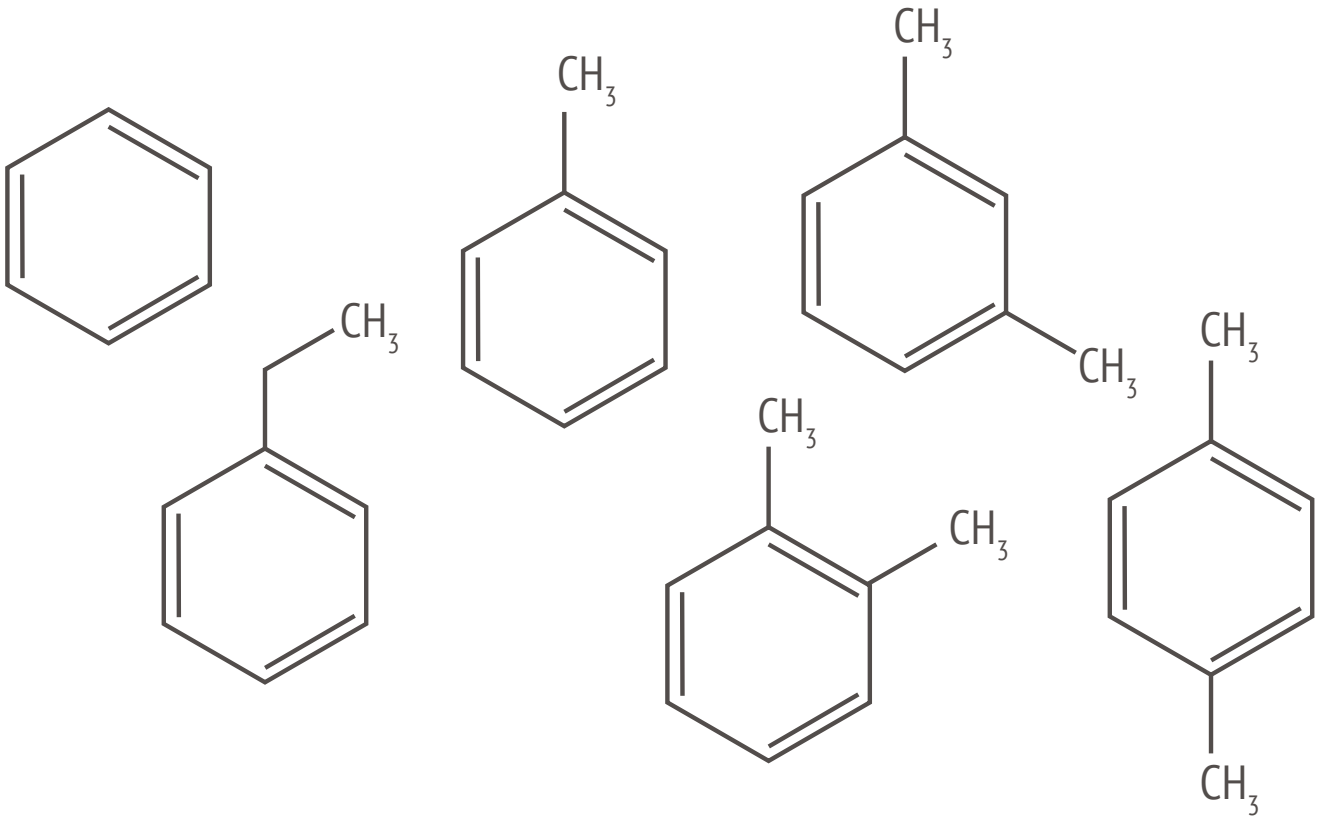
Ellutia
Chromatography Solutions



Application Report

BTEX Separation on the Ellutia 200 FID GC

Ellutia
Chromatography Solutions

Application Report

Company:	Element Materials Technology
Address:	Wilbury Way, Hitchin, SG4 0TW
Contact Name:	Nickie Smith
Customer PO:	
Telephone:	01462 427 856
Fax:	01462 427 851
email:	nickie.smith@element.com
Analysis Date(s):	15/07/14 to 17/07/14
Analyst:	Dr. Mark Landon
Instrument 1:	200 Series GC (FID)
Instrument 2:	
Ellutia Ref:	1406

BTEX Separation on the Ellutia 200 FID GC

GC Conditions

Injector Temperature (°C):	250
Liner Type:	Focus Liner With Wool
Carrier Gas Type:	Hydrogen
Carrier Gas Control Method:	Programmed Pressure
Initial Pressure (psi)	2.5
Upper Pressure (psi @ T)	5.0 @ 70
Upper Pressure (psi @ T)	6.0 @ 200
Split Flow (ml min^{-1})	4
Injection Volume (ml)	0.2
Column Type	Wax
Column Length (m)	30
Column Internal Diameter (mm)	.53
Column Film Thickness (µm)	1.0
Column Temperature Program:	
Initial Temperature (°C)/Hold Time (min)	45/6
Temperature Ramp 1 (°C min^{-1})	4
Column Temperature 1 (°C)/Hold Time (min)	70/0
Temperature Ramp 2 (°C min^{-1})	20
Column Temperature 2 (°C)/Hold Time (min)	200/1
Detector Temperature (°C)	250
Detector Range	x10
Detector Flows:	
Hydrogen (ml min^{-1})	30
Air (ml min^{-1})	300

Samples were prepared by the customer containing 5, 1 and 0.1 g/l of each BTEX in a matrix of heptane, cyclohexane and trimethylbenzene. Initial experiments into the separation of components using a purchased BTEX standard where performed on wax columns employing a thinner film thickness. These were found to give separation of the ethylbenzene, p-xylene and m-xylene, but didn't produce full baseline separation. Once a suitable method employing a thicker film column had been produced, work was performed on the customer's low standard. A 0.5 l liquid injection sample gave huge peaks using the above conditions which caused slight overloading of the column, broader peaks and lost resolution (especially of the ethylbenzene, p-xylene and m-xylene).

As the quantities of the components were high, the decision was made to take headspace samples for injection. A 1 ml aliquot was placed in a 20 ml headspace vial and held at 30°C for ten minutes. A 0.2 ml gas aliquot was taken from half way down the vial and injected into the GC. The chromatogram of the headspace injection is shown in Figure 1.

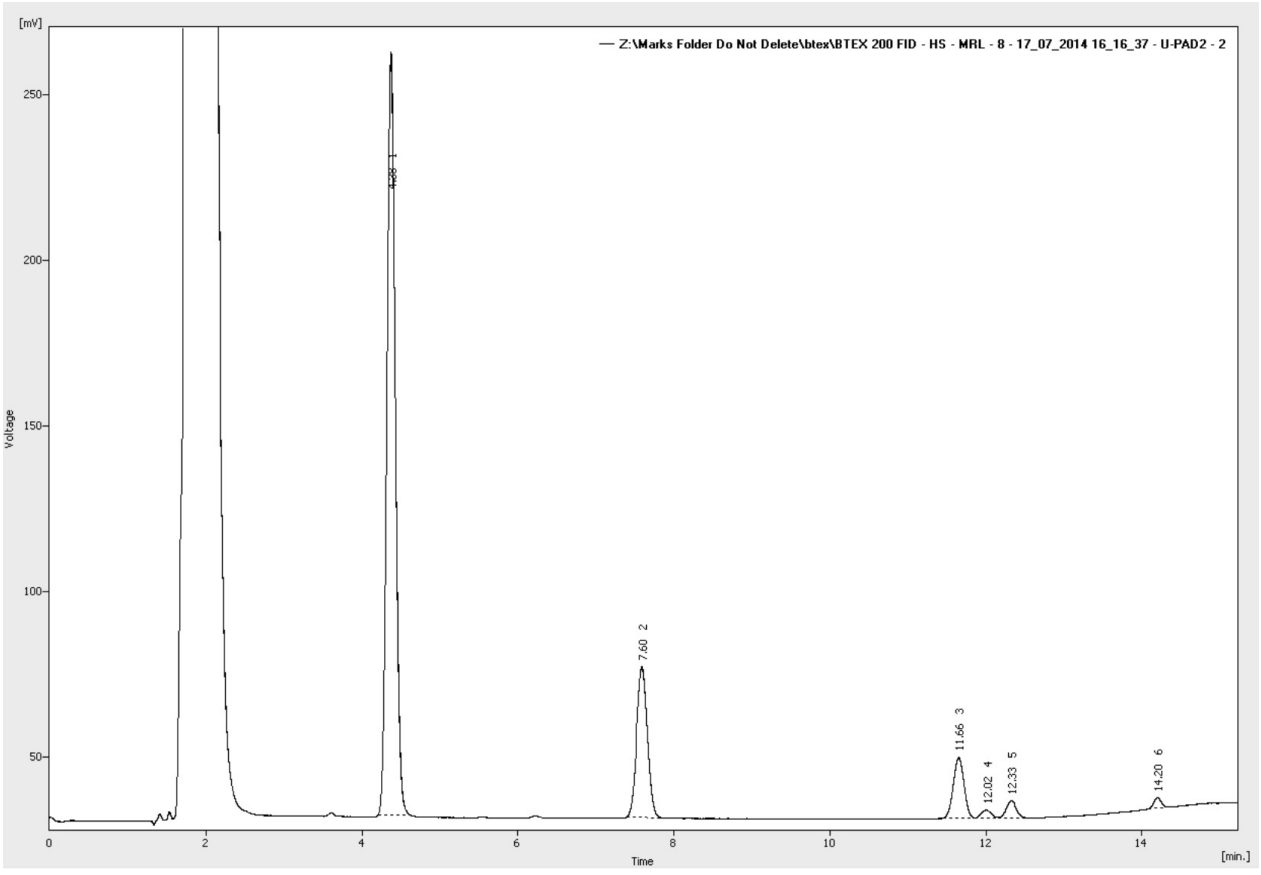


Figure 1: 0.2 ml headspace injection of 0.1 g/l BTEX standard, peak number: 1. benzene, 2. toluene, 3. ethylbenzene, 4. p-xylene, 5. m-xylene and 6. o-xylene

The six peaks relating to the BTEX components are labelled with the large solvent peak (unlabelled) at the beginning of the chromatogram. Even though peaks four through six appear small, the noise level of the baseline was very low with 0.0664 mV. Therefore, the smallest peak obtained (relating to p-xylene) possessed a height of 2.354 mV showing potential detection limits of at least 10-fold less, under these chromatographic conditions.

As the benzene peak was of considerable size in the lowest standard, the higher standards would have caused saturation of the phase and detector under the same conditions. As we can comfortably see the lower standard, the need to test the higher levels was seen to be unnecessary, as methods for reducing amount of sample to the GC are common. Some common examples include injecting a lower volume and increasing the split flow.

Towards the end of the run (after the elution of o-xylene), the column was cleaned by ramping the oven to remove the trimethylbenzene, which possesses a longer retention time (Figure 2.) than the BTEX compounds. The heptane and cyclohexane were eluted together early in the chromatogram.

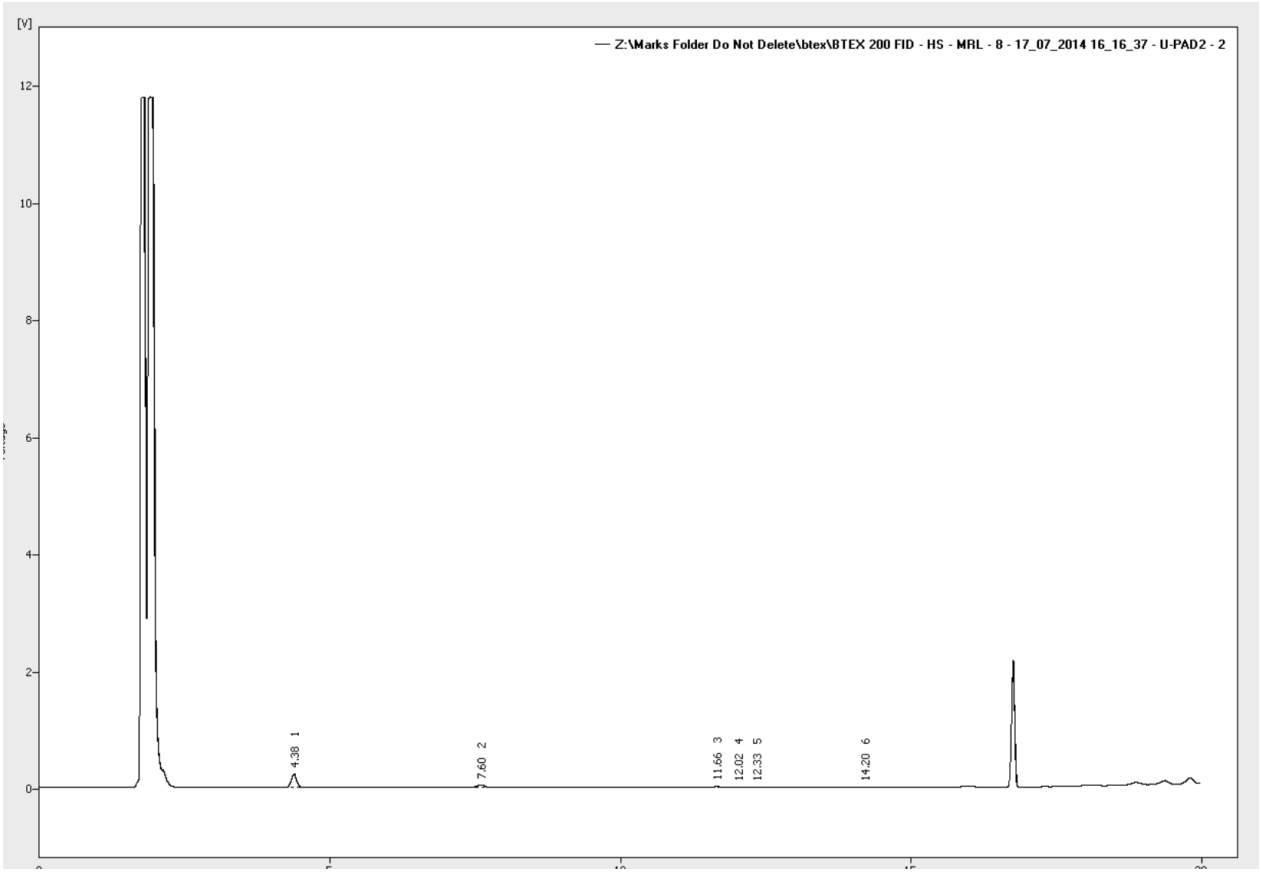


Figure 2: Expanded view of chromatogram showing large peaks before and after components of interest relating to solvent matrix.

Conclusion

Separation of the BTEX components can be performed on the Ellutia 200 Series GC, fitted with an FID. The matrix components do not interfere or co-elute with the compounds of interest. The full baseline separation of all the xylene isomers can be performed on the wax column and is more efficient with thicker films.

Ellutia
Chromatography Solutions

To learn more about the Ellutia 200 Series GC, please scan the QR code below or visit https://www.ellutia.com/200-series-gas-chromatograph

Colston House, 200 Lancaster Way Business Park, ELY, Cambridgeshire, CB6 3NX, UK
Tel: +44 (0)1353 669916 Web: www.ellutia.com
Ellutia Limited Registered in England Number 2967460
Registered Address Colston House,200 Lancaster Way Business Park, Ely, Cambridgeshire, England, CB6 3NX

SUBSCRIBE CONNECT SOLVE

MADE IN BRITAIN®