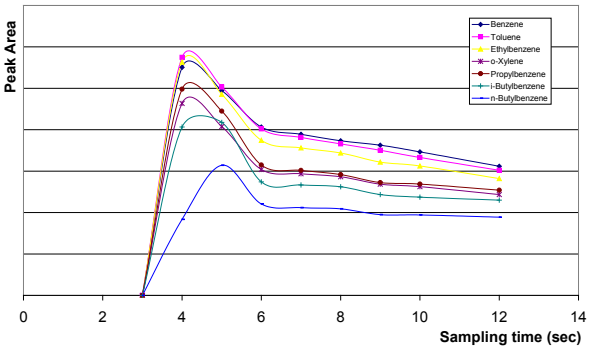

Figure 4 Sample transport through the transfer line.

Figure 4 shows the measured peak area for selected components as a function of the GC sampling time. Note that prior to a sampling time of 4 seconds, no component peak areas were measurable by the 490-GC. After a sampling time of 4 to 5 seconds, an increase could be seen in the amount of component peak area measured. However, at ~ 7 seconds and beyond, the individual peak area values "flatten" out considerably, which adds to the robustness of the method. The desorption of the components from the adsorption tube is in back flush mode. The concentration of the components in the desorption profile is dependant on the volatility/boiling point of the component and its affinity for the adsorption material.

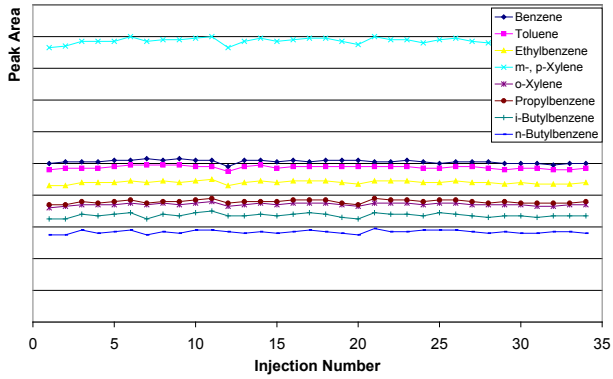

Figure 5 Repeatability results over 34 successive injections.

Figure 5 shows that the repeatability for 34 successive analyses ranges from 1.0 to 1.9%, with a sampling time of 7 seconds. It was also noted that the use of a shorter sampling time (5 seconds) resulted in RSD values (1.2 – 3.6%) that were slightly lower but with slightly higher sample enrichment. As with most methods, the analyst must strike a balance between the desired detection levels and required level of performance.

Another important factor in application/method optimization is to determine the capacity of the adsorption material. To assess this, the 1 ppm sample was drawn through the adsorption tube for varying times at a fixed pumping rate (450 mL/min), desorbed at a fixed heating value/time onto the 490-GC and peak areas measured (Figure 6).

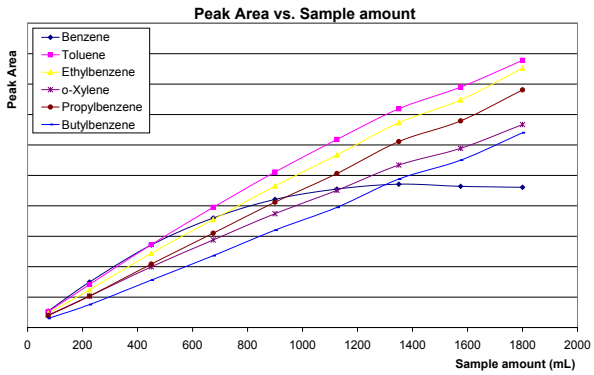

Figure 6 Sampling linearity and breakthrough.

As can be seen from Figure 6, the breakthrough volume varies considerably and ranges from benzene at 700 mL to 1400 mL or greater for toluene.

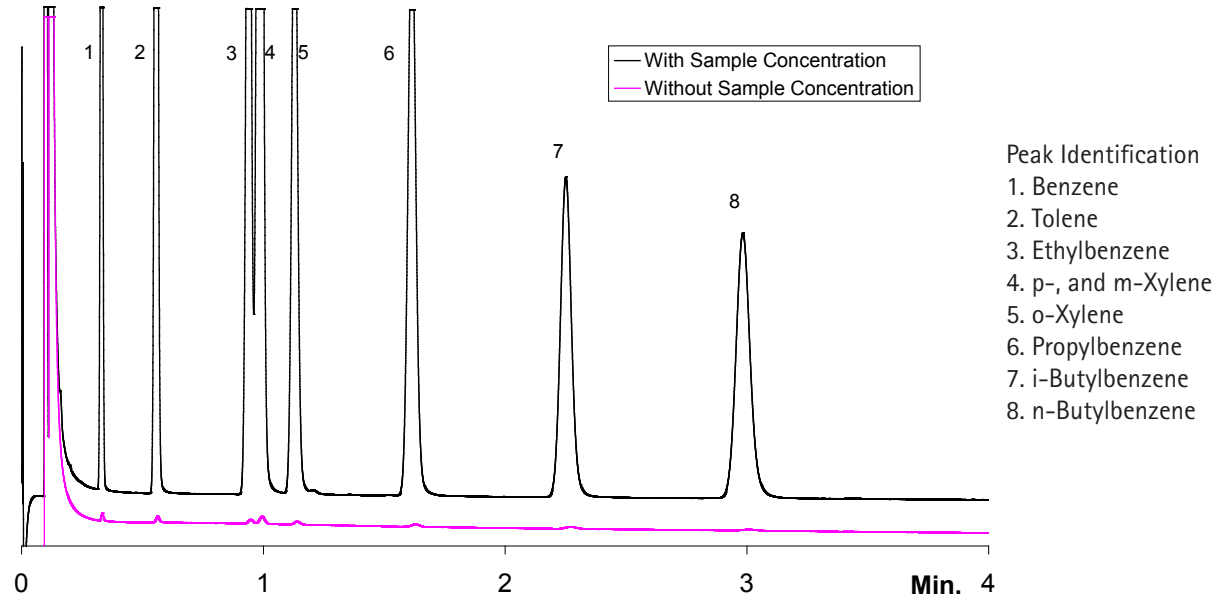

Figure 7. Enrichment Details.

To determine the enrichment factors, the chromatogram results produced from the 1 ppm sample, after direct injection, were compared with the results generated using analysis with sample concentration (1800 mL total flow). The results have been overlayed in Figure 7. The actual sample enrichment factor varied component to component, as illustrated in Tables 1 and 2.

Table 1 Enrichment factors.

	Benzene	Toluene	Ethylbenzene	Propylbenzene	Butylbenzene
With EDU	23.05	43.90	42.60	39.05	32.00
Without EDU	0.16	0.19	0.18	0.17	0.14
Enrichment Factor	146	237	234	230	226

Table 2 Summary results.

	Benzene	Toluene	Ethylbenzene	Propylbenzene	n-Butylbenzene
Original Conc. (ppm)	1.42	1.46	1.59	1.38	2.79
RSD (%)	1.08	1.01	1.12	1.41	1.83
RSD (ppm)	0.015	0.015	0.018	0.019	0.051
Enrichment Factor	146	237	234	230	226
Approx Det. Lim. (ppb)	7	4	4	4	4

Conclusions

The experimental data clearly show that routine detection levels can be significantly improved when the 490-GC Micro-GC is used with sample concentration provided by the Varian Enrichment and Desorption Unit prior to analysis.

Enrichment factors as much as 230 x or more can be obtained depending on the sample component and chromatographic conditions. In this case, the detection limit was improved from 1 ppm (without sample concentration) to 5 ppb with sample concentration for several aromatic compounds. For benzene, the enrichment factor, approximately 150, was the lowest because this component was sampled beyond the breakthrough volume of the trap. With these settings enrichment factors from 150 to 230 were reached.

RSDs of peak area were below 2%, indicating that the system stability and repeatability was quite good.

The EDU-Varian Sample Concentrator and 490-GC Micro-GC provide an excellent solution for achieving much lower detection limits when conducting fast GC analysis.

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