Fast Analysis of Low Level BTEX using the Agilent Micro GC with Sample Concentrator

Application Note

Micro Gas Chromatography, Mobile Measurement, Environmental Analysis

Authors

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Abstract

Detection limits can be improved if analytes are concentrated before being injected onto the Agilent 490 Micro GC. Using the Enrichment and Desorption Unit (EDU) sample concentrator, the sample is adsorbed onto a porous medium. The trapped components are then desorbed and transferred to the Micro GC for separation and detection. This application note shows an analysis of BTEX using the EDU sample concentrator, resulting in decreased detection limits by more than 140 to 200 times.

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Introduction

In general, the typical detection limit for the TCD used in the Agilent 490 Micro GC is approximately 1–10 ppm and is compound and column dependent. These detection limits can be improved by concentrating the sample prior to introduction to the Micro GC through the use of an adsorption technique in which the sample is passed over/through a porous media where the constituent analytes in the sample are trapped. The trapped components are then desorbed by heating the trapping material and transferred to the Micro GC for separation and analysis.

Instrumentation, Conditions and Sample Information

For this application note, a dual channel cabinet 490 Micro GC equipped with a single column channel was used. The EDU sample concentrator was used for sample enrichment. Conditions and sample information are found in Tables 1 and 2.

- Agilent 490 Micro GC equipped with
- 4 m CP-Sil 5 CB column channel
- EDU Sample Concentrator with
- Tenax TA adsorption tube
- Agilent EZChrom 3.3.2 for Micro GC control and
- EDU software for control of the sample concentrator

Table 1. Typical Conditions

Agilent 490 Micro GC	
Column	CP-Sil 5 CB, 4 m
Column temperature	100 °C
Carrier gas	helium, 150 kPa
Injector temperature	110 °C
Injection time	255 ms
Sample flow mode	continuous
Micro GC sampling time	optimized, 7 s
Sample line temperature	110 °C
EDU – Sample concentrator	
Transfer gas	helium, 85 kPa
Sampling	240 s, 450 mL/min, 30 °C
Desorption	120 s, 180 °C
Injection	30 s, 180 °C
Cleaning	90 s, 220 °C
Cooling	100 s

Table 2. Sample Information

Component	Concentration (ppm)
Nitrogen	Balance
Benzene	1.42
Toluene	1.46
Ethylbenzene	1.59
m-Xylene	1.63
p-Xylene	1.53
o-Xylene	1.72
Propylbenzene	1.38
i-Butylbenzene	2.35
n-Butylbenzene	2.79

EDU Sample concentrator - Principle of Operation

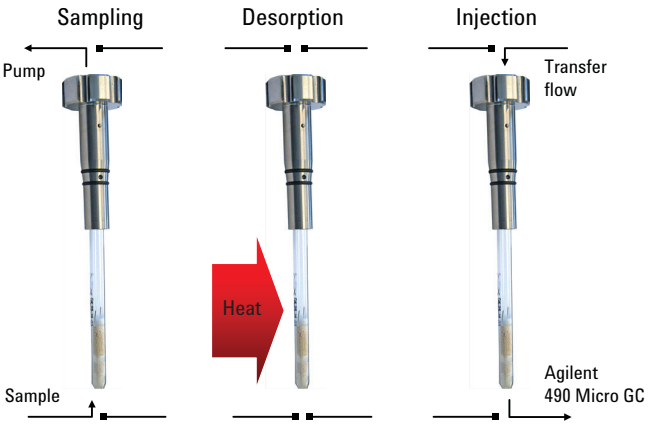

Figure 1. EDU sample concentrator - principle of operation.

During sampling the gas sample is pumped for the programmed time and flow from its container (tedlar bag) or ambient air through a heated sample line onto the adsorption tube, Figure 1. In the next step, the desorption phase, the adsorption tube is heated in 'stop flow' mode. To perform the injection, the adsorption tube is set in backflush mode and the components flow through a heated transfer line to the Micro GC. For cleaning the instrument, the adsorption tube is rinsed with zero gas and heated to a high temperature. After cooling to the given sampling temperature, the instrument is ready for the next enrichment.

Results and Discussion

The chromatogram, shown in Figure 2 is obtained using the instrumentation and conditions shown in Table 2 and Figure 1. Analysis time is just over 3 minutes for separation of all components of interest. Run-to-run time, including the EDU cycle time, is approximately 10–12 minutes.

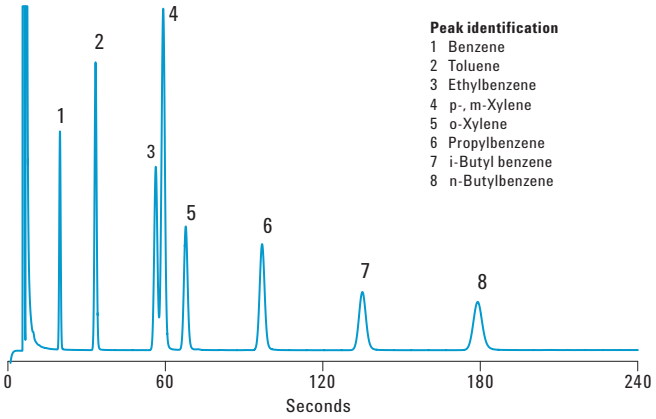

Figure 2. Chromatogram for BTEX analysis on a CP-Sil 5 CB column.

For each application, the system requires some method optimization; of particular note are:

- Transport delay of the sample from the EDU sample concentrator to the Micro GC
- The adsorption trap material and trap capacity

Figure 3 shows peak area for selected components as a function of the Micro GC sampling time. Note that prior to a sampling time of 4 seconds no peak areas were measured by the Micro GC. After 4 to 5 seconds sampling time, an optimum peak area amount was obtained for all components measured. However, at approximately 7 seconds and beyond, the individual peak area values flatten out considerably and add to the robustness of the method.

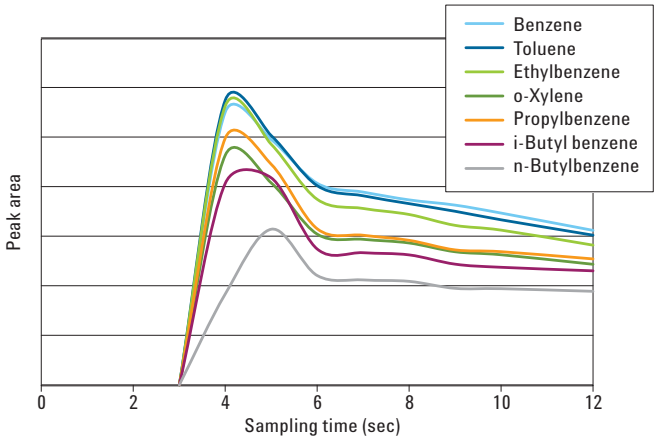

Figure 3. Sample transport time from EDU to Agilent 490 micro GC.

Using an optimal Micro GC sampling time, in this case 7 seconds, a repeatability experiment was conducted. Figure 4 shows that the repeatability for 30 successive analyses ranges from 1.0 and 1.9%. Note that the use of a shorter sampling time (5 seconds), resulting in slightly higher peak areas, show slightly worse RSD values (1.2 – 3.6%). As with most methods, the analyst must strike a balance between the desired detection levels and the required performance level.

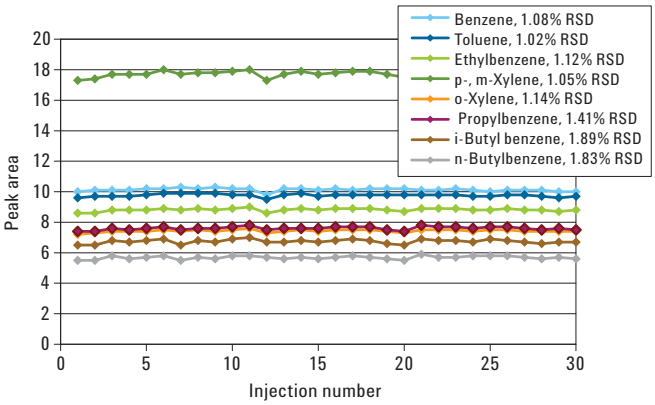

Figure 4. Repeatability results – 30 successive injections.

Another important function of method optimization is to determine the capacity of the adsorption material. To assess this, a 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 Micro GC and then, measured the peak areas. As shown in the graph in Figure 5, the breakthrough volume varies considerably and ranges from benzene 700 mL to 1,400 mL or greater for toluene and the other components.

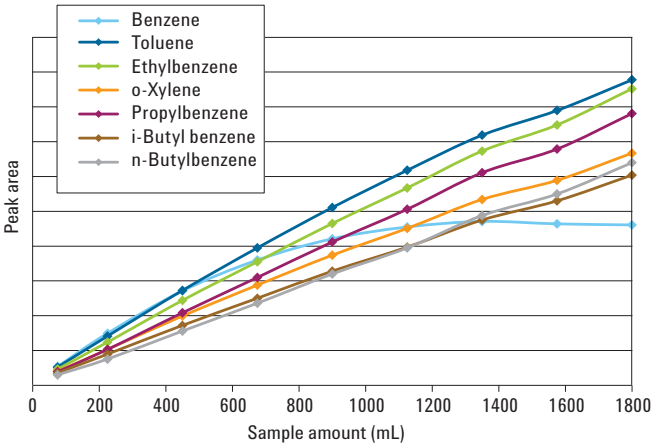

Figure 5. Sampling times – breakthrough of adsorption tube.

To determine the enrichment factors, the results from a low ppm sample (1–2 ppm, see Table 2) of a direct injection, are compared with the results generated using the sample concentration (1,800 mL total flow); an overlay chromatogram is displayed in Figure 5.

The actual sample enrichment factor varies from component to component as illustrated in Table 3. For benzene, the enrichment factor, approximately 150, is the lowest component in this example, sampled beyond the breakthrough volume of the trap. With these settings, enrichment factors, calculated from the peak areas of a regular analysis and the analysis with sample concentration, ranges from 150 to 240. The limits of detection are estimated at approximately 4 to 7 ppb, compared to 1–2 ppm without the use of sample enrichment.

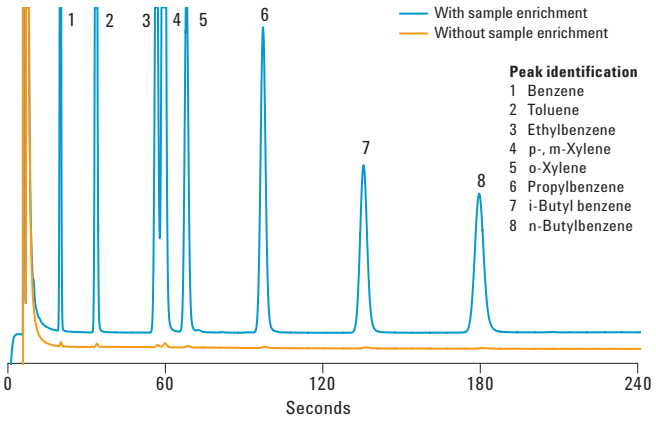

Figure 6. Overlay of 1 ppm level chromatograms - direct analysis and with sample enrichment.

Table 3. Enrichment Factors and Estimated Detection Limit

Component	Original concentration (ppm)	Enrichment factor	Estimated detection limit (ppb)
Benzene	1.42	146	7
Toluene	1.46	237	4
Ethylbenzene	1.59	234	4
m & p-Xylene	3.16	243	4
o-Xylene	1.72	192	5
Propylbenzene	1.38	230	4
i-Butylbenzene	2.35	171	6
n-Butylbenzene	2.79	226	4

Conclusions

The experimental data depicted in this application note clearly show routine detection levels can be significantly improved with the Agilent 490 Micro GC if a sample concentrator is used prior to injection on the gas chromatograph.

The EDU sample concentrator in combination with the 490 Micro GC provides a tool for achieving much lower detection limits when conducting fast GC analysis. Enrichment factors as much as 240 times can be obtained, depending on the sample component and chromatographic conditions. In this case, the detection limit was improved from 1–2 ppm (without sample concentration) to approximately 4 to 7 ppb with sample concentration for several aromatic compounds.

Repeatability determined at below than 2% RSD for peak area indicating that the systems' stability and repeatability is very good.

Agilent delivers lab quality instrumentation when and where you need it. The small form factor of both the 490 Micro GC and the EDU Sample Concentrator, and the low gas consumption of these instruments, makes it an ideal solution for analysis of low level organic gas components with mobile laboratories.

For More Information

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