CHEMICAL PURITY ANALYSIS

Technology Advantage: Agilent Intuvo 9000 GC with FID

Introduction

Monocyclic aromatic hydrocarbons are important commodity chemicals used to manufacture polymers. ASTM Committee D16 designates purity specifications for many of these chemicals. The ASTM D7405 method supports these specifications by using gas chromatography to measure overall chemical purity and the content of key impurities. These analyses are often performed by manufacturing technicians who are not trained analytical chemists. To simplify the technique while maintaining precision, the D7504 method eliminates sample preparation and instrument calibration by using Effective Carbon Number (ECN) responses. For this technique to be effective, sample components from 10^{-4} to 99.5 weight % must be detected in a single run.

The Agilent Intuvo 9000 GC is Designed to Make Routine Chemical Purity Analysis Fast and Easy

- Click-and-run column design removes the need for expert column installation skills.
- An auto-ranging flame ionization detector can quantitatively measure peak response across a large concentration range without overload.
- Small footprint and low resource usage is suited for production labs.
- Simple, intuitive touchscreen interface.

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Methodology

GC Instrument Conditions

Parameter	Value
Inlet	100:1 split, 270 °C
Injection volume	0.5 µL
Column	Agilent Intuvo HP-Innowax, 60 m × 0.32 mm, 0.5 µm (p/n 19091N-216-INT)
Constant column flow	2.1 mL/min helium
Column temperature	60 °C for 10 minutes 5 °C/min to 150 °C hold 10 minutes
Detector	Flame ionization 300 °C

Results and Discussion

Figure 1 shows 10 sequential injections of a mixed xylene sample. In a single injection, the Agilent Intuvo 9000 GC flame ionization detector (FID) is able to quantify the four large C8 aromatic peaks along with the smaller impurities. Table 1 shows the quantitative results. Single lab precision was met for all components found in this sample.

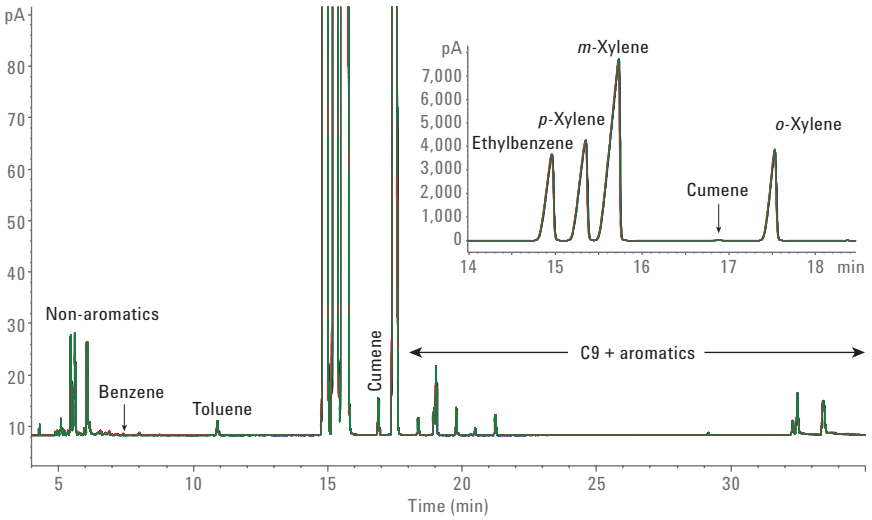

Figure 1. Ten overlays of mixed xylene analysis. Autoranging FID quantitatively detects small and large peaks in a single run.

Table 1. Results for 10 D7504 analyses of mixed xylenes.

Run	Nonaromatics wt %	Toluene wt %	Ethylbenzene wt %	*p*-Xylene wt %	*m*-Xylene wt %	Cumene wt %	*o*-Xylene wt %	C9+ Aromatics wt %
1	0.1982	0.0101	16.84	21.05	46.43	0.0221	15.29	0.1667
2	0.1991	0.0100	16.83	21.05	46.43	0.0219	15.30	0.1662
3	0.1986	0.0100	16.84	21.05	46.43	0.0218	15.29	0.1646
4	0.1994	0.0100	16.83	21.05	46.43	0.0219	15.30	0.1595
5	0.1984	0.0100	16.83	21.04	46.43	0.0219	15.30	0.1623
6	0.1993	0.0101	16.84	21.04	46.43	0.0219	15.31	0.1651
7	0.2008	0.0100	16.83	21.04	46.43	0.0219	15.30	0.1632
8	0.1998	0.0101	16.84	21.04	46.43	0.0219	15.30	0.1566
9	0.2005	0.0100	16.83	21.04	46.43	0.0219	15.30	0.1624
10	0.2005	0.0100	16.83	21.04	46.43	0.0219	15.31	0.1633
Mean	0.1995	0.0100	16.83	21.04	46.43	0.0219	15.30	0.1630
StdDev	0.00092	0.00005	0.004	0.004	0.003	0.00007	0.006	0.00307
ASTM SD	0.00700	0.01400	0.007	0.029	0.021	0.00003	0.010	0.00100

A high purity toluene sample was also run to demonstrate the large, automated response range of the Intuvo 9000 FID (Figure 2). Note that the detector is able to respond to peaks from 0.16 pA to 13,500 pA. This represents a concentration difference from 18 ppm to 99.97 weight %.

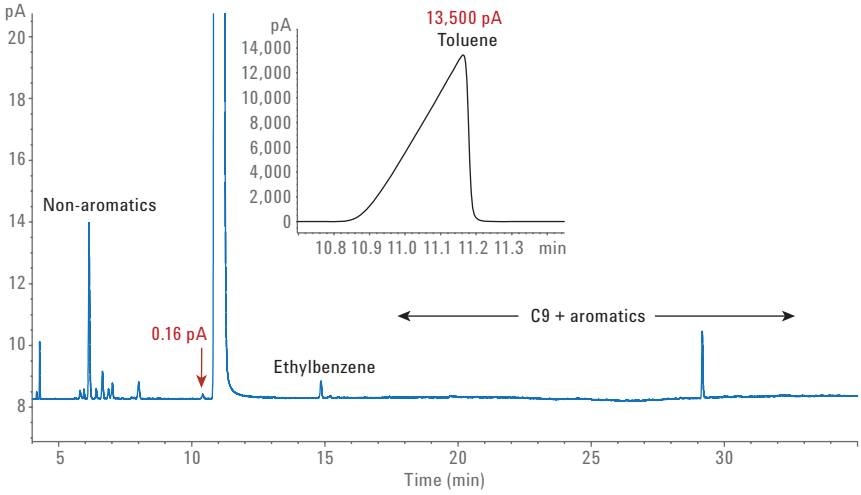

Figure 2. D7504 Analysis of purified toluene. Very large components (99.97 %) and very small components (18 ppm) were measured in a single run.

Conclusion

The chemical industry has recognized the need for simple, easy-to-use methods for the routine GC analysis of monocyclic aromatics. A significant development towards this goal was the development of ASTM method D7504, which uses ECN response to eliminate the need for expert sample preparation and calibration. To support this drive for easier GC methods, the Agilent Intuvo 9000 GC incorporates click-and-run column installation technology so that operators with any level of experienced can quickly and correctly change GC columns. Additionally, the auto-ranging FID makes implementing ECN response methods easy and effective, providing analysis results with a high level of precision.

Reference

1. ASTM D7504-15e1, Standard Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography and Effective Carbon Number, ASTM International, West Conshohocken, PA, 2015, www.astm.org

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