Application Note

Energy & Chemicals

A Unified Method for the Analysis of Monocyclic Aromatic Solvents Using the Agilent 8860 GC System and On-Board Data Processing

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Abstract

For those customers with simplified workflows needing only simple data analysis, the Agilent Browser Interface is available onboard the Agilent 8860 gas chromatography (GC) system with Agilent DA Express. Combining Browser Interface with DA Express allows users to do acquisition, basic integration, calibration, and reporting of onboard data with no additional workstation software needed. This Application Note describes a unified method for aromatic solvent purity analysis using Browser Interface and DA Express.

Introduction

Measuring and determining the purity of aromatic hydrocarbons is a routine analysis for many of the QA/QC labs in the chemical and petrochemical industry. Determining the type and amount of hydrocarbon impurities remaining from the manufacture of specific aromatic compounds such as benzene, toluene, ethylbenzene, *p*-xylene, *o*-xylene, styrene, and mixed xylenes used as chemical intermediates and solvents is often required. The ASTM D7504 method[1] is suitable for setting specifications and as an internal quality control tool where these products are produced or are used.

Most of China's national standards in the petrochemical industry comply with the ASTM methods. Many labs need to run several different ASTM methods to determine the impurity of the aromatic hydrocarbons, as currently there is no national standard method based on ASTM D7504 using helium as the carrier gas in China. A single, simple method for the impurity analysis of aromatic hydrocarbons was needed for the routine work. Considering that nitrogen is a common carrier gas in China, this method develops two universal methods using helium and nitrogen carrier gases separately.

The Browser Interface is a key feature of the 8860 GC. It can be accessed from a desktop, laptop, or tablet. It allows users to have easy access to most functions of the 8860 GC. Users can edit, modify, and save methods, and sequence directly onboard the GC. For labs with simplified workflows needing only simple data analysis, the Browser Interface on the 8860 GC offers the optional DA Express. This software allows users to do basic integration, linear calibration, and reporting of onboard data. The "Complete Solution" concept combines methods, sequences, and DA Express to provide a simplified workflow for the analysis of aromatic solvent purity.

Experimental

This study was performed on an Agilent 8860 GC equipped with a split/splitless inlet and a flame ionization detector (FID). Tables 1 and 2 show the details of the operating conditions using the helium and nitrogen as the carrier gas separately. Data acquisition was finished by running the sequences through the Browser Interface, and data integration was done by DA Express, as shown in Figures 1A and 1B.

Chemicals, standards, and samples

Single standards of 26 solvents (>98% purity) and *n*-hexane were purchased from ANPEL Laboratory Technologies (Shanghai) Inc.

A *n*-hexane solution was prepared containing 0.1 wt% of all the aromatic solvents and impurities specified in the ASTM D7504 method, as listed in Figure 2. The standard sample for toluene, *o*-xylene, *p*-xylene, ethylbenzene, and styrene were used for injection.

Table 1. Conditions for unified aromatic solvents separations using helium carrier gas.

Parameter	Value
GC	Agilent 8860 gas chromatography system
Software	Agilent Browser Interface and DA Express
Inlet	Split/splitless; 270 °C; split ratio 100:1
Liner	Ultra Inert, split, low pressure drop, glass wool (p/n 5190-2295)
Column	Agilent J&W HP-INNOWax, 60 m × 0.32 mm, 0.5 µm (p/n 19091N-216I)
Carrier	Helium, 2.1 mL/min, constant flow
Oven	60 °C (10 minutes), then 5 °C/min to 150 °C (10 minutes)
Detector	FID, 300 °C
Injection Size	0.6 µL (suggested by ASTM D7504 method) (p/n 5181-8810)

Table 2. Conditions for unified aromatic solvents separations using nitrogen carrier gas.

Parameter	Value
GC	Agilent 8860 gas chromatograph system
Software	Agilent Browser Interface and DA Express
Inlet	Split/splitless; 270 °C; split ratio 100:1
Liner	Ultra Inert, split, low pressure drop, glass wool (p/n 5190-2295)
Column	Agilent J&W HP-INNOWax, 60 m × 0.32 mm, 0.5 µm (p/n 19091N-216I)
Carrier	Nitrogen, 1.5 mL/min, constant flow
Oven	65 °C (23 minutes), then 5 °C/min to 150 °C, then postrun 220 °C (5 minutes)
Detector	FID, 300 °C
Injection Size	0.6 µL (suggested by ASTM D7504 method) (p/n 5181-8810)

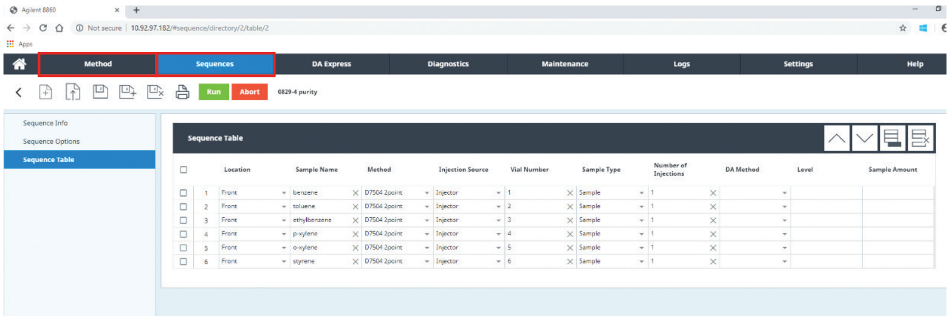

Figure 1A. Editing and running the sequences through the Agilent Browser Interface.

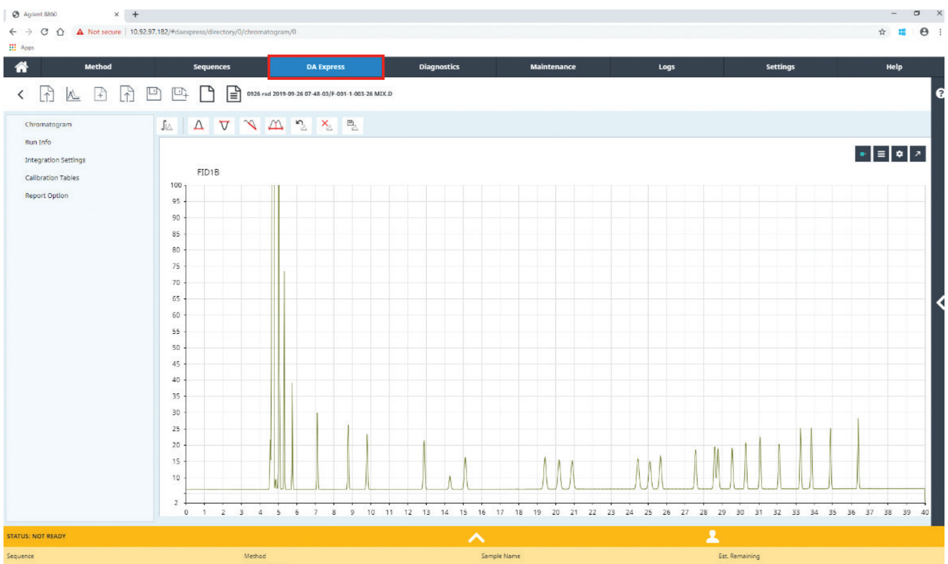

Figure 1B. Data analysis by Agilent DA Express.

Results and discussion

The ASTM D7504 method specifies the use of gas chromatography to measure overall chemical purity and the content of key impurities. Figure 2 shows a typical chromatogram of the hexane solution containing an aggregate of aromatic solvents and impurities using helium (He) carrier gas. As indicated in the chromatogram, baseline resolution was achieved for most of the compounds except for partially resolved *p*-ethyltoluene and *m*-ethyltoluene.

Considering that nitrogen (N_2) is a common and economical carrier gas in China, this Application Note develops a method using N_2 as the carrier gas. The type of carrier gas and its velocity highly impact resolution and retention time. It is important to set an appropriate gas velocity to achieve a balance of resolution and analysis time. The run time using N_2 as carrier gas is longer than when using He as the carrier gas. To ensure that some unrelated high-boiling impurities in the sample can be removed from the column and the analysis time can be shortened as much as possible, the post run function was adopted in the N_2 method. Figure 3 shows a

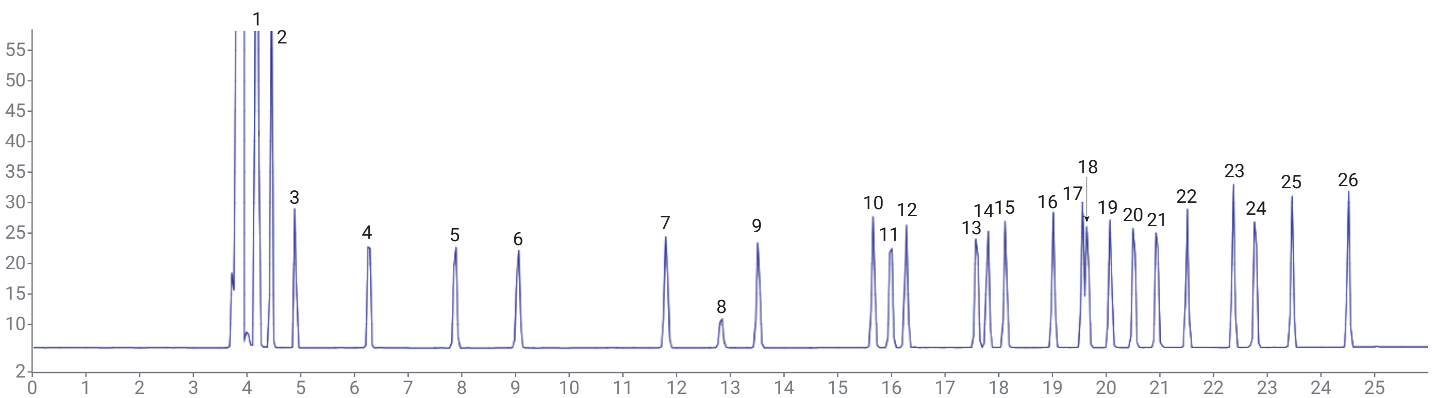

Figure 2. Chromatogram of a unified method for separating 26 compounds with He as the carrier gas.

chromatogram of the hexane solution containing the same 26 compounds using N_2 as the carrier gas. Compared to Figure 2, similar chromatographic performances, such as resolution and response, were achieved with a longer total analysis time. To verify the stability of the system, repeatability was determined by nine sequential injections of the 26-compound mixture. Table 3 illustrates that for all the compounds, the area %RSD is less than or equal to 2.06%, with the retention time %RSD less than or equal to 0.026%.

Table 3. RSD results for 26 compounds using N_2 carrier gas

		%RSD (n = 9)	
No.	**Name**	**RT**	**Area**
1	Heptane	0.021	1.07
2	Cyclohexane	0.022	1.19
3	Octane	0.023	1.67
4	Nonane	0.02	1.85
5	Benzene	0.019	0.98
6	Decane	0.023	1.89
7	Toluene	0.023	1.34
8	1,4-Dioxane	0.023	1.14
9	Undecane	0.023	1.75
10	Ethylbenzene	0.023	1.85
11	*p*-Xylene	0.026	1.62
12	*m*-Xylene	0.022	1.7
13	Cumene	0.014	1.91

		%RSD (n = 9)	
No.	**Name**	**RT**	**Area**
14	Dodecane	0.011	2
15	*o*-Xylene	0.013	1.75
16	Propylbenzene	0.009	1.9
17	*p*-Ethyltoluene	0.008	1.87
18	*m*-Ethyltoluene	0.007	1.88
19	*t*-Butylbenzene	0.006	1.87
20	*sec*-Butylbenzene	0.007	1.79
21	Styrene	0.005	1.73
22	Tridecane	0.004	2.03
23	1,3-Diethylbenzene	0.004	2
24	*n*-Butylbenzene	0.004	2.06
25	α-Methylstyrene	0.003	1.91
26	Phenylacetylene	0.003	1.58

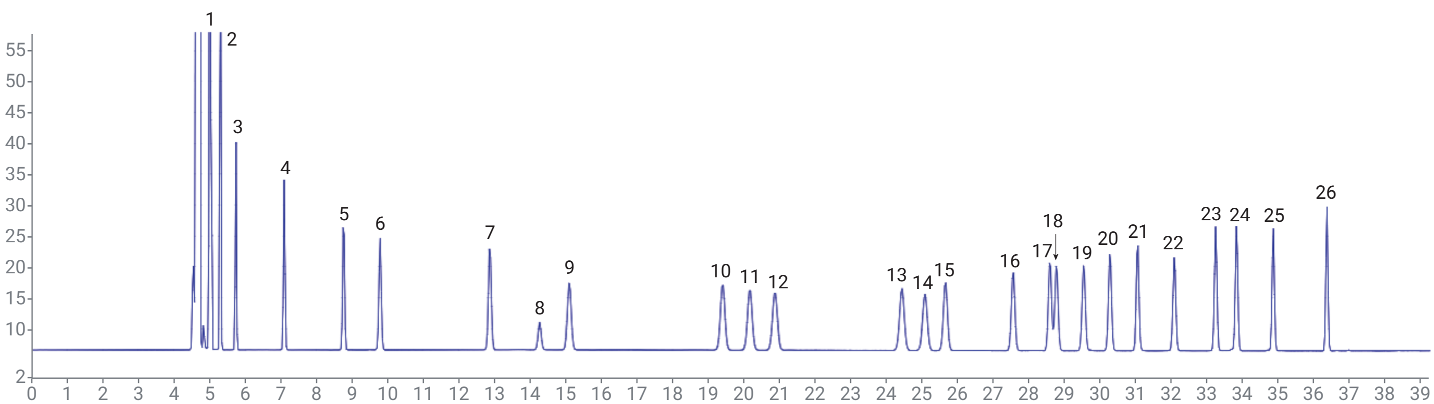

Figure 3. Chromatogram of a unified method for separating 26 compounds with N_2 as the carrier gas.

Purity analysis of toluene, ethylbenzene, *o*-xylene, *p*-xylene, and styrene

The ASTM D7504 method covers the determination of total nonaromatic hydrocarbons and monocyclic aromatic hydrocarbons in benzene, toluene, ethylbenzene, *p*-xylene, *o*-xylene, styrene, and mixed xylenes by GC using He carrier gas. In this Application Note, the N_2 method (Table 2) covers the determination of known hydrocarbon impurities in toluene, ethylbenzene, *p*-xylene, *o*-xylene, and styrene, and the measurement of each compound purity separately. It is generally used for the analysis of those five compounds of 99% or greater purity. Figures 4, 5, 6, 7, and 8 show the chromatograms of toluene, ethylbenzene, *o*-xylene, *p*-xylene, and styrene standard sample separately. The ASTM D7504 method also eliminates sample preparation and instrument calibration using effective carbon number (ECN) responses. The peak area of each component was measured and adjusted using ECN response factors. The concentration of each component was calculated based on its relative percentages of total adjusted peak area and normalized to 100%. DA Express can be used for qualitative analysis with a semiquantitative result using normalized area percent. This is often sufficient for labs doing internal QA or process monitoring. If a full quantitative report is required, the onboard data could be integrated by DA Express, and the peak areas exported to Excel to show a full quantitative report based on ECN. Results in Tables 4, 5, 6, 7, and 8 show the weight % of toluene, ethylbenzene, *o*-xylene, *p*-xylene, and styrene, and their impurities separately based on ECN calculation.

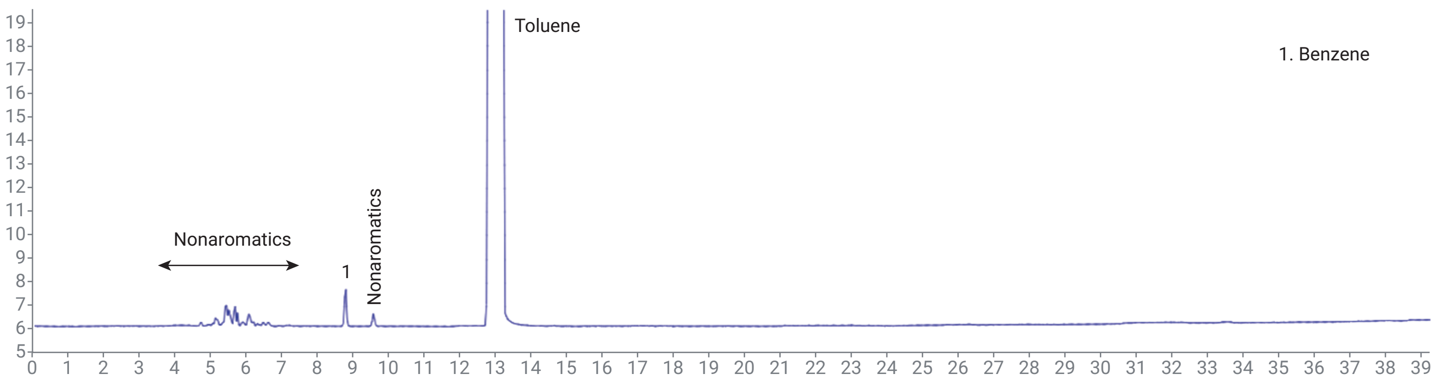

Figure 4. Toluene standard sample run using N_2 carrier gas.

Table 4. Toluene purity and impurities using N_2 carrier gas.

Compound	Results (wt %)
Nonaromatics	0.0272
Benzene	0.0063
Toluene	99.9665

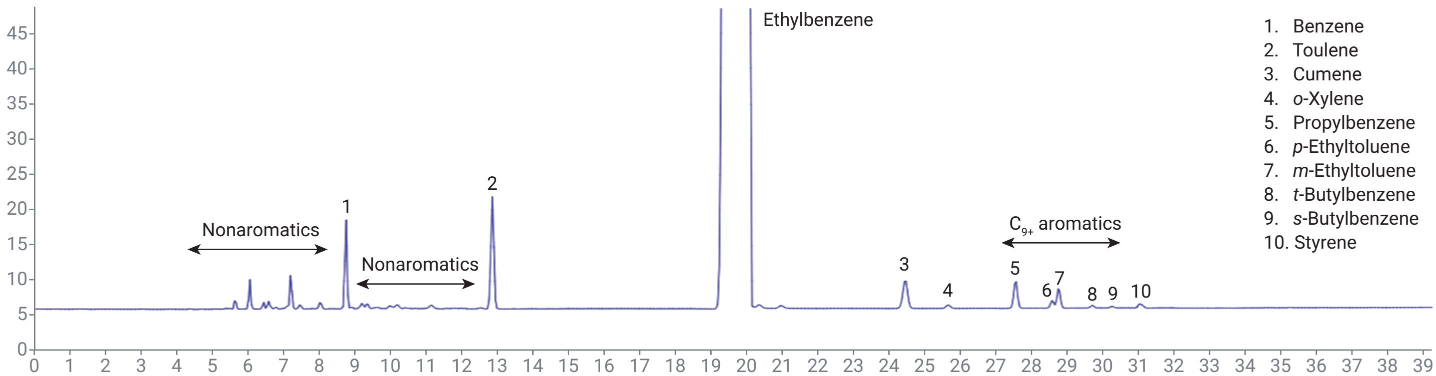

Figure 5. Ethylbenzene standard sample run using N_2 carrier gas.

Table 5. Ethylbenzene purity and impurities using N_2 carrier gas.

Compound	Results (wt %)
Nonaromatics	0.0975
Benzene	0.0503
Toluene	0.0861
Ethylbenzene	99.6661
Cumene	0.0351
o-Xylene	0.0042
Styrene	0.0052
C_{9+} Aromatics	0.0555

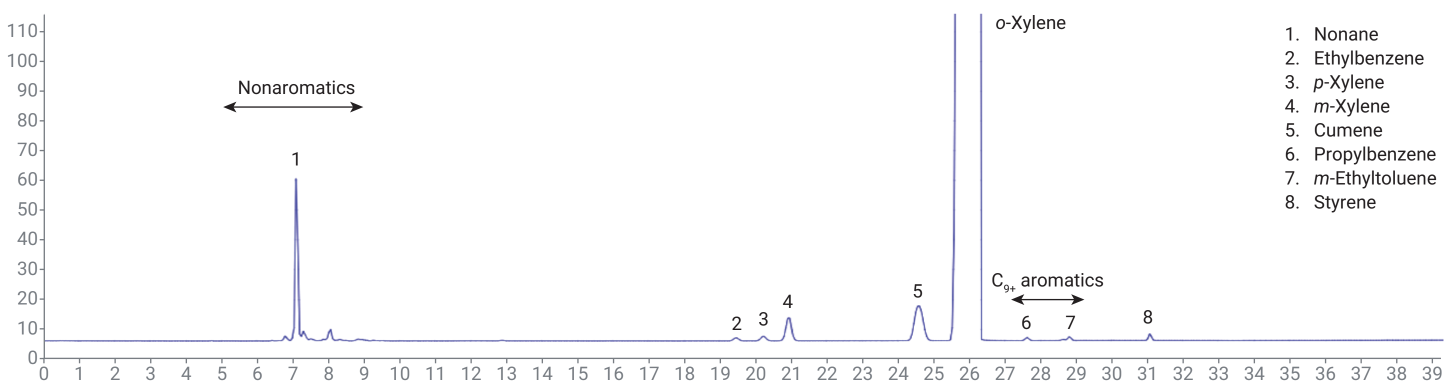

Figure 6. *o*-Xylene standard sample run using N_2 carrier gas.

Table 6. *o*-Xylene purity and impurities using N_2 carrier gas.

Compound	Results (wt %)
Nonaromatics	0.3711
Ethylbenzene	0.0097
p-Xylene	0.0148
m-Xylene	0.0781
Cumene	0.1798
o-Xylene	99.327
Styrene	0.0123
C_{9+} Aromatics	0.0072

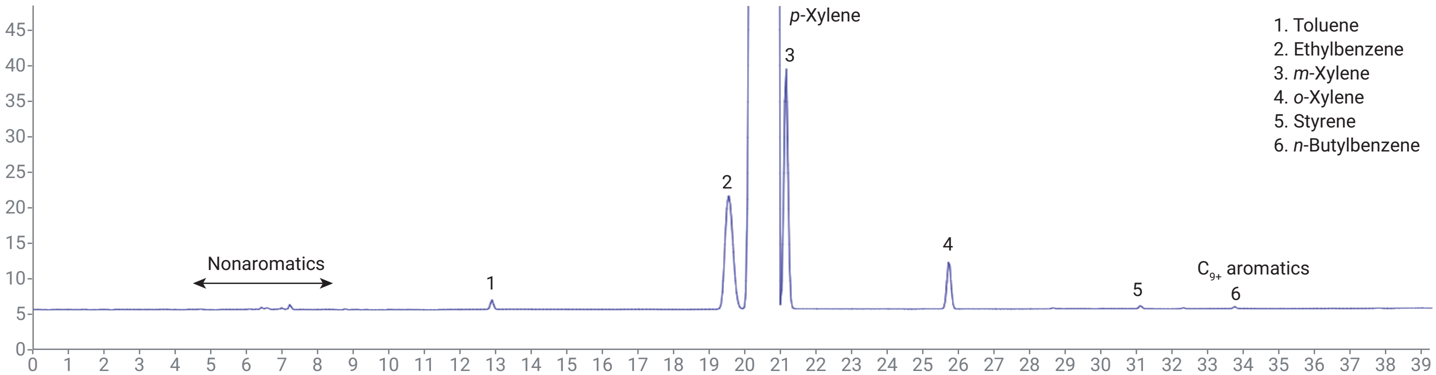

Figure 7. *p*-Xylene standard sample run using N_2 carrier gas.

Table 7. *p*-Xylene purity and impurities using N_2 carrier gas.

Compound	Results (wt %)
Nonaromatics	0.0105
Toluene	0.0079
Ethylbenzene	0.2307
p-Xylene	99.4344
m-Xylene	0.26
o-Xylene	0.052
Styrene	0.0028
C_{9+} Aromatics	0.0017

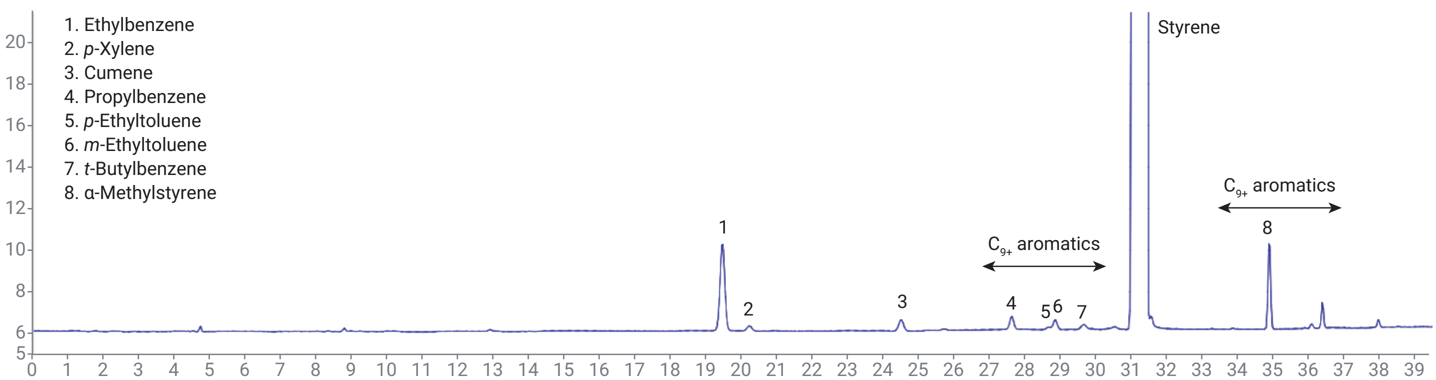

Figure 8. Styrene standard sample run using N_2 carrier gas.

Table 8. Styrene purity and impurities using N_2 carrier gas.

Compound	Results (wt %)
Ethylbenzene	0.0353
p-Xylene	0.0011
Cumene	0.0054
Styrene	99.921
C_{9+} Aromatics	0.0372

Conclusion

The Agilent 8860 GC and Browser interface with DA Express software was shown to successfully run the ASTM D7504 method for the determination of monocyclic aromatic solvent purity. This system also shows great applicability using N_2 as the carrier gas for the same compounds analysis. DA Express provides an easy way to carry out data analysis, which when combined with the Browser Interface acquisition methods and sequences, facilitates a powerful and simple approach to conducting aromatic solvent analysis.

References

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