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Gas Chromatograph Brevis™ GC-2050

Application News

Aromatic Component Analysis of Gasoline According to ASTM D5580 Using the Brevis GC-2050 Gas Chromatograph

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User Benefits

- Levels of aromatics in gasoline containing many difficult-to-separate impurities were determined using ASTM D5580.
- The levels of specific aromatics were determined by using two columns with different polarities and different flow path switching times.
- This system, which includes the compact GC-2050 gas chromatograph, can determine oxygenates in gasoline (using ASTM D4815) and a variety of different gasoline additives.

■ Introduction

Aromatics, such as toluene and xylene, are sometimes added to gasoline to improve the octane number. Measuring the levels of these aromatics is essential for quality control in gasoline production. It is also important for environmental protection, in particular, for reducing emissions of benzene, a hazardous substance, as well as for sustainable energy use that reduces the environmental impact of gasoline while maintaining its performance.

ASTM D5580[1)] is a standard test method that uses gas chromatography to measure aromatics in gasoline. Gasoline aromatics can be separated more effectively by switching between two gas chromatography columns of different polarities and venting non-aromatic components from the system. This Application News describes using the Brevis GC-2050 gas chromatograph to determine aromatics in gasoline by applying ASTM D5580.

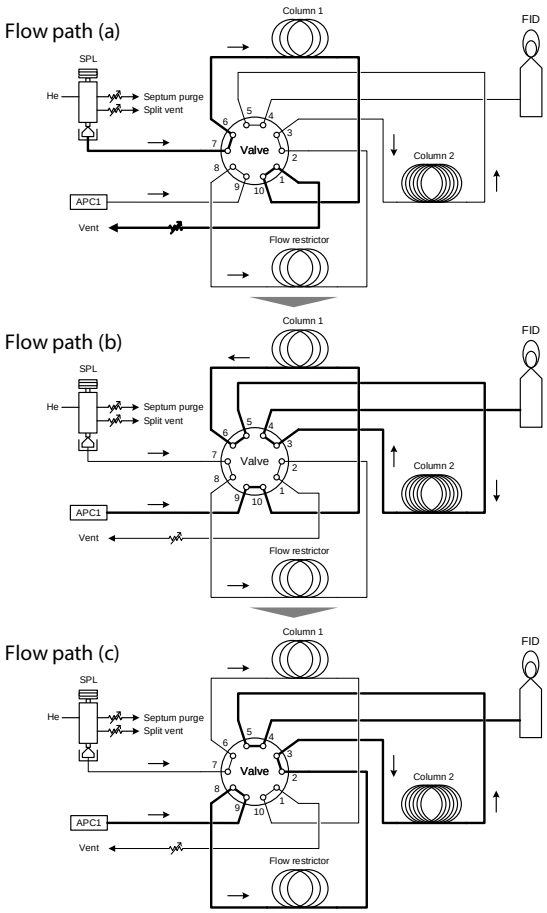

Fig. 1 Analysis Process

■ Outline of Analysis Process

This method uses ASTM D5580 to analyze a single sample using two columns, as described below. Benzene, toluene, and ethylbenzene are determined using **Analysis Conditions A**, and *p*/*m*-xylene, *o*-xylene, and C_9+ aromatics are determined using **Analysis Conditions B**. Gasoline aromatics that are separated in column 1 are either sent to column 2 or an FID detector in the order of flow paths (a), (b), and (c) (Fig. 1).

Analysis Process:

(a): The injected sample is passed through a sample injection unit and into a polar column (column 1). Aromatics are retained in the column, and non-aromatics that would affect the determination of aromatics are vented.

(b): The system is switched to flow path (b) just before benzene is vented from column 1 under **Analysis Conditions A** and just before 2-hexanone (I.S.) is vented from column 1 under **Analysis Conditions B**. Note that attaching an optional thermal conductivity detector (TCD) to the vent outlet provides an effective method of determining when to switch flow paths. The flow is reversed, and the gasoline components remaining in column 1 are sent to a non-polar column (column 2). Each aromatic is then eluted in order of boiling point from the non-polar column.

(c): The flow is reversed again, and the gasoline components remaining in column 2 are introduced to the FID detector without separation. C_9+ aromatics are determined at this point under **Analysis Conditions B**.

■ Equipment Configuration and Analytical Conditions

The system comprises a Brevis GC-2050 gas chromatograph, an AOC-30 for sample injection, and a VB-30-S heated valve box. Aromatics were determined using two sets of Analysis Conditions that were tailored to measure different components (Table 1). Note that the timing of the flow path switching and other conditions needs to be adjusted according to the columns being used and the operating environment.

Table 1 Example Analytical Conditions

INJ. Volume:	1 µL
INJ. Temperature:	200 °C
Career Gas:	He
INJ. Pressure:	130 kPa
Split Ratio:	11:1
Septum Purge:	3.0 mL/min
Column 1:	TCEP 20 % micro-packed column (0.6 m, I.D. 0.8 mm)
Column 2:	SH-1 (30 m, I.D. 0.53 mm, 3.0 µm)
FID Temperature:	250 °C
FID Make Up Flowrate:	24 mL/min (He)
FID H_2 and Air Flowrate:	H_2 32 mL/min, Air 200 mL/min
VB-30-S:	150 °C
Analysis Conditions A	
Column Temperature:	60 °C (6.0 min) – 2.0 °C/min – 115 °C
Flow Path Program:	(a) 0.0 to 1.3 min (b) 1.3 to 16.0 min (c) 16.0 to 33.5 min
APC1:	130 kPa (16 min) – 20 kPa/min – 180 kPa (15 min)
Analysis Conditions B	
Column Temperature:	60 °C (6.0 min) – 2.0 °C/min – 115 °C (10 min)
Flow Path Program:	(a) 0.0 to 3.9 min (b) 3.9 to 26.0 min (c) 26.0 to 43.5 min
APC1:	130 kPa (26 min) – 20 kPa/min – 180 kPa (15 min)

■ Determining Aromatics in Gasoline

A gasoline sample was prepared by adding 2 to 10 wt% aromatics and 10 wt% of an internal standard (2-hexanone) and then analyzed under **Analysis Conditions A** and **Analysis Conditions B**. Fig. 2 shows the chromatograms obtained under each set of Analysis Conditions. Despite gasoline containing large amounts of hydrocarbons, oxygenates, and other impurities, these impurities did not appear as peaks in the chromatograms, and only the aromatics added to the gasoline were detected. These results verify the utility of using flow path switching to effectively utilize the characteristics of two different columns.

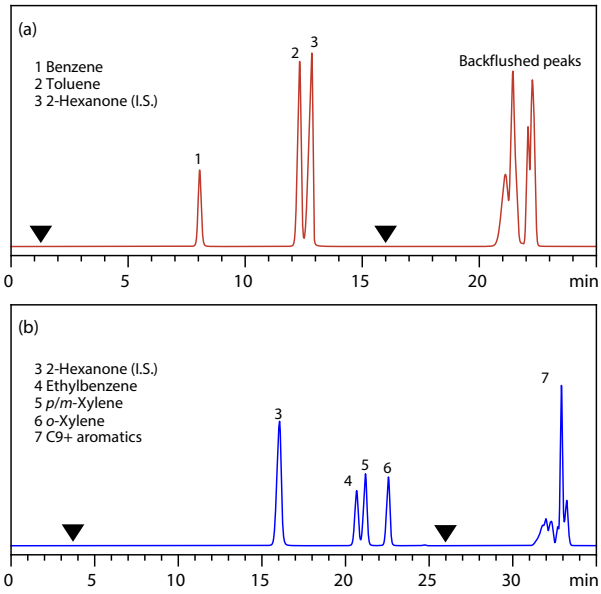

Fig. 2 Chromatograms of Aromatics Added to Gasoline
(a) Recorded under **Analysis Conditions A** and (b) recorded under **Analysis Conditions B**. Flow paths were switched at the solid arrows (▼).

■ Assessing Linearity for Each Aromatic

Linearity in the measurement range was verified for each aromatic. Single-component reagents were used to prepare samples of each aromatic at five concentrations. An internal standard (2-hexanone) was also added to each sample to 10 wt%, and these mixtures were analyzed. Note that *p*/*m*-xylene was determined using the calibration curve for *o*-xylene, and C_9+ aromatics were determined using the calibration curve for 1,2,4-trimethylbenzene.
The five calibration curves prepared are shown in Fig. 3. The correlation coefficient (R^2) of each calibration curve was at least 0.999, showing good linearity.

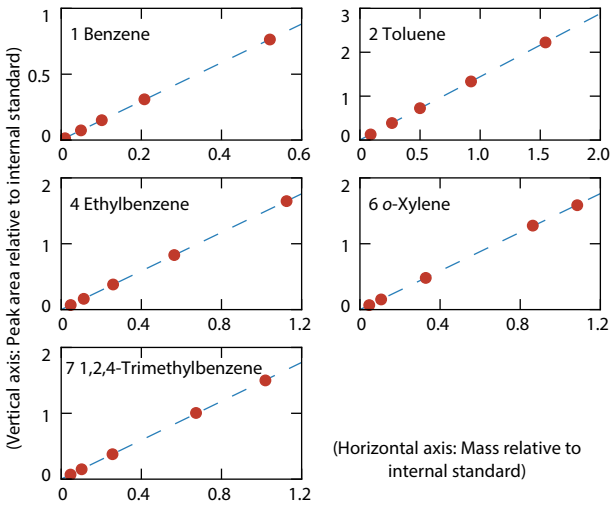

Fig. 3 Calibration Curves for Each Aromatic

■ Verifying Reproducibility

The accuracy of the within-lab repeatability and between-lab reproducibility of the analytical instruments was evaluated based on ASTM D5580. Using isooctane as a solvent, samples of each aromatic were prepared at concentrations close to those described in ASTM D5580, and each sample was measured 10 times in succession using **Analysis Conditions A** and **Analysis Conditions B**. The concentrations of aromatics in each sample were determined using the calibration curves mentioned earlier. Table 2 shows the mean concentration after 10 measurements, the maximum difference between this mean concentration and the measurements, and the permissible difference for each aromatic according to ASTM D5580. The maximum difference between the mean and measured concentration was within the permissible difference for all aromatics (Table 2).

Between-lab reproducibility was verified by analyzing the calibration samples that contained aromatics of known concentrations. Six samples of the six aromatics were analyzed under the two sets of conditions. The maximum difference between the mean concentration and the known concentration of each aromatic is shown in Table 2. The maximum difference was larger with these calibration samples than with the samples prepared to verify within-lab repeatability. But as with within-lab repeatability, all aromatics were within the permissible difference according to ASTM D5580.

Table 2 Within-Lab Repeatability and Between-Lab Reproducibility for Aromatic Samples

	Aromatic Component	Conc. [wt%]	Max. Conc. Difference [wt%]	ASTM D5580 Permissible Difference
Repeatability				
1	Benzene	1.426	0.017	0.033
2	Toluene	2.576	0.014	0.048
4	Ethylbenzene	0.979	0.004	0.029
5	*p*/*m*-Xylene	2.776	0.013	0.071
6	*o*-Xylene	0.825	0.005	0.027
7	C9+	8.524	0.063	0.198
Reproducibility				
1	Benzene	0.450	0.028	0.073
2	Toluene	9.000	0.141	0.278
4	Ethylbenzene	0.900	0.030	0.163
5	*p*/*m*-Xylene	4.000	0.182	0.452
6	*o*-Xylene	2.250	0.051	0.175
7	C9+	9.000	0.244	0.991

■ Conclusion

A flow path switching valve was installed on a Brevis GC-2050 gas chromatograph to enable the determination of gasoline aromatics (benzene, toluene, ethylbenzene, *p*/*m*-xylene, *o*-xylene, and C_9+ aromatics) according to ASTM D5580.

This system can switch between different columns during analysis to determine specific aromatics from among the many compounds present in gasoline. Analysis was performed under two sets of conditions, each using different flow path switching times. This method met the requirements for linearity and reproducibility described in ASTM D5580. The timing of flow path switching can also be adjusted to allow the targeted determination of oxygenates in gasoline. The ability to perform multiple analyses on a single system means the Brevis GC-2050 offers even greater functionality as a compact gas chromatograph.

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