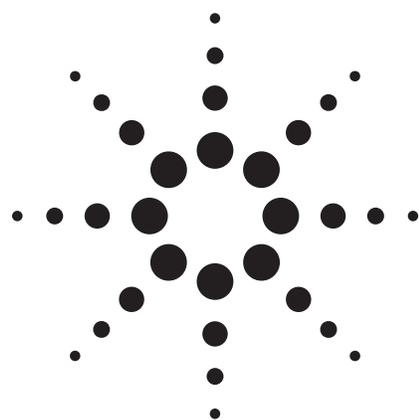


Fast Analysis of Aromatic Solvent with 0.18 mm ID GC column



Application

Gas Chromatography

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Abstract

Fast GC is one possible way to improve productivity. By reducing the internal diameter of the capillary column, a higher efficiency per unit of column length is obtained in capillary GC. Combined with shorter column length, the application of high-efficiency 0.18-mm-id GC column results in faster analyses compared to conventional 0.25-mm- or 0.32-mm-id columns without losing measurement performance. A single, rapid GC method for aromatic solvent purity analysis is described.

Introduction

Determination of the purity of aromatic hydrocarbons is critical for many QA and QC laboratories in the chemical and petrochemical industry. In an effort to standardize analysis procedures, the American Society of Testing and Materials (ASTM) has developed and published a number of GC methods specifically for an aromatic compound or a class of aromatic compounds such as styrene, o-xylene, p-xylene, and ethylbenzene. Table 1 lists 10 ASTM methods along with the recommended columns and specifications [1].

Many QA/QC labs need to run these different ASTM methods to ensure the quality of all products. These analyses can be difficult and expensive to perform. Because many of these ASTM methods are remarkably alike, it is highly desirable to develop a single method that is the chromatographic equivalent of the individual methods. Detailed discussions on a unified aromatic solvent method are available in the literature [2, 3].

Due to demands for increased productivity, many QC/QA laboratories need to analyze large numbers of samples every day. Faster analysis is highly desirable for increased sample throughput and therefore lower cost per sample.



Table 1. Ten ASTM Methods for the GC Analysis of Aromatic Solvents

| ASTM Method | Title | Liquid phase | Column type | Report specifications |
|-------------|---|------------------------------|------------------------------------|---|
| D2306 | Std test for C8 aromatic hydrocarbons | 0.25 μ m Carbowax | Capillary 50 m \times 0.25 mm | wt% of individual C8 HC |
| D2360 | Std test for trace impurities in monocyclic hydrocarbons | 0.32 μ m Carbowax | Capillary 60 m \times 0.32 mm | wt% of individual aromatic impurities, total impurities, purity |
| D3760 | Std test for cumene | 0.25 μ m Carbowax | Capillary 50 m \times 0.32 mm | wt% of individual impurities, cumene purity (wt%) |
| D3797 | Std test for o-xylene | 0.5 μ m Carbowax | Capillary 60 m \times 0.32 mm | wt% of individual impurities, o-xylene purity (wt%) |
| D3798 | Std test for p-xylene | 0.25 μ m Carbowax | Capillary 50 m \times 0.32 mm | wt% of individual impurities, total impurities, p-xylene purity (wt %) |
| D4492 | Std test for benzene | 0.25 μ m Carbowax | Capillary 50 m \times 0.32 mm | wt% of individual impurities, benzene purity (wt%) |
| D4534 | Std test for benzene in cyclic products | 10% TCEPE on Chromasorb P | Packed 3.7 m \times 3.175 mm | wt% of benzene |
| D5060 | Std test for impurities in ethylbenzene | 0.5 μ m Carbowax | Capillary 60 m \times 0.32 mm | wt% of individual impurities, ethylbenzene purity |
| D5135 | Std test for styrene | 0.5 μ m Carbowax | Capillary 60 m \times 0.32 mm | wt% of individual impurities, styrene purity |
| D5917 | Std test for trace impurities in monocyclic hydrocarbons (ESTD Cal) | 0.25 μ m Carbowax | Capillary 60 m \times 0.32 mm | wt% individual impurities, wt% total nonaromatics, wt% total C9 aromatics, purity of main component |

Experimental

High-Efficiency Capillary GC Columns

Efficiency is often related to the number of theoretical plates, which increases linearly with decreasing column internal diameter (id). For instance, 0.18 mm id columns typically produce 5,800 to 6,600 theoretical plates per meter, whereas columns with 0.25 to 0.32 mm id typically produce 3,600 to 4,600 plates per meter. The efficiency improvement for the 0.18 mm id columns allows for better signal-to-noise ratios. Since decreasing the internal diameter results in an increase of the column efficiency per meter, the column length can be reduced while keeping the resolution constant. Therefore, the use of 0.18 mm id columns, also known as the high-efficiency GC columns, can help gas chromatographers substantially reduce their sample analysis time.

While it is true that an even smaller id column, such as 0.1 mm id, could lead to higher efficiency per meter, routine analysis with such a column imposes high demands on instrumentation. It requires higher inlet pressures, better split control,

and faster oven temperature heating rates. On the contrary, 0.18 mm id columns are conveniently compatible with existing standard GC equipment without the need for system modifications. Smaller id, shorter length columns require less carrier flow to achieve separations, thus reduce carrier gas usage. Therefore, high-efficiency 0.18 mm id columns can provide an easy and inexpensive way to speed up GC analysis without compromising resolution.

One note of caution when going to smaller id columns is lower sample capacity. With some special samples, it is important to find a balance among speed, sensitivity, and resolution to meet the laboratory goals. For most applications in the chemical, petrochemical, food, or flavor/fragrance industries, however, the use of HE GC columns can offer an important reduction in analysis time and, consequently, a higher sample throughput.

The purpose of this application is to demonstrate in depth the use of high-efficiency 0.18 mm id columns for faster analysis of aromatic solvents with the unified aromatic solvent analysis method.

Results and Discussion

One Agilent 6890N Series gas chromatograph and two Agilent 7890 gas chromatographs were used for this work. Each GC was equipped with a split/splitless capillary inlet, a flame ionization detector (FID), and an Agilent 7683 Automatic Liquid Sampler (ALS). The split/splitless inlets were fitted with a long-lifetime septa (Agilent part no. 5183-4761) and split-optimized liners (Agilent part no. 5183-4647). Injections were made using 10- μ L syringes (Agilent part no. 5181-3354). Agilent ChemStation was used for all instrument control, data acquisition, and data analysis.

A 50-mL n-Hexane solution was prepared containing 0.1 wt% of 27 compounds; that is, all the aromatic solvents and impurities specified for analysis by the 10 ASTM methods.

Table 2 lists the experimental conditions for Method 1 where the unified aromatic solvent analysis was performed using a conventional 60 m \times 0.32 mm \times 0.5 μ m HP-INNOWax column (Agilent part no. 19091N-213). The GC chromatogram is shown in Figure 1.

Table 2. Conditions for Unified Aromatic Solvents Method Using a Conventional Column (Method 1)

| | |
|-----------------------|---|
| Column | HP-INNOWax, 60 m \times 0.32 mm \times 0.50 μ m |
| Carrier gas | Helium at 20.00 psi constant pressure mode |
| Inlet | Split/splitless at 250 $^{\circ}$ C 100:1 split ratio |
| Oven temp | 75 $^{\circ}$ C (10 min); 3 $^{\circ}$ C/min to 100 $^{\circ}$ C (0 min) 10 $^{\circ}$ C/min to 145 $^{\circ}$ C (0 min) |
| Detector | FID at 250 $^{\circ}$ C |
| Data acquisition rate | At 20 Hz |
| Injection size | 1 μ L |

The experiment was then repeated with a high-efficiency 20 m \times 0.18 mm \times 0.18 μ m HP-INNOWax column (Agilent part no. 19091N-577) (Method 2). Agilent GC Method Translation Software (<http://www.chem.agilent.com/cag/servsup/usersoft/files/GCTS.htm>) was used to translate Method 1 to Method 2. Three translation modes, namely the “translate only,” “best efficiency,” and “fast analysis,” were attempted with the new column dimensions. However, co-elution of dodecane and o-xylene was observed for all three translated methods. According to ASTM methods, some modi-

fications of the temperature programs were therefore necessary to achieve a similar resolution to Method 1. The resulting experimental conditions are provided in Table 3 along with the chromatogram in Figure 2.

Table 3. Conditions for Aromatic Solvents Separations on a High-Efficiency Column (Method 2)

| | |
|-----------------------|---|
| Column | HP-INNOWax, 20 m \times 0.18 mm \times 0.18 μ m |
| Carrier gas | Helium at 25.00 psi constant pressure mode |
| Inlet | Split/splitless at 250 $^{\circ}$ C 100:1 split ratio |
| Oven temp | 50 $^{\circ}$ C (2 min); 15 $^{\circ}$ C/min to 90 $^{\circ}$ C (0 min); 20 $^{\circ}$ C/min to 145 $^{\circ}$ C (1min) |
| Detector | FID at 250 $^{\circ}$ C |
| Data acquisition rate | At 50 Hz |
| Injection size | 0.2 μ L |

In order to achieve even faster separation while balancing speed and resolution, Agilent GC Method Translation Software was used to translate Method 1 to Method 3 while selecting “fast analysis” mode and using the same high-efficiency GC column. But dodecane and o-xylene could not achieve baseline separation with the obtained method as stated previously. According to ASTM methods, the obtained method conditions were used with minor adjustments of the initial temperature from 75 $^{\circ}$ C to 70 $^{\circ}$ C and the initial hold of 2 minutes to 3 minutes. Then baseline separation was obtained for dodecane and o-xylene (R_s = 2.78). Detailed experimental conditions are provided in Table 4 with the GC chromatogram in Figure 3.

Table 4. Conditions for Fast Aromatic Solvents Analysis (Method 3)

| | |
|-----------------------|--|
| Column | HP-INNOWax, 20 m \times 0.18 mm \times 0.18 μ m |
| Carrier gas | Helium at 33.00 psi constant pressure mode |
| Inlet | Split/splitless at 250 $^{\circ}$ C 100:1 to 600:1 split ratio |
| Oven temp | 70 $^{\circ}$ C (3 min); 45 $^{\circ}$ C/min to 145 $^{\circ}$ C (1 min) |
| Detector | FID at 250 $^{\circ}$ C |
| Data acquisition rate | At 50 Hz |
| Injection size | 0.2 to 1.0 μ L |

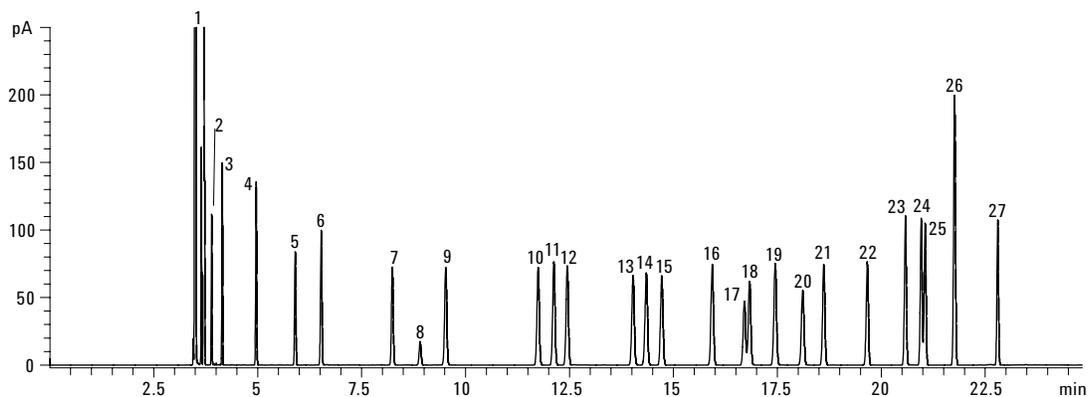


Figure 1. Unified aromatic solvent method with a 60 m × 0.32 mm × 0.5 μm HP-INNOWax column.

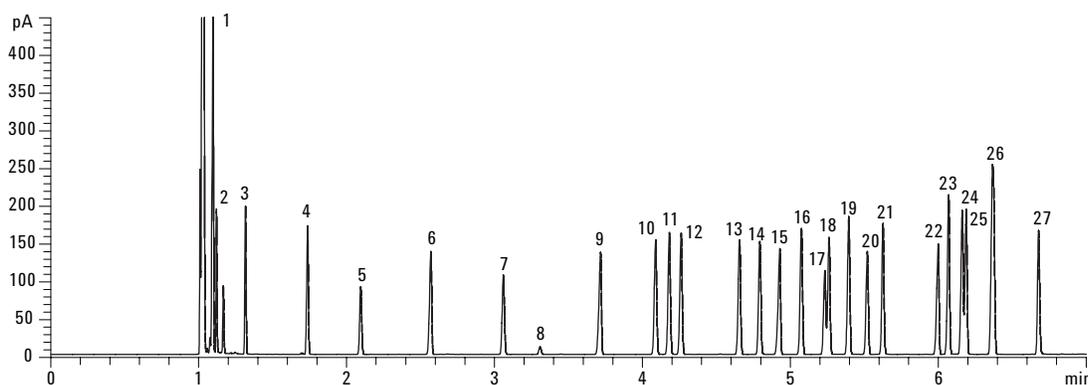


Figure 2. Separation of the same aromatic solvent with a 20 m × 0.18 mm × 0.18 μm HP-INNOWax column.

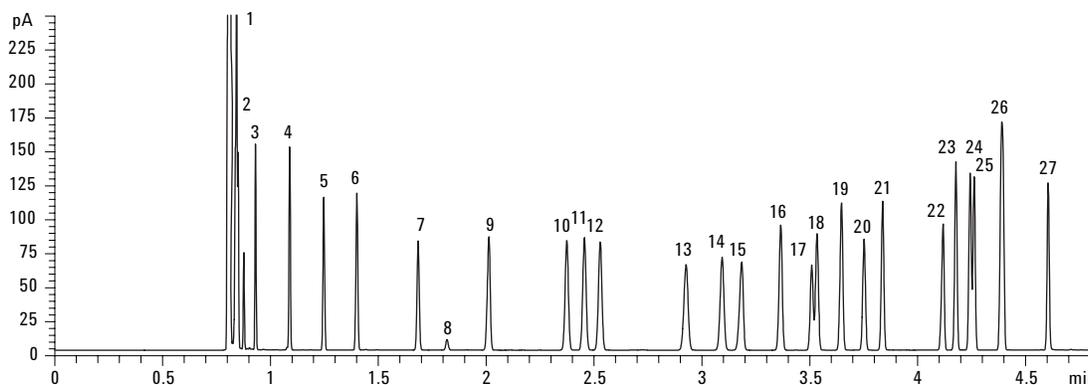


Figure 3. Optimized unified aromatic solvent method with a 20 m × 0.18 mm × 0.18 μm HP-INNOWax column.

| | | | |
|---------------|-----------------|-------------------|-----------------------|
| 1 Heptane | 8 1,4-Dioxan | 15 o-Xylene | 22 Tridecan |
| 2 Cyclohexane | 9 Undecane | 16 Propylbenzene | 23 1,3-Diethylbenzene |
| 3 Octane | 10 Ethylbenzene | 17 p-Ethyltoluene | 24 1,2-Diethylbenzene |
| 4 Nonane | 11 p-Xylene | 18 m-Ethyltoluene | 25 n-Butylbenzene |
| 5 Benzene | 12 m-Xylene | 19 t-Butylbenzene | 26 a-Methylstyrene |
| 6 Decane | 13 Cumene | 20 s-Butylbenzene | 27 Phenylacetylene |
| 7 Toluene | 14 Dodecane | 21 Styrene | |

Figures 1, 2, and 3 show the chromatograms of the hexane solution containing an aggregate of aromatic solvents and impurities for Method 1, Method 2, and Method 3, respectively. As indicated in the three chromatograms, baseline resolution was achieved for most of the compounds of interest except for two compound pairs, which were

only partially resolved. The first pair, p-ethyltoluene and m-ethyltoluene, was also not resolved in the original ASTM method (D-5060, impurities in ethylbenzene) and, along with o-ethyltoluene, was reported as total ethyltoluene. A second pair, diethylbenzene and n-butylbenzene, was also partially resolved. However, this should not present a

problem since they are not typically found together within the same material. Diethylbenzene is sometimes found as a contaminant in ethyl benzene (ASTM Method D-5060) while n-butyl benzene is used as the internal standard for cumene analysis (ASTM Method D-3760).

The sample run time for Method 1 was 23 minutes (Figure 1), whereas it was 7 minutes for Method 2 (Figure 2). The 3x speedup was achieved by using a shorter and narrower bore high-efficiency column. The optimized Method 3 allowed for even faster analysis time at 5 minutes (Figure 3), resulting in 4.6x speedup as compared to Method 1. As shown in Table 5, similar resolution was obtained in spite of significant acceleration, indicating that fast sample throughput can be achieved with the high-efficiency columns without compromise on resolution.

Influence of Carrier Gas on Analysis Time

The type of carrier gas and its velocity highly impact resolution and retention time. Too high or too low of a carrier gas velocity results in loss of resolution. It is therefore important to set a correct gas velocity to achieve a right balance of resolution and analysis time.

Hydrogen, helium, and nitrogen are the most common carrier gases used. The use of hydrogen as a carrier gas provides a faster analysis with almost equivalent resolution because the optimum linear carrier gas velocity is higher due to the higher diffusivity of hydrogen. At the optimal flow rates of 12, 20, and 35 cm/s for nitrogen, helium, and hydrogen, respectively, the analysis times would be 35/12 to 35/20 to 1 for nitrogen, helium, and hydrogen, respectively.

Nitrogen vs. Helium Carrier Gas

To investigate the effect of carrier gas on sample analysis time, Agilent GC method translation software was used where “translate only” mode was

chosen so that all experimental conditions were held constant except for the carrier gas. Method 1 was translated to Method 4 where a nitrogen carrier was used (see Figure 4 and Table 6). As shown in Figure 5, the run time for a nitrogen carrier was about 60 minutes compared to 23 minutes with a helium carrier when using a 60 m × 0.32 mm × 0.5 μm HP-INNOWax column.

| Criterion: | Translate Only | Best Efficiency | Fast Analysis | None | Speed gain: 0.43501 |
|----------------------------------|-----------------|-----------------|--|-------|---------------------|
| Column | Original Method | | Translated Method | | |
| Length, m | 60 | | <input type="checkbox"/> 60 | | |
| Internal Diameter, μm | 320 | | <input type="checkbox"/> 320 | | |
| Film Thickness, μm | 0.500 | | <input type="radio"/> Unlock | | |
| Phase Ratio | 160.0 | | <input checked="" type="radio"/> 0.500 | | |
| | | | <input type="radio"/> 160.0 | | |
| Carrier Gas | Helium | | Nitrogen | | |
| Enter one Setpoint | | | | | |
| Head Pressure, psi | 20 | | 7.518 | | |
| Flow Rate, mL/min | 2.2911 | | 0.7160 | | |
| Outlet Velocity, cm/sec | 55.79 | | 17.43 | | |
| Average Velocity, cm/sec | 31.48 | | 13.69 | | |
| Hold-up Time, min | 3.17677 | | 7.30269 | | |
| Outlet Pressure (absolute), psi | 14.696 | | <input type="checkbox"/> 14.696 | | |
| Ambient Pressure (absolute), psi | 14.696 | | <input type="checkbox"/> 14.696 | | |
| Oven Temperature | 3-ramp Program | | 3-ramp Program | | |
| | Initial | | Initial | | |
| Ramp 1 | 3 | 100 | 0 | 1.305 | 100 |
| Ramp 2 | 10 | 145 | 0 | 4.350 | 145 |
| Ramp 3 | 0 | 0 | 0 | 0.000 | 0 |
| Sample Information | None | | None | | |

Figure 4. Method translation software input screen for a nitrogen carrier.

Table 6. Experimental Conditions for Unified Aromatic Solvents Method Using Nitrogen Carrier Gas (Method 4)

| | |
|-----------------------|--|
| Column | HP-INNOWax, 60 m × 0.32 mm × 0.50 μm |
| Carrier gas | Nitrogen at 7.60 psi constant pressure mode |
| Inlet | Split/splitless at 250 °C 100:1 split ratio |
| Oven temp | 75 °C (23 min); 1.3 °C/min to 100 °C (0 min) 4.4 °C/min to 145 °C (0 min) |
| Detector | FID at 250 °C |
| Data acquisition rate | At 20 Hz |
| Injection size | 0.2 μL |

Table 5. Comparison of Resolution of Difficult-to-Separate Compound Pairs Under Different Experimental Conditions

| Compound | Ethylbenzene/p-xylene | p-Xylene/m-xylene | p-Ethyltoluene/m-ethyltoluene | Diethylbenzene/n-butylbenzene |
|----------|-----------------------|-------------------|-------------------------------|-------------------------------|
| Method 1 | 3.25 | 3.10 | 1.10 | 1.11 |
| Method 2 | 3.14 | 2.72 | 1.00 | 0.97 |
| Method 3 | 2.84 | 2.47 | 0.94 | 0.88 |

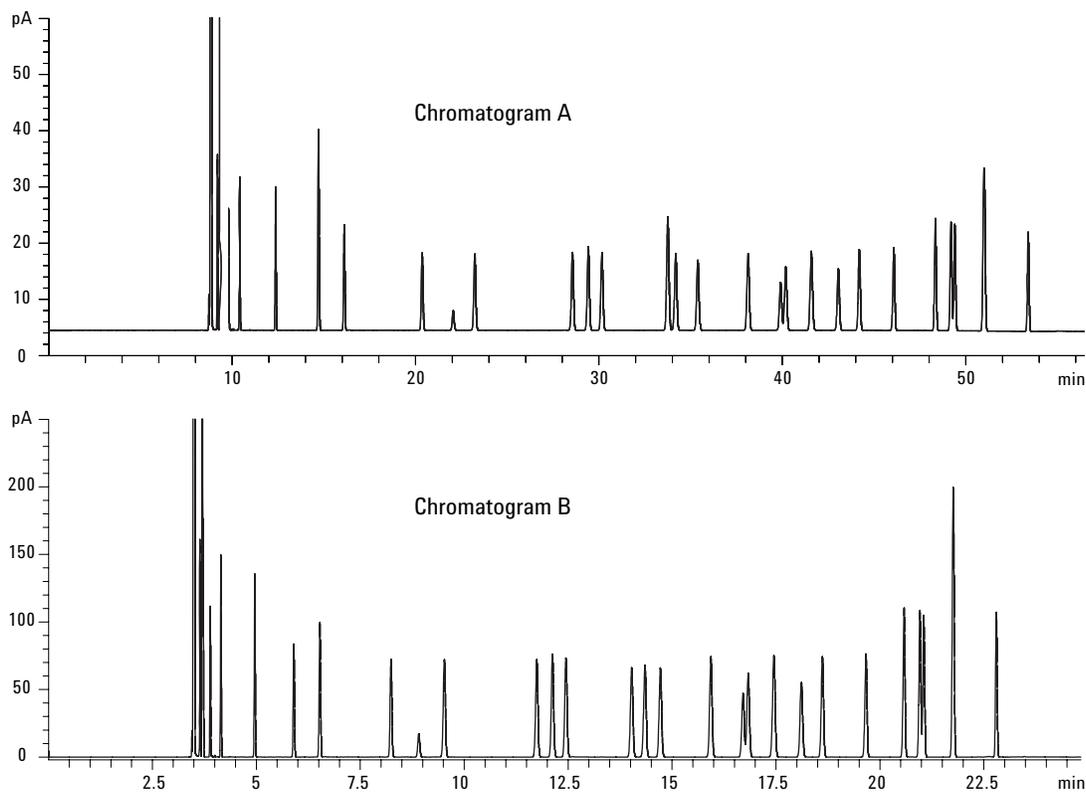


Figure 5. Comparison of unified aromatic solvent analysis using nitrogen and helium carrier gases with a 60 m × 0.32 mm × 0.5 μm HP-INNOWax column. 5a. Nitrogen carrier gas (Method 4). 5b. Helium carrier gas (Method 1).

Hydrogen vs. Helium Carrier Gas

A faster analysis can be achieved by switching the carrier gas from helium to hydrogen on the same column. Method 3 was translated to Method 5 using the method translation software (see Figure 6); the

detailed experimental condition is provided in Table 7. As shown in Figure 7, the total run time was decreased from 5 to 3 minutes by changing the carrier gas from helium to hydrogen while keeping the peaks well separated.

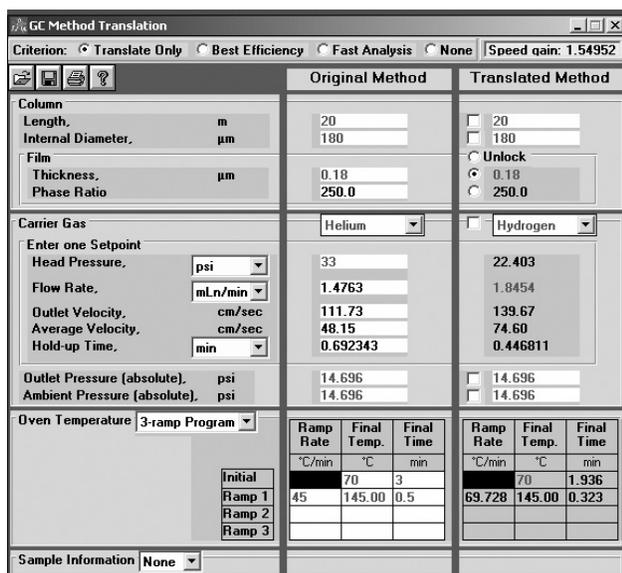


Figure 6. Method translation software input screen for a hydrogen carrier.

Table 7. Experimental Conditions for Unified Aromatic Solvents Method Using Hydrogen Carrier Gas (Method 5)

| | |
|-----------------------|---|
| Column | HP-INNOWax, 20 m × 0.18 mm × 0.18 μm |
| Carrier gas | Hydrogen at 22.00 psi constant pressure mode |
| Inlet | Split/splitless at 250 °C 100:1 split ratio |
| Oven temp | 70 °C (2 min); 70 °C/min to 145 °C (0.5 min) |
| Detector | FID at 250 °C |
| Data acquisition rate | At 50 Hz |
| Injection size | 0.2 μL |

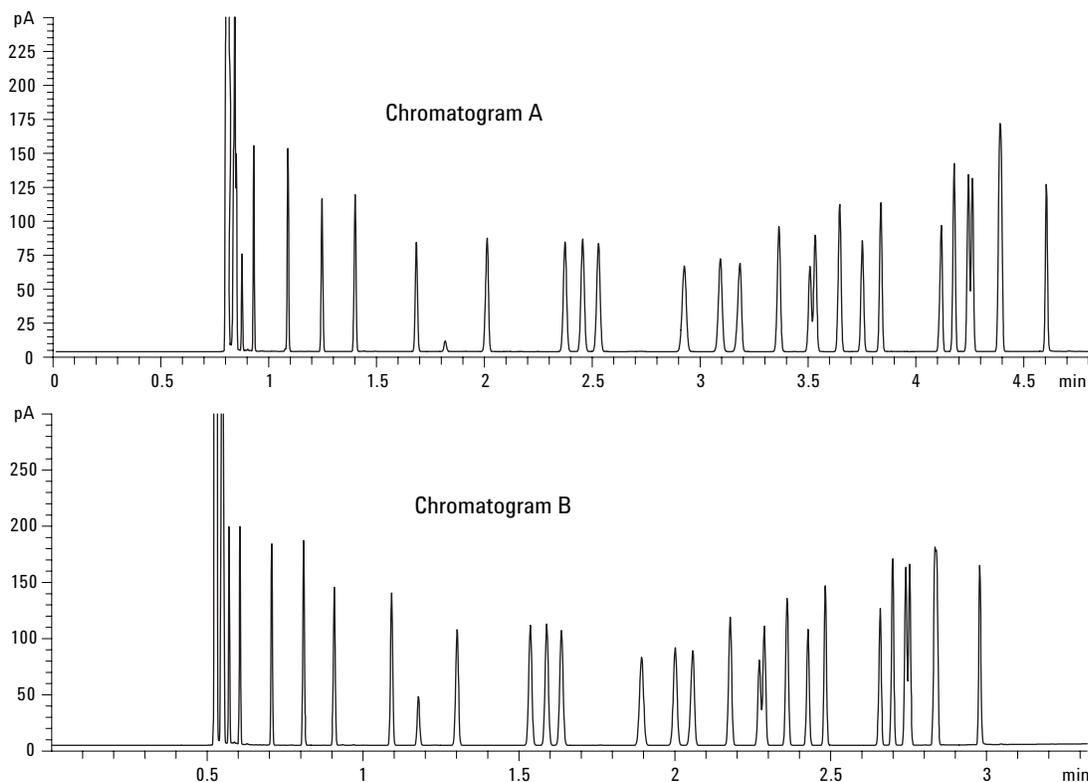


Figure 7. Comparison of unified aromatic solvent analysis using helium and hydrogen carrier gases with a 20 m × 0.18 mm × 0.18 μm HP-INNOWax column. 7a. Helium carrier gas (Method 5). 7b. Hydrogen carrier gas (Method 3).

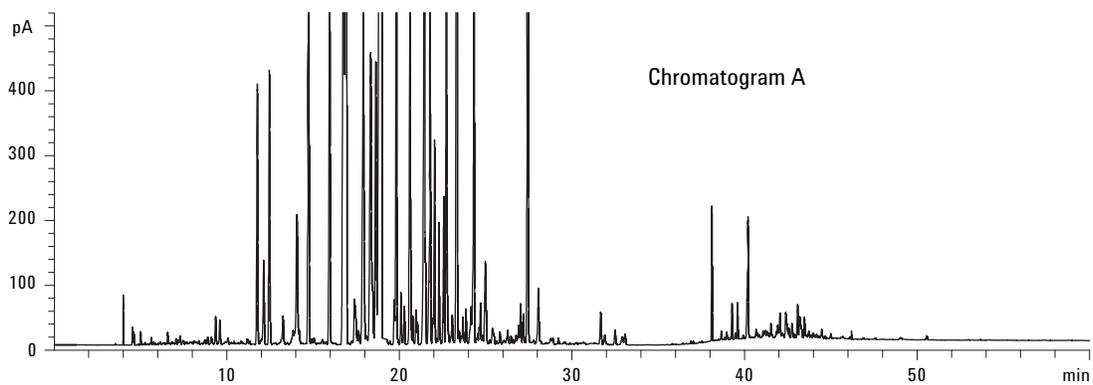
Complex Matrix Sample

To validate the practicality of fast GC application using high-efficiency GC columns, a real aromatic solvent sample offered by a large-scale integrated petrochemical company was analyzed using the same experimental conditions as those for the standards (Methods 1, 2, and 3); the chromatograms are provided in Figures 8a, 8b, and 8c. A detailed comparison of the center sections is also provided in Figures 8d and 8e.

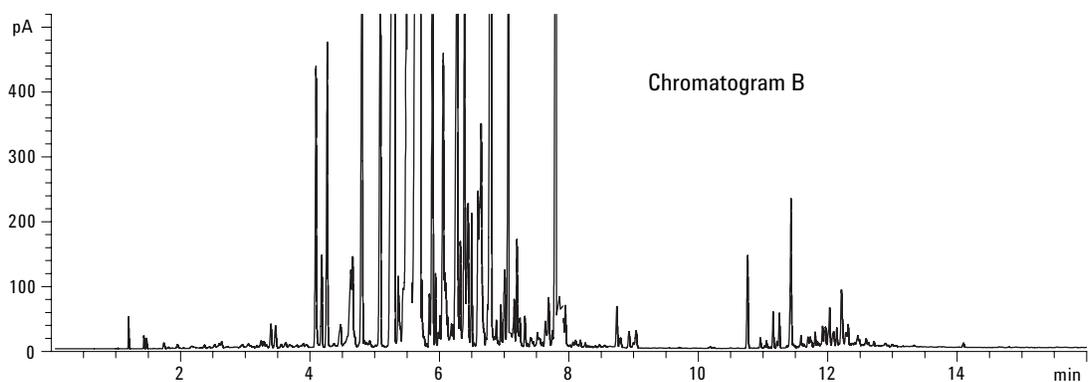
Although the analysis time is a bit longer with Method 2 compared to Method 3, the overall resolution obtained is slightly better for Method 2 (see

Figures 8e and 8f). On the other hand, all the key compounds, including benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, propylbenzene, and a-methylstyrene, were well separated with all three methods.

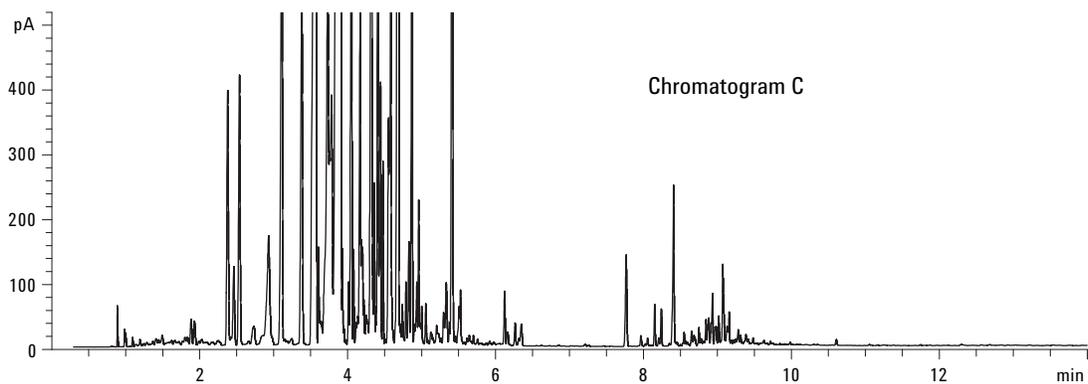
For complex matrix samples, a balance between speed and resolution must be selected according to the laboratory goals. In this case, it demonstrates that a complex matrix sample can be separated well on a high-efficiency 0.18 mm id GC column, where a more than 3x improvement in run time was accomplished compared to a 0.32 mm id column using a helium carrier.



Column HP-INNOWax, 60 m × 0.32 mm × 0.50 μm
 Carrier gas Helium at 20.00 psi constant pressure mode
 Inlet Split/splitless at 250 °C; 50:1 split ratio
 Oven temp 75 °C (10 min); 3 °C/min to 100 °C (0 min); 10 °C/min to 145 °C (12.17 min), 25 °C/min to 220 °C (22 min)
 Detector FID at 250 °C
 Injection size 0.2 μL



Column HP-INNOWax, 20 m × 0.18 mm × 0.18 μm
 Carrier gas Helium at 25.00 psi constant pressure mode
 Inlet Split/splitless at 250 °C; 150:1 split ratio
 Oven temp 50 °C (2 min); 15 °C/min to 90 °C (0 min); 20 °C/min to 145 °C (3 min), 80 °C/min to 220 °C (8 min)
 Detector FID at 250 °C
 Injection size 0.2 μL



Column HP-INNOWax, 20 m × 0.18 mm × 0.18 μm
 Carrier gas Helium at 33.00 psi constant pressure mode
 Inlet Split/splitless at 250 °C; 150:1 split ratio
 Oven temp 70 °C (3 min); 45 °C/min to 145 °C (3 min), 80 °C/min to 220 °C (8 min)
 Detector FID at 250 °C
 Injection size 0.2 μL

Figure 8. Comparison of real aromatic solvent sample separations (a) and (d) Method 1, (b) and (e) Method 2, and (c) and (f) Method 3.

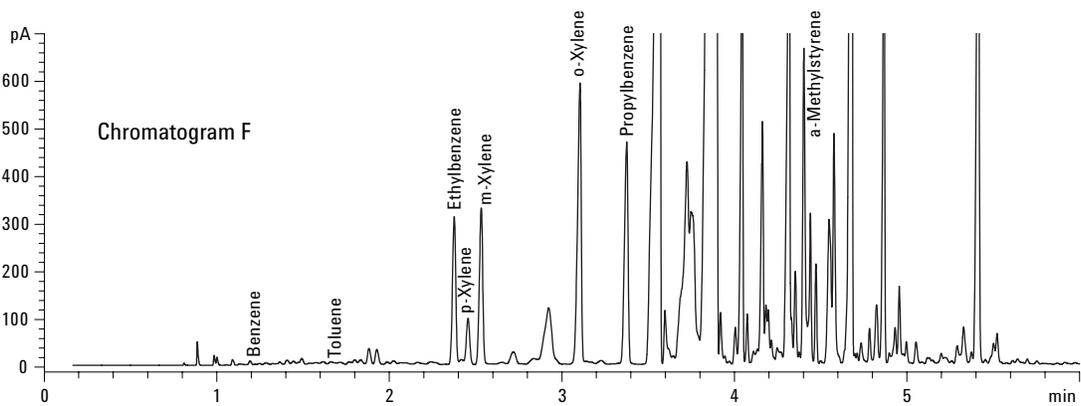
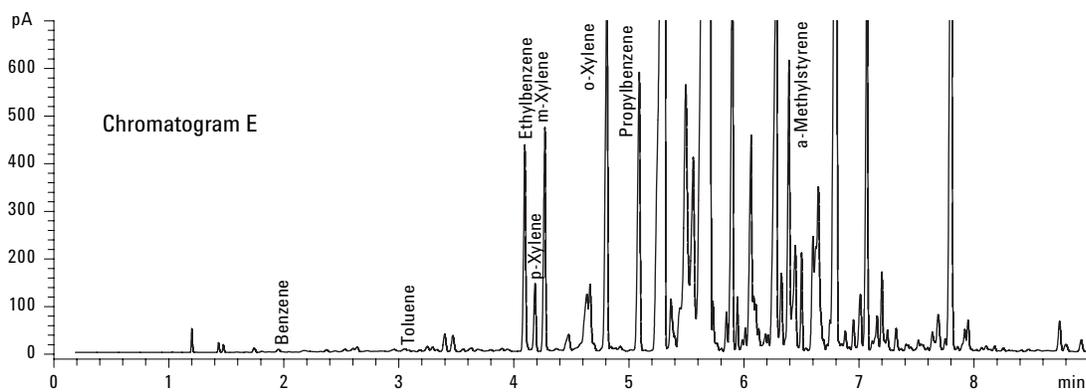
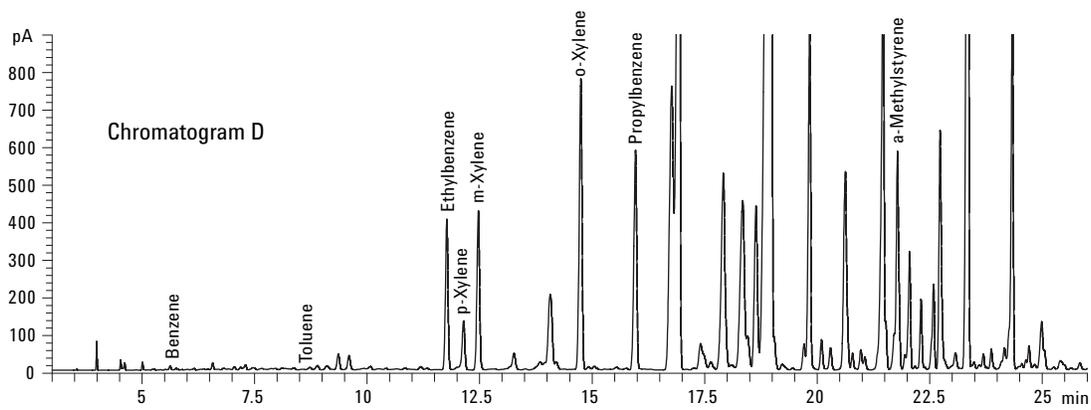


Figure 8. Comparison of real aromatic solvent sample separations (a) and (d) Method 1, (b) and (e) Method 2, and (c) and (f) Method 3. (continued)

Evaluation of Individual ASTM Calibration Standards

To evaluate the applicability of high-efficiency GC columns on individual ASTM calibration standards, experiments were carried out with Methods 1 and 3, respectively, on a 7890 gas chromatography system. All standards were prepared as outlined by the ASTM methods.

D2306 – Standard Test for C8 Aromatic Hydrocarbons

Concentration of ASTM D2306 standard calibration mix is quite high. It is therefore a challenge

regarding the capacity of the high-efficiency 0.18 mm id column. The workaround is to inject a small volume with a high split ratio. In this experiment, the injection size was 0.2 μL and the split ratio was 600:1. As shown in Figure 9, the run time for the high-efficiency GC column was about 4.5 times shorter than that of the traditional one. The resolution is acceptable in spite of the high concentration of the calibration standard (see Table 8).

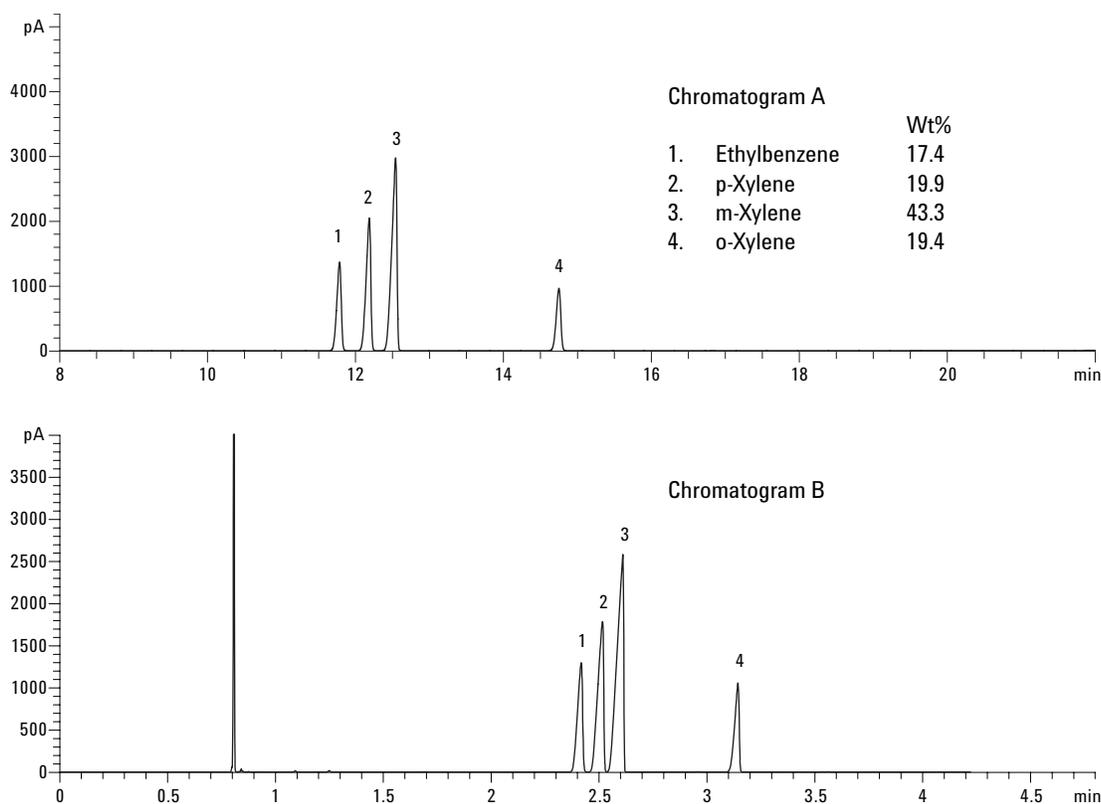


Figure 9. ASTM D2306 C8 aromatic hydrocarbon quantitative calibration standards (a) on a standard column (Method 1) and (b) on a high-efficiency GC column (method 3).

Table 8. Comparison of Resolution Under Different Experimental Condition

| Compound | Ethylbenzene/p-xylene | p-Xylene/m-xylene | m-Xylene/o-xylene |
|----------|-----------------------|-------------------|-------------------|
| Method 1 | 3.52 | 2.86 | 18.11 |
| Method 3 | 2.10 | 1.73 | 11.20 |

D2360 – Standard Test for Trace Impurities in Monocyclic Hydrocarbons

The standard calibration mix specified by D2360 was prepared in p-xylene. Injection size for this run was 0.2 μ L and the split ratio was 200:1.

Similar resolution was obtained for the compounds of interest (Figure 10), except for the sample run time being decreased from 21.05 minutes (Method 1) to 4.28 minutes (Method 3).

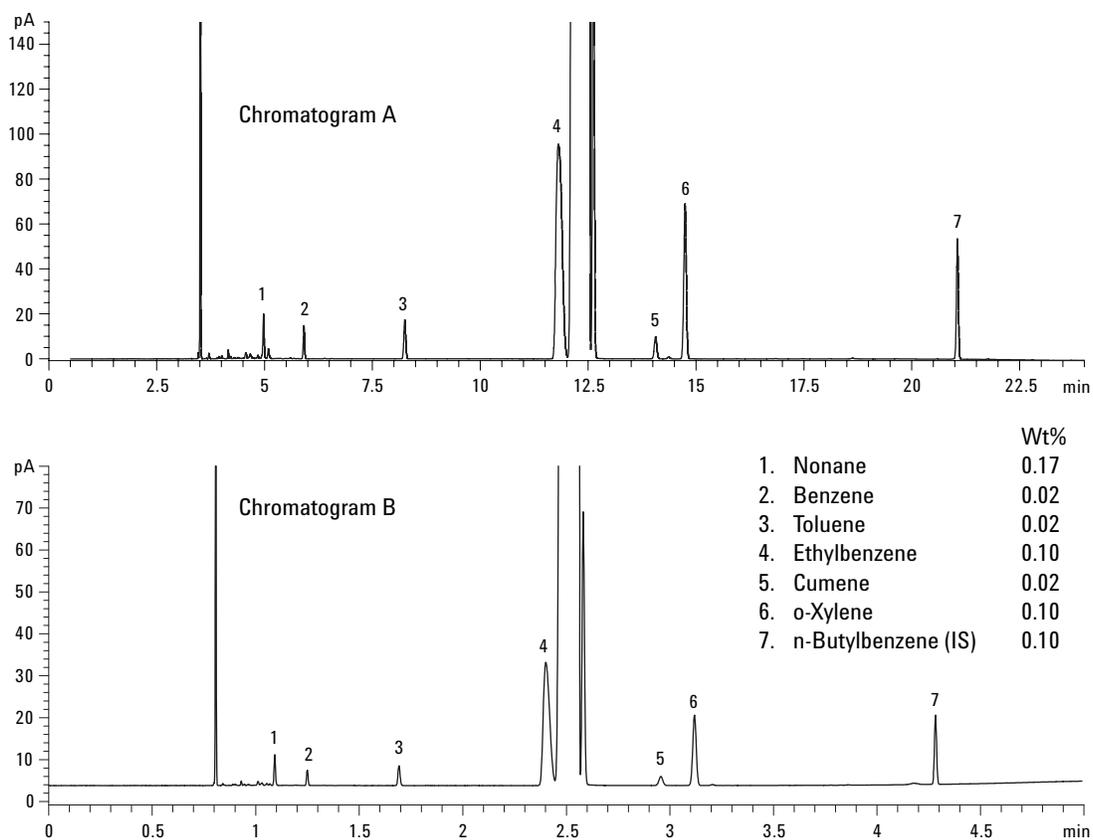
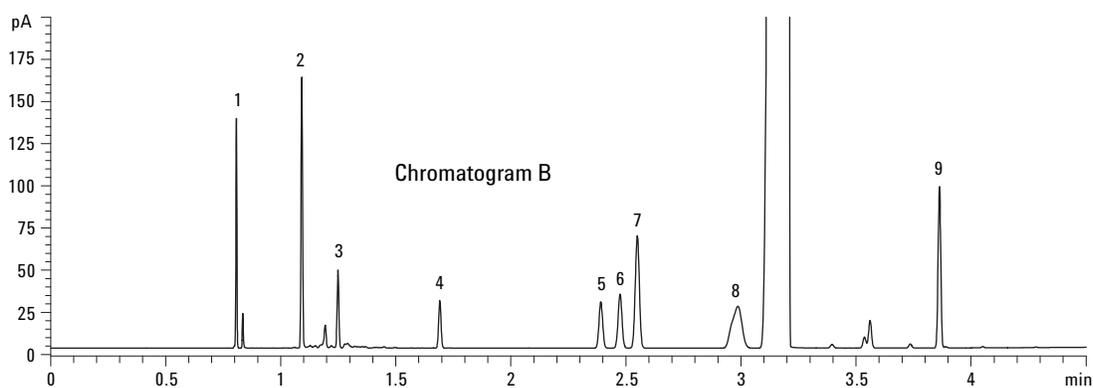
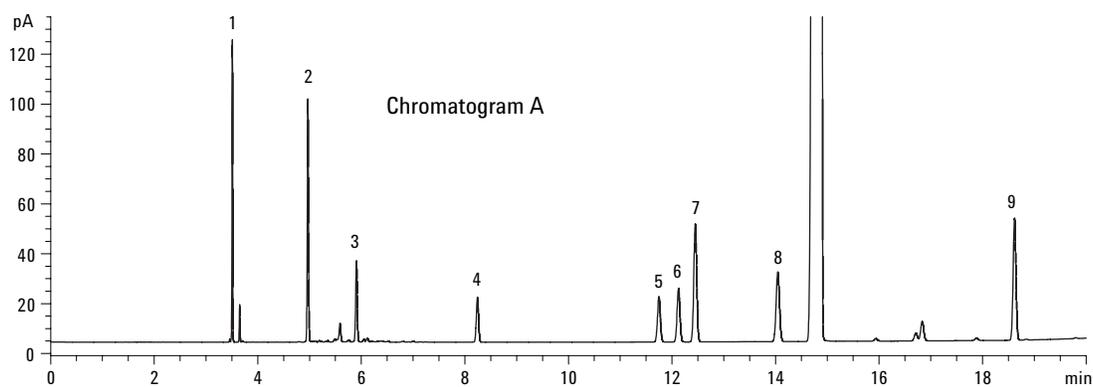


Figure 10. ASTM D2360 monocyclic hydrocarbon quantitative calibration standard run (a) on a standard column (Method 1) and (b) on a high-efficiency GC column (Method 3).

D3797 – Standard Test Method for Analysis of o-Xylene

Figure 11 shows the chromatograms of the D3797 calibration standard. Injection size for this run was 0.2 μ L and the split ratio was 100:1.

The broadening of the cumene peak was due to the reverse solvent effect of the overloaded o-xylene peak. This was also observed in the original ASTM D3797 method [4]. Comparison of the chromatograms in Figure 11 indicates that the D3797 calibration standard can be separated well on a high-efficiency 0.18 mm id GC column without loss of resolution.



| | Wt% | | Wt% |
|-------------------|------|-------------|------|
| 1. Isooctane (IS) | 0.05 | 6. p-Xylene | 0.21 |
| 2. Nonane | 0.21 | 7. m-Xylene | 0.42 |
| 3. Benzene | 0.20 | 8. Cumene | 0.31 |
| 4. Toluene | 0.21 | 9. Styrene | 0.12 |
| 5. Ethylbenzene | 0.21 | | |

Figure 11. o-Xylene standard run (a) on a standard column (Method 1) and (b) on a high-efficiency GC column (Method 3).

D3798 – Standard Test Method for Analysis of p-Xylene

This test method covers the determination of known hydrocarbon impurities in p-xylene and the measurement of p-xylene purity by GC. It is generally used for the analysis of p-xylene of 99% or greater purity.

Figure 12 shows the chromatograms of the D3798 calibration standard. Injection size for this run

was 0.2 μ L and the split ratio was 100:1. The original ASTM D3798 method specifies that the valley points between the large p-xylene peak and the ethylbenzene and m-xylene contaminants should be less than 50% of the contaminants' peak height. Excellent separation was obtained for the critical compounds (Figure 13) with great reproducibility (Figure 14) when using a high-efficiency GC column.

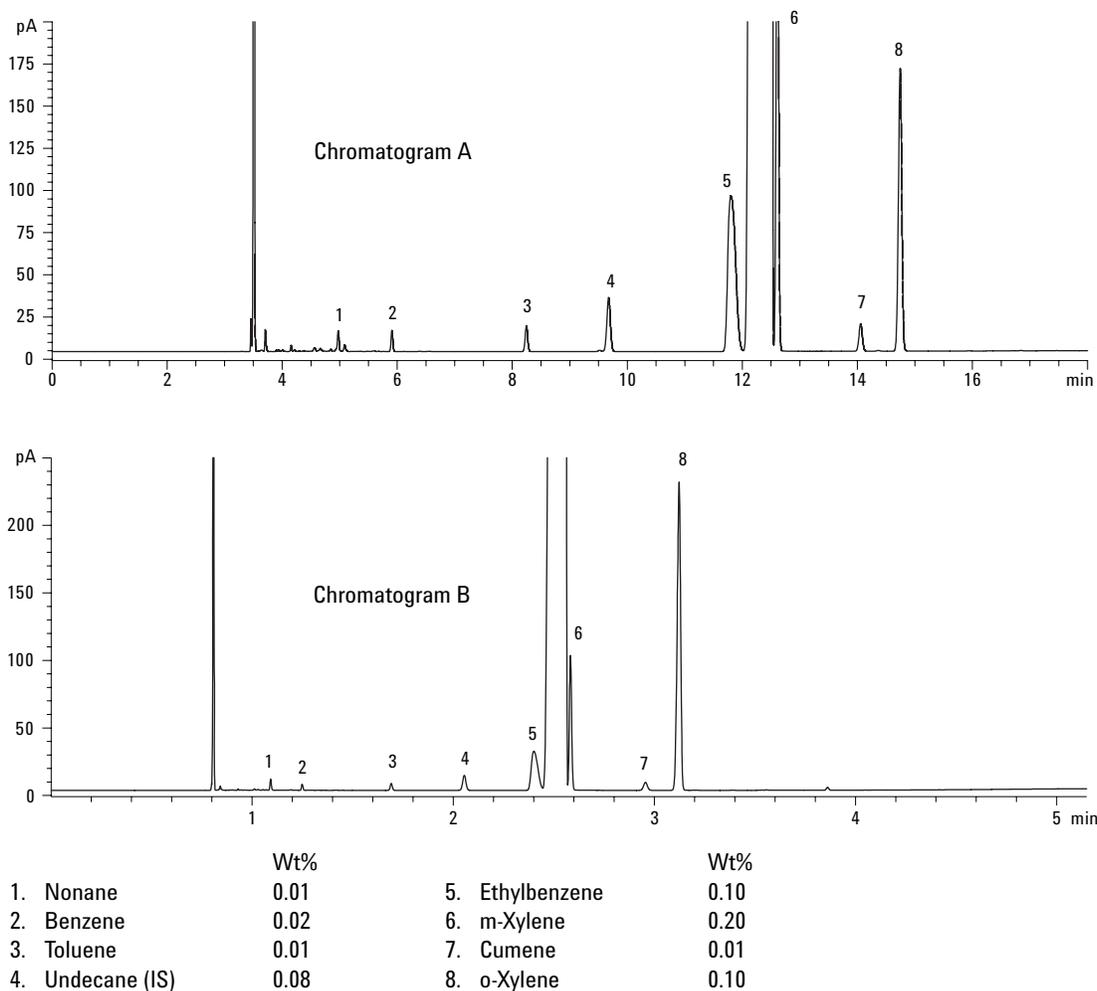


Figure 12. p-Xylene standard run (a) on a standard column (Method 1) and (b) on a high-efficiency GC column (Method 3).

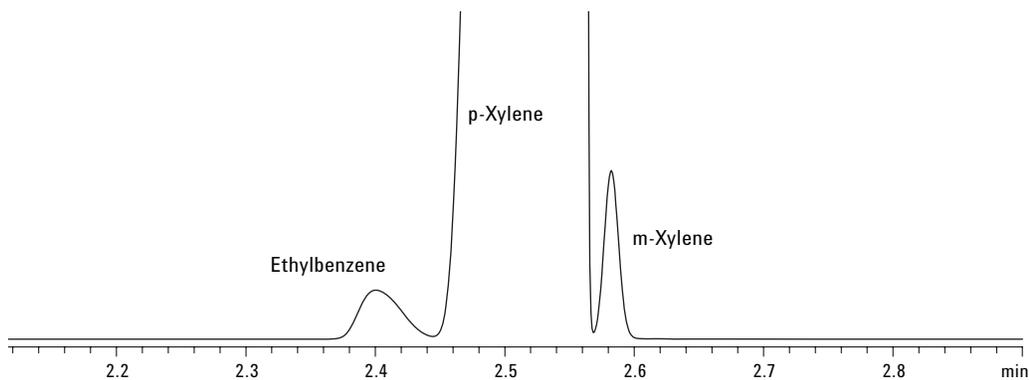


Figure 13. Expanded view from Figure 7 shows excellent separation of m-Xylene peak from p-Xylene peak using the fast GC method.

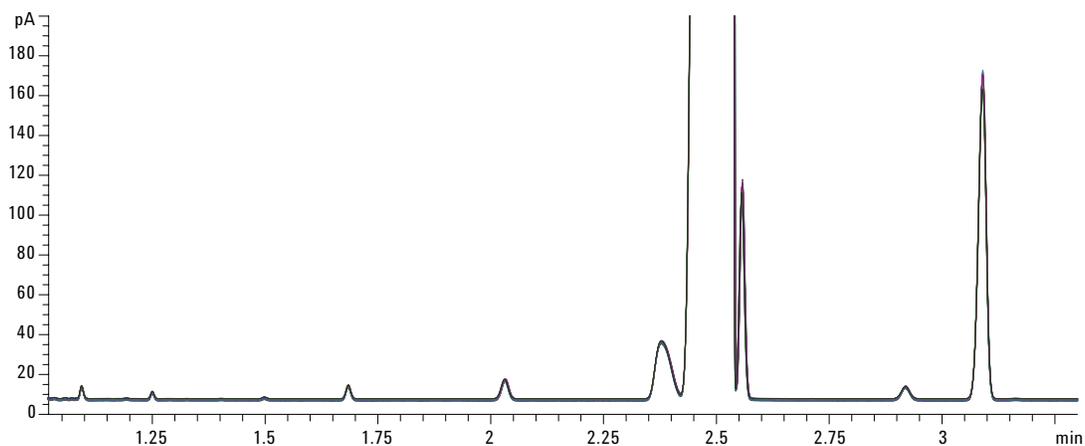


Figure 14. D3798 standard 30th run overlaid using a high-efficiency GC column.

D4492 – Standard Test for Analysis of Benzene

This test method determines the normally occurring trace impurities in, and the purity of, finished benzene. It is applicable for aromatic impurities from 0.001 to 0.010 weight % in benzene. Injection size for this run was 0.2 μ L and the split ratio was 50:1.

Figure 15 compares the chromatograms of the D4492 calibration standard with Methods 1 and 3, where good separation of the D4492 calibration standards can be achieved with a high-efficiency column but with 80% saving on analysis time.

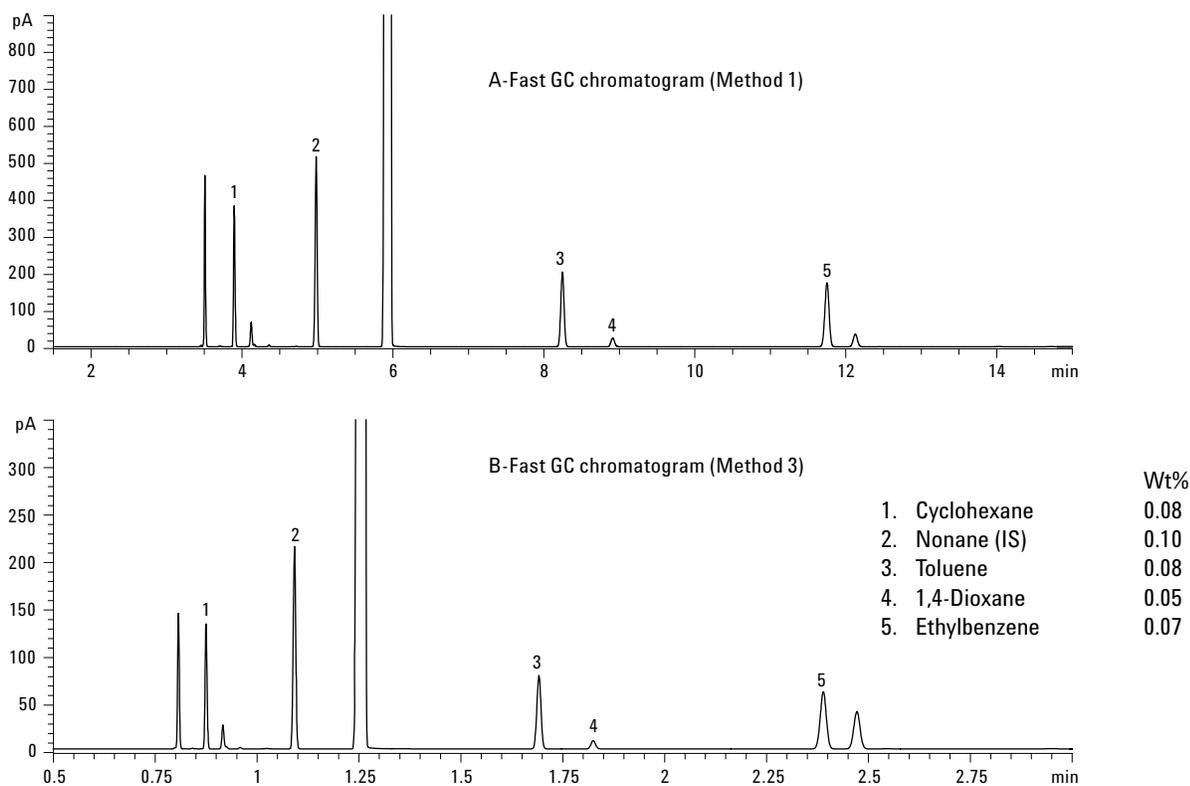


Figure 15. ASTM D4492 benzene quantitative calibration standard run (a) on a standard column (Method 1) and (b) on a high-efficiency GC column (Method 3).

In summary, the analysis time for Method 3 is on average 5x shorter than that for Method 1 when working with the calibration standard samples.

Conclusions

Fast GC applications can significantly improve laboratory productivity by decreasing analysis time. This application showcases the practicality of high-efficiency GC columns in daily aromatic solvent analysis and the associated time savings achieved with these columns. By using high-efficiency GC columns with smaller inner diameters and shorter column lengths as well as an appropriate carrier gas (for example, helium or hydrogen), higher sample throughput and lower cost per sample is achievable [5] for chemical and petrochemical laboratories.

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