Analysis of aromatic compounds in gasoline with flow-switching comprehensive two-dimensional gas chromatography

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Abstract

A comprehensive two-dimensional gas chromatography (GC × GC) instrument has been created by coupling a flow-switching modulator and a standard gas chromatograph. The instrument was used to characterize the aromatic composition of gasoline. The high-resolution separation produced by flow-switching GC × GC allowed gasoline aromatics to be fully resolved from saturated components. The aromatic compounds were further separated into groups having the same carbon number. A standard gasoline sample was analyzed to evaluate the quantitative accuracy and precision of this technique. The data show that flow-switching GC × GC produces results that are comparable to gas chromatography–mass spectrometry (GC–MS) and thermal modulation GC × GC. The simple, low-cost, and robust nature of flow-switching GC × GC makes it an ideal technique for the routine analysis of aromatic compounds in gasoline.
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1. Introduction

Comprehensive two-dimensional gas chromatography (GC × GC) has emerged as a powerful tool for separating complex mixtures of organic compounds. However, GC × GC has not been widely adopted for routine analysis. This may be due, in part, to the fact that commercially available GC × GC systems employ cryogenic modulators. Such instruments produce separations with unsurpassed resolution but also require substantial quantities of liquid cryogen and compressed gases. A simple flow-switching modulator has been developed that generates GC × GC separations without additional consumables [1,2]. The device is similar in construction to a Deans switch [3]. The ultimate peak capacity of flow-switching GC × GC is less than that of cryogenic modulation GC × GC, but the simplicity of the instrumentation makes it a potentially powerful approach for routine analysis.

A simple GC × GC system would be a valuable tool in the petrochemical processing industry. Numerous arti-

cles have been published on the application of GC × GC analysis to complex hydrocarbon mixtures [4–26]. Thermal modulation GC × GC has been shown to provide comprehensive group-type separations of fuels and petroleum distillates [4–6,8–11,13–22,24,26]. Valve-based GC × GC coupled with chemometrics has been used to extract specific information on select classes of compounds [7,15,23,25]. This article describes the use of flow-switching GC × GC to determine the aromatic content of finished gasoline.

Accurate and precise analytical methods for aromatics in gasoline are required for both regulatory and product quality needs. The 1990 Clean Air Act and subsequent amendments have resulted in the development of a reformulated gasoline program aimed at reducing toxic air emissions. The act limits the amount of benzene in fuels to no more than 1% (v/v) and total aromatics to no more than 25% (v/v). During gasoline production, refiners add aromatic compounds and monitor higher boiling alkylbenzenes and naphthalenes to adjust the octane rating and monitor refining processes.

Currently there are standard gas chromatography (GC) methods used to measure some or all of the aromatic content

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in gasoline. The American Society for Testing and Materials (ASTM) methods D3606 [27] and D5580 [28] use flame ionization GC with multiple packed or capillary columns to measure benzene, toluene and an aggregate of higher aromatics. While these methods are generally easy to use, they do not provide accurate data beyond benzene and toluene measurements, especially for fuels with high olefin content. In Europe, a heart-cutting two-dimensional GC method, EN12177 [29], is used to selectively measure benzene in gasoline. ASTM Method D5769 [30] uses single column GC–MS to selectively detect benzene and a number of individual alkylbenzenes and naphthalenes. While these methods meet the regulatory requirements for benzene and total aromatic measurements, they do not provide the detailed class analysis needed by chemical engineers to optimize gasoline production.

The primary goal of this study is to evaluate the efficacy of flow-switching GC × GC as a tool for characterizing the aromatic composition of gasoline. The accuracy, precision, and overall performance of this technique will be compared to that produced by GC–MS and thermal modulation GC × GC.

2. Experimental

The GC × GC system is essentially a standard gas chromatograph with a few additional components. A schematic of the system highlighting the custom components is shown in Fig. 1. The additional components are either hardware required for modulation or software required to analyze the two-dimensional chromatograms.

An Agilent Technologies (Wilmington, DE, USA) 6890N gas chromatograph equipped with dual flame-ionization detection (FID) systems was used as the experimental platform. Ultra-high purity hydrogen was used as the carrier gas in the primary and secondary columns. A $0.20\,\mu l$ quantity of neat sample was injected into the primary column through a split inlet (250:1 split ratio, 250 °C) with an Agilent 7683 Series Injector equipped with a $10\,\mu l$ syringe. A $15.0\,m \times 250\,\mu m$ DB-1 capillary column (dimethylpolysiloxane, $0.50\,\mu m$ film thickness, Agilent Technologies) was used as the primary column. The primary column flow was maintained at $1.0\,ml\,min^{-1}$ with a split inlet electronic pneumatic control (EPC) module. The primary column effluate passed into the flow-switching device for modulation. An auxiliary flow of $20.5\,ml\,min^{-1}$ also entered the flow-switching device. The auxiliary flow was regulated by an EPC module operated in constant flow mode. The flow exiting the switching device was divided equally between two secondary columns with a tee union. A $5.0\,m \times 250\,\mu m$ DB-Wax column (polyethylene glycol, $0.10\,\mu m$ film thickness, Agilent Technologies) and a $5.0\,m \times 250\,\mu m$ DB-1701 column (14% cyanopropylphenyl-methylpolysiloxane, $0.25\,\mu m$ film thickness, Agilent Technologies) were used as the secondary columns. Effluent from each secondary column was passed through a flame-ionization detector at 250 °C. The signals from the detectors were monitored at 200 Hz with Agilent ChemStation software. The oven temperature was held at 35 °C for 3.0 min, and then ramped to 250 °C at a rate of 8 °C min⁻¹. Custom software was used to convert the one-dimension signal arrays into two-dimensional chromatograms. Both secondary columns produced similar retention times, but the DB-Wax column produced narrower peaks. Only the chromatograms generated with the DB-Wax secondary column were considered in this study. Thus, the DB-1701 secondary column and second
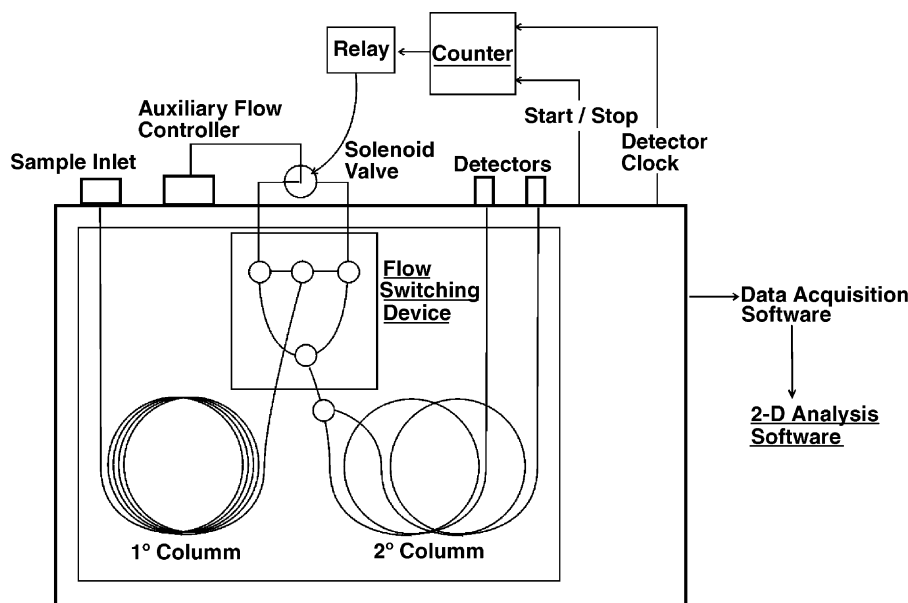

Fig. 1. Schematic of the flow-switching GC × GC apparatus. The system is mostly comprised of commercially available components. The required custom components (the flow-switching device, the counter circuit, and 2D analysis software) are underlined.
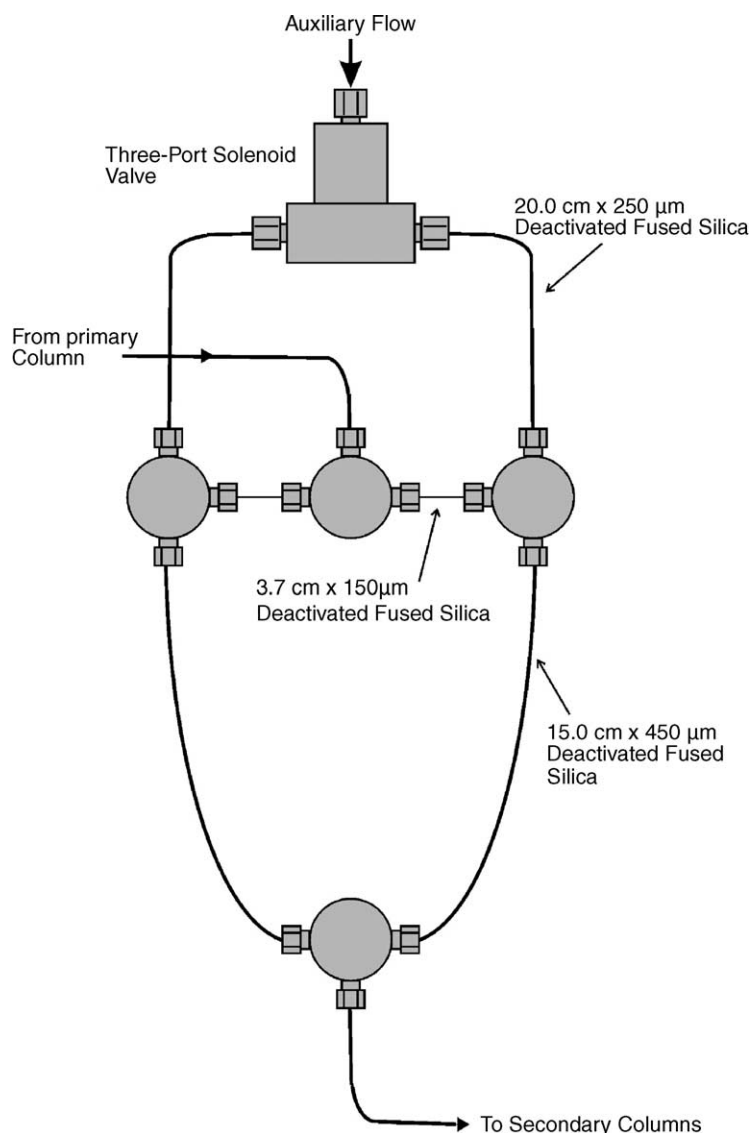
Fig. 2. Schematic of the flow-switching modulator.

FID were not required to perform the gasoline aromatic analysis.

A schematic of the flow-switching device is shown in Fig. 2. Detailed descriptions of the operating principles of the device have been published [1,2]. The device was constructed with deactivated fused silica tubing, four tee-unions (stainless steel, 0.25 mm i.d. orifices, VICI, Houston, TX, USA, part number MT.5CS6), and a three-port solenoid valve (Parker-General Valve, Fairfield, NJ, USA, part number 009-0284-900). An auxiliary flow of carrier gas was connected to the common port of the three-port solenoid valve. The solenoid valve was positioned outside of the column oven. The remainder of flow-switching device was mounted on a thin piece of stainless steel sheet metal and housed inside the column oven. The output ports of the solenoid valve were connected to the peripheral unions of the switching device with two pieces of 20 cm × 250 μm fused silica capillary tubing. The

downstream end of the primary column was connected to the center union. The center union was connected to the peripheral unions with two pieces of 3.7 cm × 150 μm deactivated fused silica capillary tubing. The peripheral unions were connected to the lower union with two pieces of 15 cm × 450 μm deactivated fused silica capillary tubing. The primary column flow and auxiliary flow exited the device through the lower union. During a chromatographic analysis, the solenoid valve was switched every 1.50 s by a custom counter/relay circuit. The timing of the valve switching was controlled by a 100 Hz clock signal from the chromatograph.

For the purpose of comparison, a reference gasoline sample was analyzed according to ASTM Method D5769 using an Agilent Technologies 5973*inert* GC/MS system. The instrument was equipped with a 60.0 m × 250 μm HP-1 capillary column (dimethylpolysiloxane, 1.0 μm film thickness, Agilent Technologies) and ultra-high purity helium was used

as the carrier gas with an initial average column flow rate of 2 ml min^{-1}. This capillary column was interfaced directly to the mass spectrometer source with no effluent splitting. A 0.10 μl quantity of neat sample was injected through a split inlet (250:1 split ratio, 250 °C) with an Agilent 7683 Series Injector equipped with a 5 μl syringe. The oven temperature was held at 60 °C for 0 min, and then ramped to 120 °C at a rate of 3 °C min^{-1}. A second oven ramp of 10 °C min^{-1} to 250 °C was applied. The electron ionization source was held at 250 °C and the ionization voltage was 70 eV. The quadrupole mass filter was run at full scan from 45 to 300 U with a scan rate of 2.89 scans s^{-1}.

3. Results and discussion

A two-dimensional chromatogram of a typical gasoline sample is shown in Fig. 3. The chromatogram has a structure similar to gasoline GC × GC chromatograms obtained with thermal modulation [9]. The chromatogram is displayed with two different maximum signal thresholds so that both the major and minor components can be seen. Peak widths at half maximum along the primary axis are approximately 4.0 s. Saturated hydrocarbons form a horizontal band at the bottom of the chromatogram. Alkanes have a peak width at half maximum along the secondary axis of 75 ms. This width is the theoretical minimum for a 1:20 differential flow ratio and a 1.5 s modulation period [2]. A small amount of peak tailing is observed along the secondary axis. Fig. 4 shows

a vertical slice of a 2D chromatogram at a primary retention time of 490.5 s. The large peak is due to toluene. Tailing is difficult to observe when the signal axis is scaled to display the entire peak (see the upper chromatogram in Fig. 4). However, tailing can be observed when signal axis range is decreased by a factor 20 (see the lower chromatogram in Fig. 4). The tail emerges from the side of the peak when the signal diminishes to approximately 1.5% of the maximum peak intensity. The area beneath the tail is approximately 3% of the total peak area. Similar peak shapes were observed for compounds with volatilities ranging from methane to n-hexacosane ($C_{26}H_{54}$) and polarities ranging from n-alkanes to aromatic alcohols. Based on these observations it has been concluded that the tailing is primarily produced by diffusion into and out of unswept regions within the modulator and not due to adsorption on the modulator surfaces.

The gasoline chromatogram in Fig. 3 clearly shows how monoaromatic hydrocarbons are fully separated from the saturated compounds and further separated into peak bands with the same carbon number. The monoaromatic compounds peaks have secondary widths at half maximum of 80 ms. Diaromatic compounds, such as naphthalene, 1-
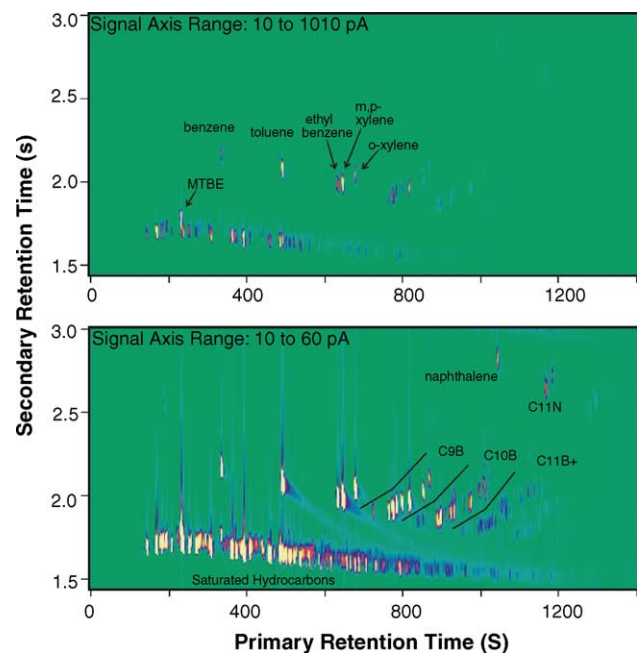

Fig. 3. Two-dimensional chromatograms of gasoline. The gasoline sample is a California Air Resources Board Phase II standard. The upper and lower chromatograms are from the same analysis, but the maximum signal has been decreased by a factor of 20 in the lower chromatogram to show the low intensity peaks.
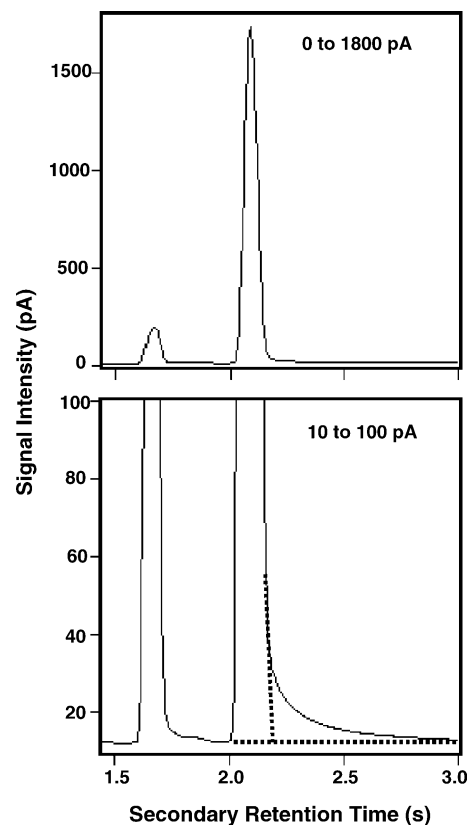

Fig. 4. A vertical slice of the gasoline chromatogram shown in Fig. 3 at a primary retention time of 490.5 s. The large peak with a secondary retention time of 2.09 s is due to toluene. The peak has a width at half maximum of 75 ms. The lower chromatogram shows the same data as the upper chromatogram, but the signal axis range has been decreased by a factor of 20 to show the magnitude of the peak tailing. The area of the tail represents approximately 3% of the total peak area.

methylnaphthalene, and 2-methylnaphthalene, exhibit the greatest secondary retention and are easily separated from the monoaromatic compounds. The diaromatic compounds have secondary widths of 85 ms.

The main goal of the analysis was to determine the aromatic composition of gasoline. Aromatic compounds were divided into 10 classes: benzene, toluene, ethylbenzene, *m*- and *p*-xylenes, *o*-xylene, C9 benzenes (C9B), C10 benzenes (C10B), benzenes with carbon number greater than or equal to 11 (C11B+), naphthalene, and methylnaphthalenes (C11N). Calibration curves were generated for these compound classes (see Table 1) using a set of standard mixtures. These solutions consisted of 23 aromatic compounds in isooctane (Spectrum Quality Standards, Houston, TX, USA, catalog number 5769 Cal-1). However, the calibration set did not contain any monoaromatic compounds with carbon number greater than or equal to 11, so the calibration curve for the C10B class was also used for the C11B+ class.

Each of the six calibration mixtures was analyzed once. Peaks in the FID signal array were integrated using Agilent ChemStation software. Custom software was used to determine the primary and secondary retention times of each peak and to assign each peak to a specific compound class. The total peak area within a compound class was plotted as a function of the reported liquid volume percentage (%, v/v). A summary of the calibration results is shown in Table 1. The calibration plots were well described by a linear trend line: all but one of the regressions had R^2 values greater than 0.999. However, the trend lines had *y*-intercepts that were slightly less than zero. This small but statistically significant trend was

possibly due to the nature of the standard solutions. An ideal set of calibration standards would have constant bulk properties with the composition of the solutions changing by no more than a few percent. However, the calibration solutions used in this study had drastic changes in composition with the most dilute mixture containing 6% aromatic compounds (the balance being isooctane) and the most concentrated mixture containing 100% aromatic compounds.

A quality control reference standard (Spectrum Quality Standards, catalog number AR3000) was used to check the accuracy of the calibration curves and the precision of GC × GC analysis. This mixture contained 14 compounds including benzene, toluene, *m*-xylene, *o*-xylene, ethylbenzene, 1,2,4-trimethylbenzene, 1,2,4,5-tetramethylbenzene, and naphthalene. These eight compounds allowed all of the calibration curves to be tested except for the C11N curve. The results from this study are shown in Table 2. Replicate analyses of the quality control reference standard ($n = 5$) produced relative standard deviations of total peak area that were generally less than 1%. The experimentally determined concentrations agreed with the reported values to within 0.1% (v/v).

The accuracy and precision of gasoline characterization was tested by analyzing a standardized gasoline sample (Spectrum Quality Standards, California Phase II gasoline, catalog number CALRR3). The results of replicate GC × GC analysis ($n = 5$) with GC × GC is shown in Table 3. The liquid volume percentages for each compound class had relative standard deviations that were less than 2% with the exception of the C11B+ class. The greater relative uncertainty of

Table 1
Calibration curve parameters resulting from the analysis of six standard mixtures

Group	Constituent compounds	Range of concentrations (%, v/v)	Slope	*y*-intercept	R^2
Benzene	Benzene	0.317–5.16	50.2 ± 0.8	−3.4 ± 2.4	0.9990
Toluene	Toluene	1.21–19.72	50.6 ± 0.6	−12.0 ± 6.8	0.9994
Ethylbenzene	Ethylbenzene	0.318–5.17	47.9 ± 0.2	−1.13 ± 0.45	1.0000
m,p-Xylene	*m*-Xylene, *p*-xylene	0.763–12.40	50.8 ± 0.4	−10.9 ± 2.7	0.9998
o-Xylene	*o*-Xylene	0.382–6.21	50.7 ± 0.4	−3.2 ± 1.4	0.9997
C9B	Isopropylbenzene *n*-Propylbenzene 3-Ethyltoluene 4-Ethyltoluene 1,3,5-Trimethylbenzene 2-Ethyltoluene 1,2,4-Trimethylbenzene 1,2,3-Trimethylbenzene Indan	1.868–30.38	49.3 ± 0.6	−8 ± 10	0.9994
C10B	1,4-Diethylbenzene *n*-Butylbenzene 1,2-Diethylbenzene 1,2,4,5-Tetramethylbenzene 1,2,3,5-Tetramethylbenzene	0.903–14.72	48.4 ± 0.8	−0.4 ± 7.0	0.9988
Naphthalene	Naphthalene	0.127–2.08	50.1 ± 0.6	−0.83 ± 0.76	0.9993
C11N	1-Methylnaphthalene 2-Methylnaphthalene	0.258–4.20	50.0 ± 0.9	−1.6 ± 2.2	0.9986

Regression parameters were derived from plots of total group area as a function of liquid volume percent.

Table 2

GC × GC and reported v/v (%) values for aromatic check standard

Compound	GC × GC (%, v/v)[a]	Actual (%, v/v)[b]
Benzene	0.876 ± 0.009	0.83
Toluene	7.66 ± 0.07	7.59
m-Xylene	2.65 ± 0.02	2.54
o-Xylene	2.48 ± 0.02	2.49
Ethylbenzene	2.55 ± 0.02	2.53
1,2,4-Trimethylbenzene (C9B)	2.62 ± 0.02	2.51
1,2,4,5-Tetramethylbenzene (C10B)	1.68 ± 0.01	1.65
Naphthalene	0.74 ± 0.01	0.74
Total aromatic compounds	21.3	20.9

[a] Liquid volume percentages generated using the calibration curve data shown in Table 1. Reported uncertainties represent the precision of the replicate analysis with $n = 5$. Uncertainties are reported as ± one standard deviation.

[b] The manufacturer reports that their concentration values have relative error of 2%.

C11B+ is most likely because this group was comprised of many low intensity peaks.

The GC × GC results for benzene and total aromatic compounds were compared to the values reported for the gasoline standard (see Table 3). The reported values were based on results from 25 refinery laboratories employing ASTM Methods D5769 and D5580. The GC × GC values of 0.825% (v/v) for benzene and 22.5% (v/v) for total aromatic compounds were well within the respective ranges determined by both ASTM Methods D5769 and D5580.

A more detailed evaluation can be obtained by comparing the GC × GC and GC–MS results. Nearly identical volume percentages were obtained for all of the aromatic classes with the exception of C10B and C11B+. Despite discrepancies between the higher molecular weight alkylbenzenes, similar values for total aromatic compounds were found with GC–MS and flow-switching GC × GC.

In the case of the C10B and C11B+ groups, the GC × GC values were somewhat higher than the GC–MS results. It is most likely that the numerous, low-intensity peaks were underestimated by GC–MS for several possible reasons.
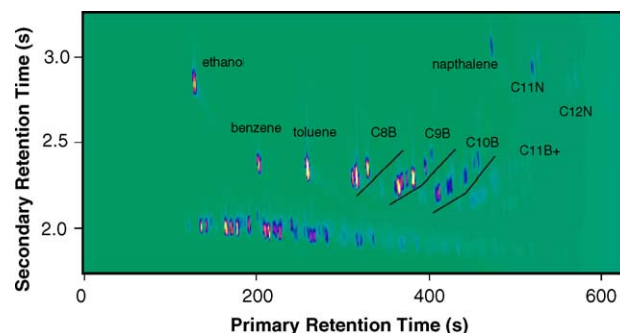

Fig. 5. Two-dimensional chromatogram of gasoline. The chromatogram was obtained with a thicker film DB-Wax secondary column that allowed a faster temperature ramp to be used. This resulted in a total separation time of approximately 10 min.

Since mass spectral detectors have less dynamic range than flame detectors, the sensitivity of the GC–MS method was reduced to obtain better results for higher concentration components such as toluene and C8 benzenes [31]. This can result in under reporting the higher molecular weight alkylbenzenes (such as the C9B, C10B and C11B+ classes) that are distributed among a large number of isomers at low concentration. Another reason lies in the GC–MS quantification using single extracted ion profiles for each compound class. For those C9B, C10B and C11B+ isomers not in the calibration mixture, a representative response factor based on one isomer was used for quantification, which assumes an identical fragmentation pattern and intensity for each compound in the class. This may introduce some error, especially when using a C10B response for all C11B+ compounds. In contrast, flame ionization detector responses are generally more uniform and stable over time.

The high resolving power of GC × GC analysis can be used to reduce analysis time. A separate set of gasoline analyses were performed using a 5.0 m × 250 μm DB-Wax secondary column with a 0.25 μm film thickness. The thicker film allowed similar secondary retention to be obtained with a faster temperature ramp rate of 21 °C min^{-1}. Fig. 5 shows a chromatogram obtained for a gasoline sample obtained from

Table 3

Results for California Reference Gasoline compared to reported results for ASTM Methods D5580 and D5769, and the GC–MS analysis

Compound/group	GC × GC (%, v/v)	D5580 (%, v/v)[a]	D5769 (%, v/v)[a]	GC–MS (%, v/v)[b]
Benzene	0.825 ± 0.007	0.79 ± 0.11	0.81 ± 0.18	0.80 ± 0.01
Toluene	5.50 ± 0.04			5.77 ± 0.11
Ethylbenzene	1.97 ± 0.01			1.87 ± 0.02
m/p-Xylenes	5.05 ± 0.04			5.10 ± 0.05
o-Xylene	1.70 ± 0.01			1.67 ± 0.02
C9B	4.95 ± 0.05			4.51 ± 0.06
C10B	1.67 ± 0.02			1.45 ± 0.04
C11B+	0.45 ± 0.02			0.15 ± 0.01
Naphthalene	0.161 ± 0.002			0.15 ± 0.01
C11N	0.178 ± 0.002			0.18 ± 0.01
Total aromatic compounds	22.5 ± 0.1	22.4 ± 1.6	21.81 ± 2.3	21.65 ± 0.28

[a] Consensus analysis from round robin study (25 laboratories).

[b] GC–MS analysis, three replicates.

a local service station. Full separation of the aromatic classes is achieved in approximately 10 min.

The accuracy and precision of the flow-switching GC × GC analysis is comparable to values previously reported for thermal modulation GC × GC [9]. Simplicity is the key advantage that flow-switching GC × GC has over thermal modulation GC × GC: the flow switching modulator has inexpensive components, few moving parts, and does not consume liquid cryogen. However, it is important to note that the isolation of specific classes of aromatic compounds is a fairly easy separation for state-of-the-art GC × GC systems. Thus, the superior resolving power produced by current thermal modulators is not fully utilized. It is probable that more challenging hydrocarbon separations, such as separating olefins from saturated compounds, are better analyzed with thermal modulation.

4. Conclusions

This study shows that a standard gas chromatograph fitted with a flow-switching modulator can perform a detailed quantitative characterization of gasoline aromatic content. The accuracy and precision of the quantitative data generated from this analysis are similar to that produced by thermal modulation GC × GC. However, flow-switching GC × GC is well suited for routine implementation because high performance is obtained with a low-cost modulator that does not require additional consumables. It is probable that the quantitative performance could be improved further with the use of internal standards.

Flow-switching GC × GC compares favorably to established ASTM GC methods for the determination of benzene and total aromatic compounds in gasoline. The higher resolution of GC × GC avoids the interferences from olefins often encountered with standard GC methods and provides detailed measurement of individual classes of alkyl substituted benzenes and naphthalenes. In addition, the GC × GC approach uses a flame ionization detector, potentially making it more robust and stable than analyzers employing quadrupole mass spectrometers. This is particularly important for routine analysis.

Acknowledgements

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