Analysis of Environmental BTEX by NovaTest P100 Compact GC

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KEY WORDS

Environmental, BTEX, Air Monitoring, Compact Gas Chromatograph

ABSTRACT

This application note describes the detection of benzene, toluene, ethylbenzene, and xylenes (BTEX) using NovaTest P100 compact gas chromatograph (GC). Five BTEX compounds were separated in 3 min with excellent peak shape. Both retention time and peak area showed great reproducibility. When sampling time was 10 min, the method detection limits (MDLs) for BTEX were as low as 0.1 ppbv, which meets the requirements of most applications and can be further improved by increasing the sampling time. The linearities were evaluated by two experiments, one was changing sampling concentrations while keeping the sampling time constant, and the other was changing sampling times while keeping the gas standard concentration constant. Great linearities were obtained for both experiments with R^2 larger than 0.99. This application note demonstrates the features of NovaTest P100 compact GC: fast, robust and easy to operate.

INTRODUCTION

The increasing usage of organic chemicals causes copious amounts of volatile organic compounds (VOCs) released to the environment. Some of the VOCs showed side effects on human health, which are irritation, mutagen risk and cancer risk, etc. Benzene, toluene, ethylbenzene and xylenes (BTEX) are

commonly seen VOCs. The sources of BTEX include vehicle emission, industrial plants emission, construction materials degradation, painting solvent release from new furniture and so forth. A number of analytical methods have been developed for the detection of BTEX, which usually involved the use of canister sampling and sorbent tubes. Despite the popularity of canister sampling, the use of a compact analytical device for environmental air analysis provides easier operation, faster results, and lowers the risk of sample loss and contamination.

In this application note, the NovaTest P100 compact gas chromatograph (GC) with a photo ionization detector (PID) was used to analyze the BTEX in an office area. The linearity, reproducibility, detection limit and practical application performance of the NovaTest P100 compact GC were discussed.

ANALYSIS SETUP

The NovaTest P100 compact GC has pre-programmed built-in methods and is fully automated. The analysis was easily set up with the following steps:

1. Take the NovaTest P100 compact GC to the field and connect the device to a laptop through USB cable;
2. Turn on the carrier gas regulator to 15 psi. Power on the device;
3. Open the NovaSoft user interface, select Run Test → BTEX. The "Run Test" mode includes a list of built-in methods, where every programming parameter has been preset to enable users to run expedited tests;
4. Input a file name, operator name, and sampling time;
5. Start analysis by clicking on "Start".

Then the sample was pumped into the system, trapped by Nanova Environmental Inc. proprietary preconcentrator and then released to GC column for separation and PID for detection. A typical NovaTest P100 compact GC setup is shown in **Figure 1**.

Figure 1. NovaTest P100 compact GC connected with a laptop.

In compliance with Method TO-14 and the instructions from the canister merchandise, BTEX standard samples at concentrations of 2 to 2000 ppbv were prepared in canisters. The real air sample was analyzed in the field by directly pumping air into the NovaTest P100 compact GC.

All the programming parameters except the sampling time were preset in the NovaSoft system and no other manual input was required.

RESULTS

CHROMATOGRAPHY

Figure 2 shows the chromatograms of BTEX at concentrations of 8 and 40 ppbv. Different sampling times at each concentration were tested.

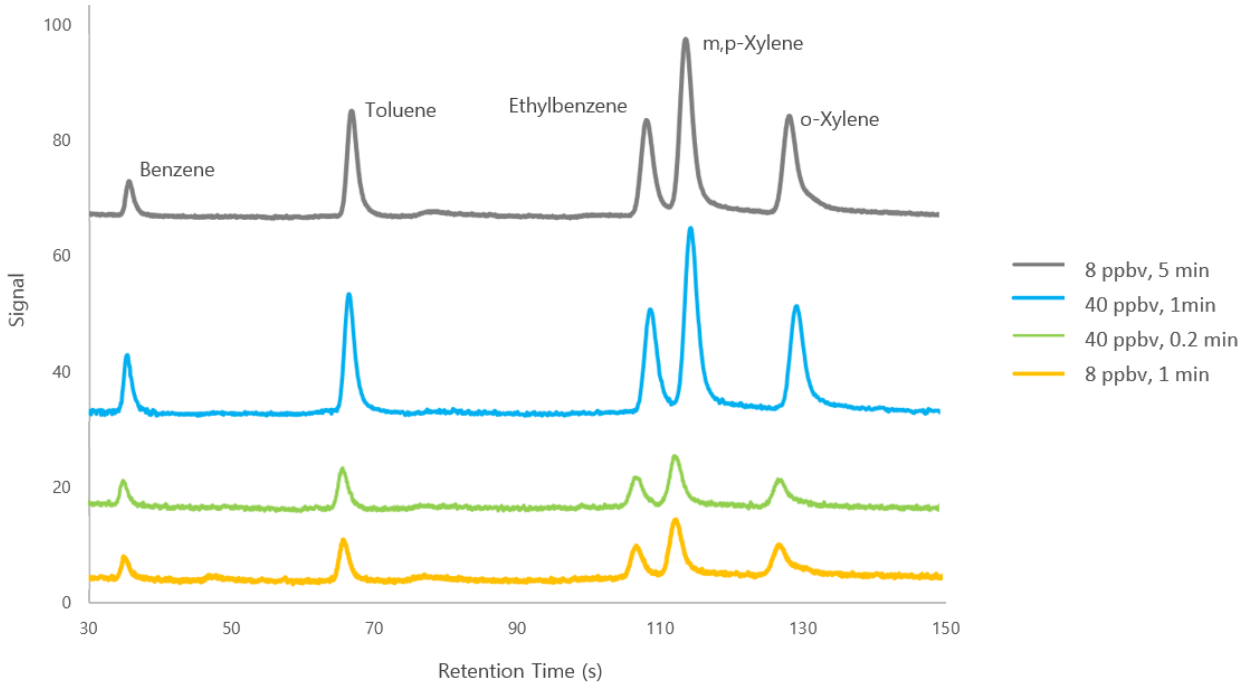

Figure 2. Representative BTEX chromatograms with NovaTest P100 compact GC

The BTEX samples were eluted within 3 min, with a full width at half minimum (FWHM) less than 5 s. Excellent peak shapes were obtained for all the samples tested. The peak area of 8 ppbv sample with 5 minutes' sampling time were similar to that of 40 ppbv sample with 1 minute's sampling time. A similar result was achieved for samples of 8 ppbv with 1 minute's sampling time and 40 ppbv with 0.2 minute's sampling time. These results indicate the feasibility of using one standard with different sampling times to calibrate the device.

REPRODUCIBILITY

The reproducibility of retention time (RT) and peak area for BTEX tests at different concentrations when using NovaTest P100 compact GC is shown in **Table 1.** All tests were repeated 3 times and the sampling time was 1 min.

Table 1. Reproducibility of retention time (RT) and peak area of BTEX over different concentrations using NovaTest P100 compact GC (n=3, sampling time=1 min).

4 ppbv for each compound				
Compound	**Avg. RT (s)**	**RT RSD (%)**	**Avg. Peak Area**	**Peak Area RSD (%)**
Benzene	35.39	0.27	3.52	9.77
Toluene	66.62	0.08	8.00	8.49
Ethylbenzene	108.86	0.20	6.19	1.59
m,p-Xylene	114.56	0.10	12.74	5.30
o-Xylene	129.31	0.17	9.40	5.33
40 ppbv for each compound				
Compound	**Avg. RT (s)**	**RT RSD (%)**	**Avg. Peak Area**	**Peak Area RSD (%)**
Benzene	35.39	0.27	32.61	4.40
Toluene	66.82	0.46	84.97	1.55
Ethylbenzene	109.31	0.23	89.83	3.58
m,p-Xylene	115.07	0.30	183.19	1.73
o-Xylene	130.11	0.35	114.95	5.56
200 ppbv for each compound				
Compound	**Avg. RT (s)**	**RT RSD (%)**	**Avg. Peak Area**	**Peak Area RSD (%)**
Benzene	35.17	0.63	140.24	1.80
Toluene	66.43	0.80	429.32	1.25
Ethylbenzene	108.45	0.96	439.13	0.55
m,p-Xylene	114.11	1.05	826.34	1.32
o-Xylene	129.12	1.08	470.93	0.55
1000 ppbv for each compound				
Compound	**Avg. RT (s)**	**RT RSD (%)**	**Avg. Peak Area**	**Peak Area RSD (%)**
Benzene	35.26	0.83	444.30	3.58
Toluene	66.69	0.94	2034.50	3.63
Ethylbenzene	108.51	0.76	2292.54	1.93

m,p-Xylene	114.14	0.76	4255.47	2.95
o-Xylene	128.96	0.75	2401.21	2.99

Excellent reproducibility was achieved. The relative standard deviation (RSD) of RT and peak area were less than 1.08 and 9.77 %, respectively. These results were well within the requirement of Method TO-14. In addition, the reproducibility of RT for each compound when changing sampling time was also compared (see **Table 2**). Various sampling times were tested, including 0.5, 1, 2, 5, 10 min. According to the results, with the tested sampling time, the RT RSD was less than 0.23 %

Table 2. Reproducibility of BTEX retention time across different sampling times.

Compound	Retention times (s) at different sampling times					Avg. (s)	STD	RSD (%)
	0.5 min	1 min	2 min	5 min	10 min			
Benzene	32.45	32.4	32.4	32.3	32.5	32.41	0.07	0.23
Toluene	57.79	57.74	57.65	57.46	57.65	57.66	0.13	0.22
Ethylbenzene	92.93	93.07	92.98	92.69	92.88	92.91	0.14	0.15
m,p-Xylene	97.73	97.97	97.97	97.58	97.78	97.81	0.17	0.17
o-Xylene	110.69	110.93	110.93	110.64	110.74	110.79	0.14	0.12

Another advantage of NovaTest P100 compact GC is that when using the built-in method of the NovaSoft, the concentration of each sample will be automated calculated and showed in the instant reports. **Table 3** shows the results of reported RT and concentration in NovaSoft for BTEX when injecting standard gas sample was at a concentration of 20 ppbv.

Table 3. Reported concentration in NovaSoft for BTEX when injecting standard gas sample at concentration of 20 ppbv across different sampling times.

Compound	Concentration (ppbv) reported in NovaSoft at different sampling times					STD	RSD (%)
	0.5 min	1 min	2 min	5 min	10 min		
Benzene	15.70	16.80	17.70	17.20	18.70	1.11	6.43
Toluene	15.90	17.20	18.00	18.60	19.60	1.40	7.85
Ethylbenzene	15.30	16.70	17.80	17.60	19.30	1.47	8.50
m,p-Xylene	17.20	17.40	18.10	17.20	18.60	0.62	3.53
o-Xylene	19.60	17.80	18.80	18.00	19.60	0.85	4.55

Compared to the true value (20 ppbv), the recoveries of all reported results were within the range of 76.5 to 98 %. Due to the limitation of sampling unit, the recoveries of reported concentration at low sampling times were less than those at higher sampling times. The RSD of reported concentration under different sampling time was less than 8.50%.

METHOD DETECTION LIMIT (MDL)

Method detection limits (MDLs) were determined at the concentration where the signal to noise ratio (S/N) equals to 3-5, and the quantification limits (QLs) were at the concentration where S/N equals to 9-10. Based on calculation, the detection limits were 0.2 ppbv for benzene (0.64 µg/m^3 at 25 °C), ethylbenzene (0.87 µg/m^3 at 25 °C) and o-xylene (0.87 µg/m^3 at 25 °C), 0.1 ppbv for toluene (0.38 µg/m^3 at 25 °C) and m,p-xylene (0.43 µg/m^3 at 25 °C) when sampling time was 10 min. And the quantification limits were 0.4 ppbv for benzene, ethylbenzene and o-xylene, 0.2 ppbv for toluene and m,p-xylene with 10 minutes' sampling.

LINEARITY

Linearities were calculated using different sample concentrations with an identical sampling time as well as different sampling times with an identical sample.

Figure 3 shows the linearities using gas standards with concentrations of 2, 4, 8, 40, 200, 1000 and 2000 ppbv. The sampling rate was 10 mL/min and the sampling time was 2 min. Figure 4 shows the linearities using 20 ppbv BTEX standard with different sampling times, including 0.5, 1, 2, 5 and 10 min. Excellent system linearities were obtained for both sets of experiments, with R^2 values larger than 0.99. This indicates that the device can be calibrated with single gas standard with different sampling times, which provides customers more convenience and flexibility.

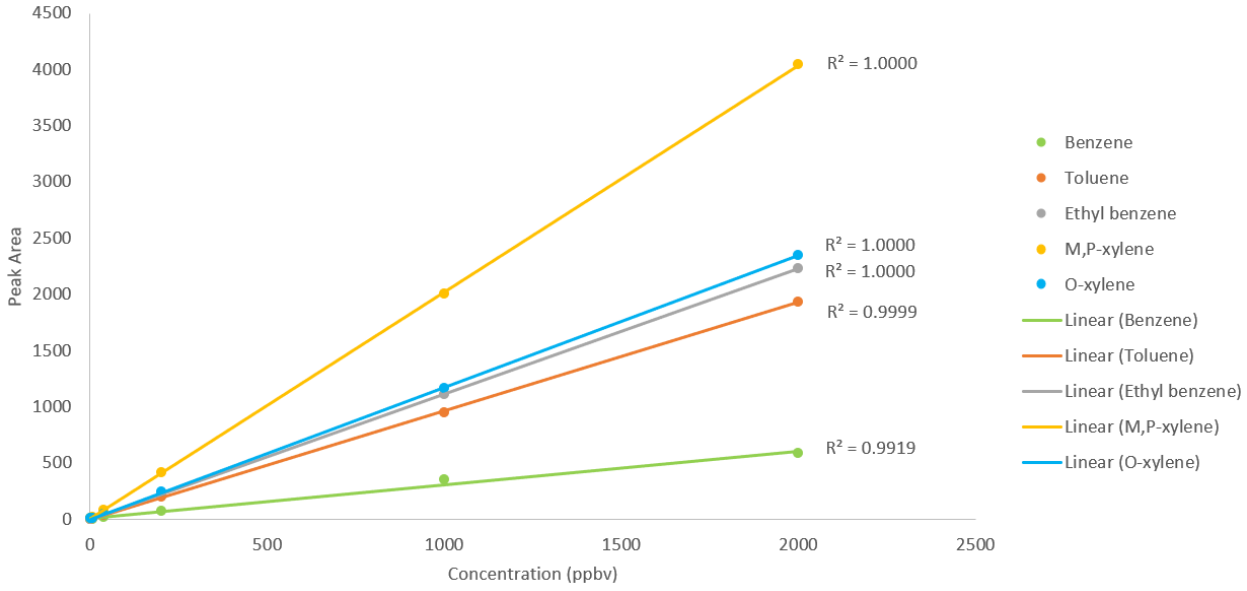

Figure 3. Linearities of BTEX with different standard gas concentrations at sampling time of 2 min using NovaTest P100 compact GC.

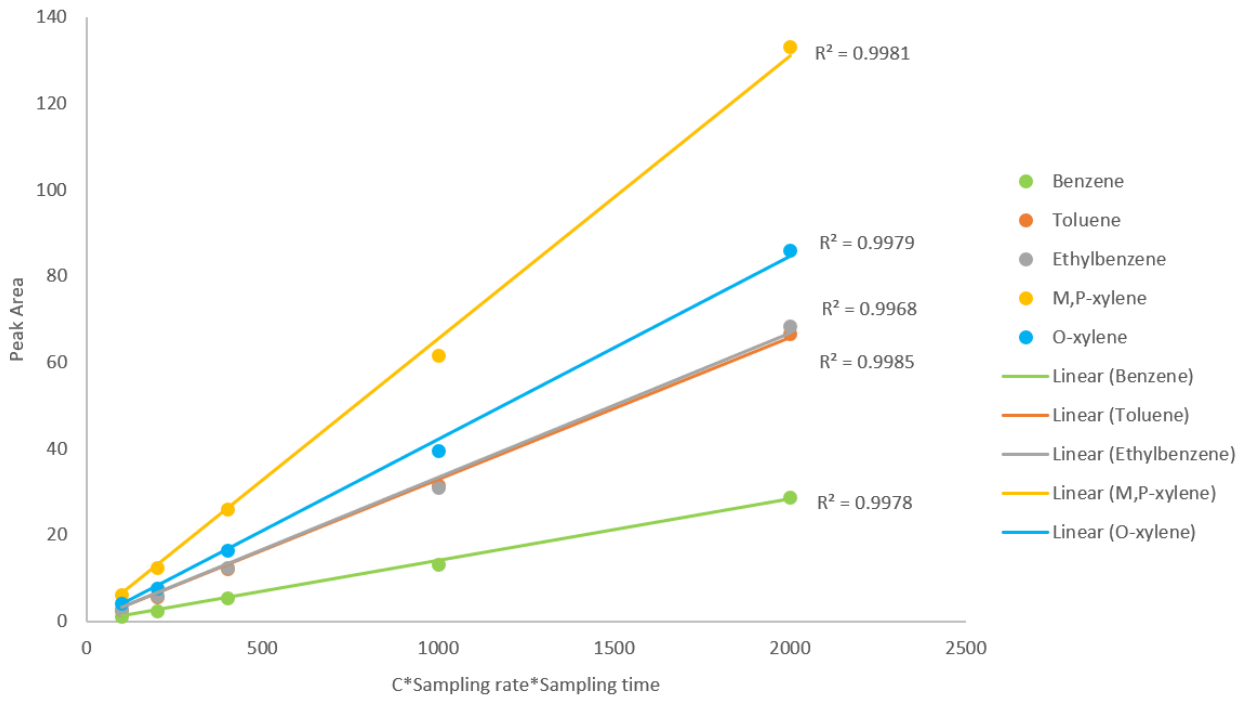

Figure 4. Linearities of BTEX with single gas standard (20 ppbv) at different sampling times using NovaTest P100 compact GC

REAL AIR SAMPLE

To demonstrate the performance of NovaTest P100 compact GC, real air samples in the office area were tested. The sampling time was set at 5 and 10 min considering the low concentration of the compounds expected in the office. Duplicates were tested for each sampling time.

With NovaSoft, detailed information, such as compound name, RT, FWHM, peak height, area, and the automated calculated concentration, was shown in the generated report. A representative report for office air sample with the sampling time of 5 min is shown in **Table 4**.

Table 4. Reported BTEX result in the office area with the sampling time of 5 min.

Peak #	Compound	RT (s)	FWHM (s)	Height	Area	Concentration (ppbv)
1	Benzene	32.59	1.25	0.32	0.38	0.5
2	Toluene	58.22	1.54	2.06	3.47	2.0
3	Ethylbenzene	94.03	3.65	0.15	0.43	0.2
4	m,p-Xylene	98.93	3.07	0.51	1.74	0.5
5	o-Xylene	114.77	3.07	0.87	3.98	1.8

Table 5. Comparison of the retention times (RTs) for each BTEX compound when using NavoTest P100 compact GC with different sampling times when testing air sample in the office area

Compound	Sampling time = 5 min		Sampling time = 10 min		Avg. (s)	RSD%, n=4
	RT 1 (s)	RT 2 (s)	RT 1 (s)	RT 2 (s)		
Benzene	32.59	32.59	32.69	32.59	32.62	0.15
Toluene	58.22	58.13	58.03	57.94	58.08	0.21
Ethylbenzene	94.03	93.74	93.55	93.46	93.70	0.27
m,p-Xylene	98.93	98.74	98.54	98.54	98.69	0.19
o-Xylene	114.77	114.96	114.48	114.67	114.72	0.17

The RTs from the 4 runs with two sampling times were compared in **Table 5**. The results indicated that the RT of each target compound was consistent with RSD less than 0.3 %.

Table 6. Comparison of reported concentrations for each BTEX compound when using NavoTest P100 compact GC with different sampling times.

Compound	Sampling time = 5 min		Sampling time = 10 min		Avg. (ppbv)	RSD%, n=4
	Run 1 (ppbv)	Run 2 (ppbv)	Run 1 (ppbv)	Run 2 (ppbv)		
Benzene	0.5	0.7	0.6	0.6	0.6	13.61
Toluene	2.0	2.3	2.1	2.4	2.2	8.30
Ethylbenzene	0.2	0.2	0.2	0.3	0.2	22.22
m,p-Xylene	0.5	0.3	0.4	0.4	0.4	20.41
o-Xylene	1.8	1.9	1.5	1.9	1.8	10.66

The reported concentrations for office air with two sampling times were compared in **Table 6**. Based on the results, all five BTEX were detected. The average concentrations of Benzene, toluene, ethylbenzene, m,p-xylene and o-xylene were all below 3 ppbv with RSD less than 23% for four tests. This indicates the capability and applicability of NovaTest P100 compact GC to detect low concentration of VOCs in the environment.

CONCLUSION

The NovaTest P100 is fully automated with built-in methods, greatly minimizing manual operation and increasing testing efficiency without sacrificing comparable performance.

Excellent chromatographs, linearity and reproducibility were obtained for BTEX tests with NovaTest P100 compact GC. When sampling time was 10 min, the MDLs for BTEX were as low as 0.1 ppbv. The NovaTest P100 compact GC is demonstrated to be fast, robust and easy to operate.

For more information about the device, please visit us at
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