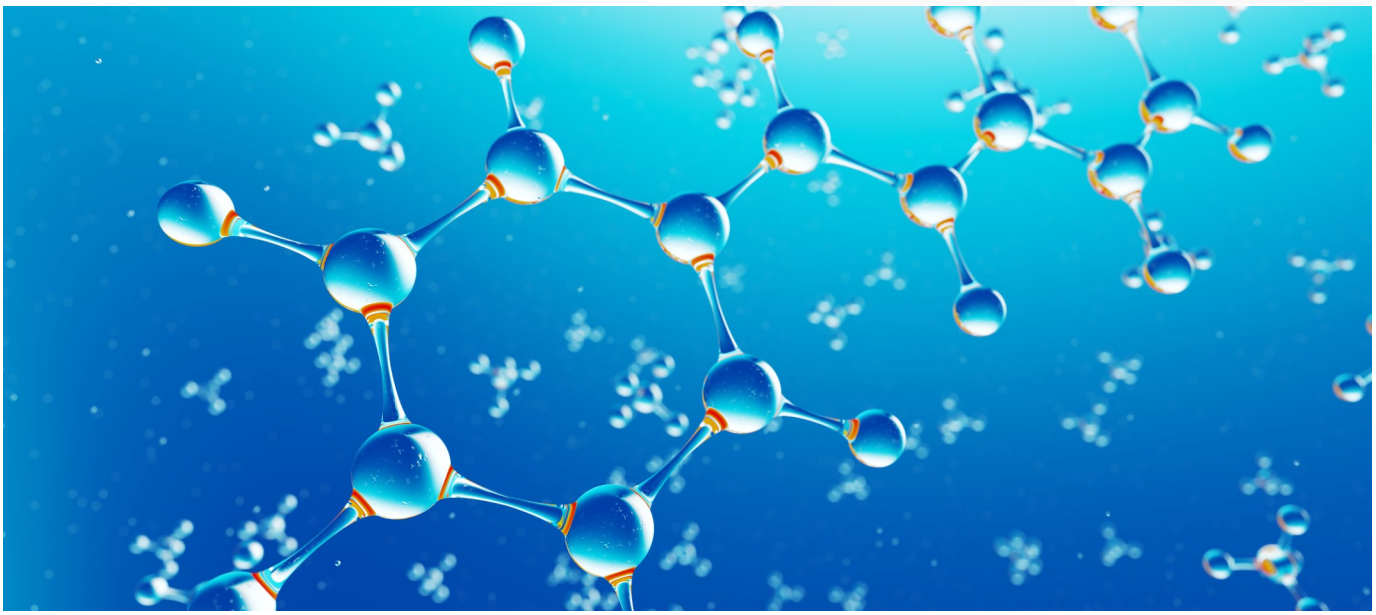
APPLICATION NOTE AN153



ASTM D7504 Standard Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography and Effective Carbon Number.

KEY WORDS: Impurities, Monocyclic Aromatic Hydrocarbons, Carbon Number

INTRODUCTION

SCION Instruments offers the solution for trace impurities in monocyclic aromatic hydrocarbons by gas chromatography and effective carbon number.

After manufacturing products might contain impurities. The ASTM D7504 describes the determination of total nonaromatic hydrocarbons and monocyclic aromatic hydrocarbons in benzene, toluene, ethylbenzene, p-xylene, o-xylene, m-xylene and styrene. This method also calculates the purity of these components.

This application is applicable on the SCION Instruments 4X6 and the new 8X00 GC-platform, Figure 1 shows the new SCION Instruments 8X00 GC platform.

Figure 1. SCION Instruments 8300 and 8500 GC platform equipped with the 8400PRO autosampler.

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RESULTS AND DISCUSSION

The ASTM D7504 is a method for the determination of the purity, and total nonaromatic hydrocarbons and monocyclic aromatic hydrocarbons in benzene, toluene, ethylbenzene, p -xylene, o-xylene, m-xylene and styrene.

After manufacturing the product might contain trace impurities, this analysis can determine the type and amount of these hydrocarbon impurities. The limit of detection (LOD) of this method is 0.0002 mass % and limit of quantitation is 0.0006 mass % for impurities in toluene, m-xylene, p-xylene, o-xylene, ethylbenzene, benzene and styrene. This test method is suitable for setting impurity specifications and for the use of an internal quality control.

Typical impurities that can be found in the products are: alkanes (1 to 10 carbon atoms), benzene, toluene, ethylbenzenes, xylenes and aromatic hydrocarbons (>9 carbon atoms).

When looking at interferences it shows that the complete separation of p-Xylene, ethylbenzene and m-Xylene can be problematic when ethylbenzene or p-Xylene is analysed. According to ASTM D7504 the separation can be considered adequate when the distance from the baseline to the valley between two peaks is not greater than 50% of the peak height, this is determined by the lowest of the two peaks.

It is important to check the separations of these components with a check sample before implementing the method.

Table 1. Analytical conditions

Injector	Split 100:1, 270 °C
Column	SCION-WAX-MS
Oven Program	60°C (10 min), 5°C/min to 150°C (10 min)
Carrier	Helium
Flow	1.5 ml/min (constant flow)
Detector	FID with ceramic jet, 300°C Air: 300 ml/min, Fuel gas (H_2): 30 ml/min, Make up (N_2): 28 ml/min
Inj. Volume	0.6 µl
Autosampler	8400PRO
Software	Compass CDS

In addition, the check sample can be used to determine the retentions times of the components.

This method uses the effective carbon number (ECN) correction factors to adjust the peak area of each component. From this the concentration of the components are calculated based on its relative percentages of total adjusted peak area and normalized to 100.0000%.

The repeatability of seven components was tested by injecting a custom made mix in tenfold. The results in table 2 show an excellent repeatability which meets the requirements set by ASTM D7504.

Figure 2 shows an example of three different samples in different matrixes, this also shows the separation between the critical components.

Table 2. Repeatability of ten consecutive injections using a custom made mix

	Non Aromatics	Benzene	Toluene	Ethylbenzene	p-Xylene	m-Xylene	o-Xylene
1	30819638	3389198	8039961	244212	223486	525397	297873
2	30884288	3427785	8122913	245452	225638	530388	299355
3	30691867	3376218	8028118	242002	223163	524414	295169
4	30659281	3368571	7997231	239844	222121	521916	292517
5	30550260	3361696	7972463	238071	220905	519089	291257
6	31115455	3421501	8109568	241633	224984	528925	294970
7	30782617	3388238	8030176	238388	222638	523070	291161
8	31032004	3406368	8109812	240582	225248	529281	293952
9	30653647	3363272	8011797	236946	222246	522349	291005
10	30882127	3397718	8078566	237804	223557	525068	290279
Rsd %	0.55	0.65	0.61	1.12	0.65	0.66	1.00

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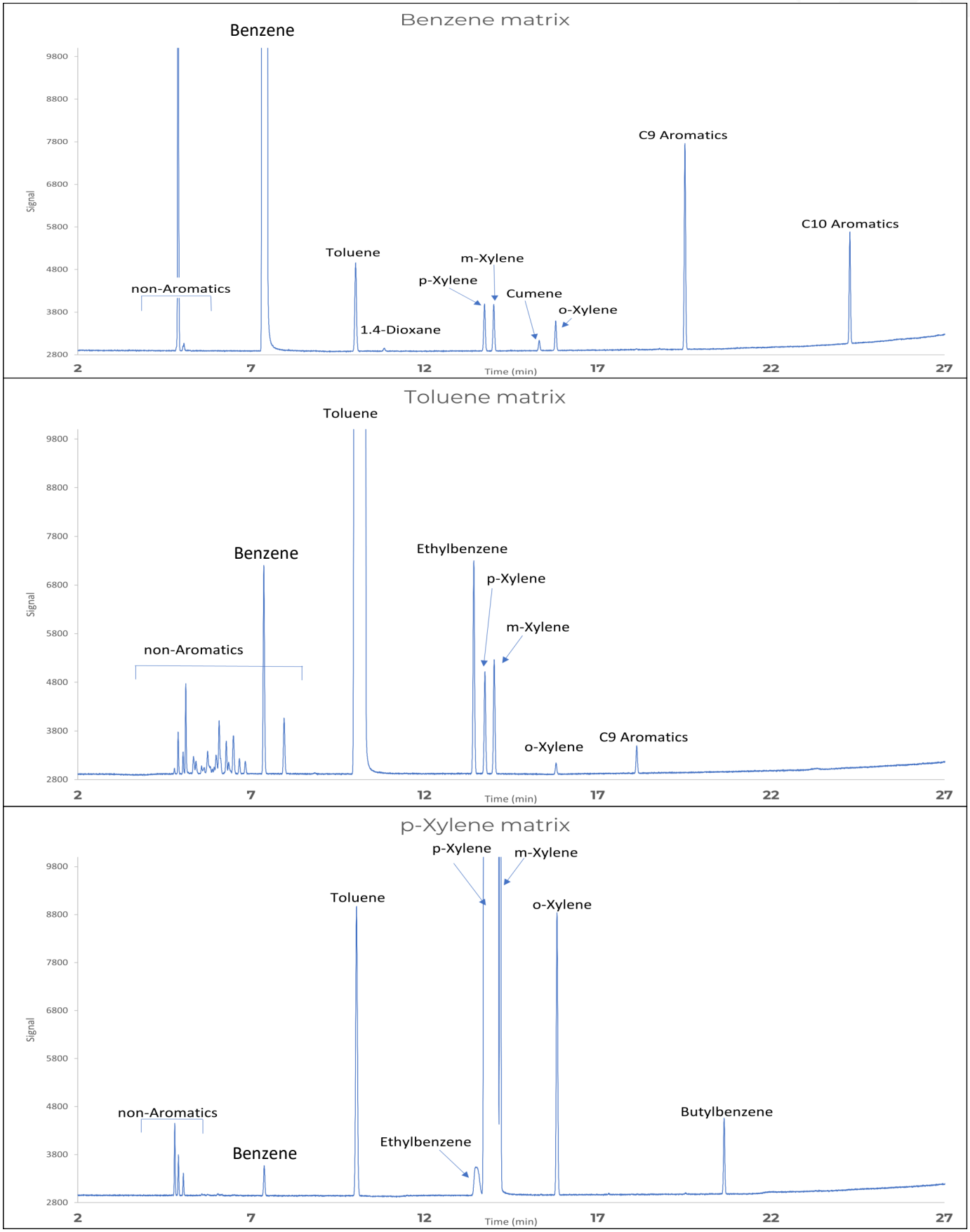

Figure 2. Example chromatograms of different sample matrixes.

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From the p-Xylene sample the %WT was calculated, these results are shown in table 3.

Table 3. example results of p-Xylene

	%WT
Non Aromatics	0.0084
Benzene	0.0028
Toluene	0.0353
Ethylbenzene	0.0095
p-Xylene	99.7877
m-Xylene	0.1212
o-Xylene	0.0282
Butylbenzene	0.0068

CONCLUSION

The SCION 8X00-GC analyser is perfectly capable of analysing trace impurities in monocyclic aromatic hydrocarbons with excellent repeatability and in compliance with ASTM D7504.

The equipment of the 8X00-GC analyser is pre determined. Ordering information can be found in the table below, for customisation please contact your local sales representative.

Although the 4X6-GC series is not shown in this application note, it is possible to perform this analysis on the SCION Instruments 4X6 GC series.

ORDER INFORMATION

Part number	Available standards
ST000032	D7504 AROMATIC MULTI-COMPONENT CHECK STANDARD IN BENZENE - 10X2 ML
ST000033	D7504 AROMATIC MULTI-COMPONENT CHECK STANDARD IN P-XYLENE - 10X2 ML
ST000034	D7504 AROMATIC MULTI-COMPONENT CHECK STANDARD IN TOLUENE - 10X2 ML

Part number	Available systems
41312408	ASTM D7504 analyzer based on 8300-GC 120V with 8400Pro AutoSampler
41312409	ASTM D7504 analyzer based on 8300-GC 120V with 8410Pro AutoSampler
41312410	ASTM D7504 analyzer based on 8300-GC 230V with 8400Pro AutoSampler
41312411	ASTM D7504 analyzer based on 8300-GC 230V with 8410Pro AutoSampler

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