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Validation Analysis of Gas Chromatography Method for Determining 8 Benzene Derivatives in Fixed Pollution Source Exhaust Gas

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Abstract Benzene derivatives are volatile organic compounds commonly present in the atmospheric environment, which are toxic and complex in composition. They have become a key regulatory object in China's atmospheric environment management. In this paper, Shimadzu Nexis GC-2030 gas chromatography was used to simultaneously detect eight benzene derivatives. According to the *Environmental Monitoring—Technical Guideline on Drawing and Revising Analytical Method Standards* (HJ 168-2010), the monitoring methods for benzene, toluene, ethylbenzene, p-xylene, m-xylene, isopropylbenzene, o-xylene, and styrene in the *Stationary Source Emission—Determination of Benzene and Its Analogies—Bags Sampling/Direct Injection—Gas Chromatography* (HJ 1261-2022) are verified, and their linear relationships, detection limits, precision and accuracy are analyzed.

Key words Benzene derivatives; Fixed pollution source exhaust gas; Method validation

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Benzene derivatives are a class of organic compounds with benzene rings, including various organic compounds such as benzene, toluene, ethylbenzene, xylene, *etc.* Most benzene derivatives have high volatility[1]. Benzene derivatives in the atmospheric environment can enter the human body directly through the respiratory tract, causing harm to the human body. At present, benzene derivatives have become a key regulatory object in China's atmospheric environmental governance[2-3]. Monitoring personnel mostly use solid adsorption/thermal desorption methods[4-6] to determine the content of benzene derivatives in the air. However, the preparation of solid adsorption/thermal desorption methods is complicated in the early stage, and there is a risk of sample tube penetration when collecting samples with high concentration. Relatively speaking, the bags sampling/direct injection directly injects the sample into the analytical instrument, which is convenient and fast[7-10]. According to the *Stationary Source Emission—Determination of Benzene and Its Analogies—Bags Sampling/Direct Injection—Gas Chromatography* (HJ 1261-2022)[11], 8 types of benzene compounds in fixed pollution source exhaust gas were determined. In accordance with the *Environmental Monitoring—Technical Guideline on Drawing and Revising Analytical Method Standards* (HJ 168-2010)[12], detection experiments were conducted, in order to provide some reference for monitoring 8 types of benzene compounds in fixed pollution sources.

1 Instruments and materials

1.1 Test instruments Instruments: Shimadzu Nexis GC-2030 gas chromatography (equipped with a 1 mL of quantitative ring and hydrogen flame ionization detector), 8030A intelligent multi-channel gas distribution device.

1.2 Experimental materials The mixed standard gas of 8 benzene derivatives produced by Foshan Kedi Gas Chemical Co., Ltd. was used in this experiment, and the sampling gas bag used was a polytetrafluoroethylene gas bag. The gas chromatography column adopted SH-Wax capillary chromatography column.

1.3 Gas chromatography conditions The inlet temperature was 200 ℃, and the detector temperature was 280 ℃. High-purity nitrogen was used as the carrier gas, hydrogen was used as the combustion gas, and air was used as the auxiliary gas. The flow rates of the carrier gas, combustion gas, and auxiliary gas were set to 30, 32, and 200 ml/min, respectively. After maintaining the instrument at 40 ℃ for 3 min, it was heated up to 50 ℃ at a rate of 5 ℃/min. After maintaining it for 2 min, it was heated up to 60 ℃ at a rate of 35 ℃/min. Finally, it was heated up to 100 ℃ at a rate of 5 ℃/min for 5 min. The diversion ratio was 5∶1, and the injection volume was 1.0 ml.

2 Test results

2.1 Standard curve 0.25, 5, and 12.5 μmol/mol of standard gases were used to configure the standard gases with different concentration gradients by an automatic gas distribution device. The concentrations of 8 benzene series standard samples were shown in Table 1. It was measured in order of concentration from low to high. Taking benzene series concentration as the horizontal axis and peak area as the vertical axis, a standard curve was drawn. The standard curve results were shown in Table 2.

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Table 1 Concentrations of 8 benzene series standard solutions prepared μmol/mol

Target object	Standard sample 1	Standard sample 2	Standard sample 3	Standard sample 4	Standard sample 5	Standard sample 6
Benzene	0.280	0.560	2.610	5.220	6.650	13.300
Toluene	0.280	0.560	2.595	5.190	6.450	12.900
Ethylbenzene	0.300	0.600	2.755	5.510	6.250	12.500
p-xylene	0.300	0.600	2.815	5.630	6.700	13.400
m-xylene	0.290	0.580	2.785	5.570	6.200	12.400
Isopropylbenzene	0.290	0.580	2.720	5.440	6.100	12.200
o-xylene	0.280	0.560	2.685	5.370	6.000	12.000
Styrene	0.260	0.520	2.685	5.370	6.500	13.000

Table 2 Regression equations and correlation coefficients of working curves for 8 benzene compounds

No.	Target object	Linear regression equation	Correlation coefficient
1	Benzene	$y = 10\,727.1x$	0.999 1
2	Toluene	$y = 12\,430.8x$	0.999 7
3	Ethylbenzene	$y = 14\,323.8x$	0.999 5
4	p-xylene	$y = 13\,748.3x$	0.999 8
5	m-xylene	$y = 14\,900.5x$	0.999 0
6	Isopropylbenzene	$y = 17\,603.8x$	0.999 8
7	o-xylene	$y = 15\,753.0x$	0.999 3
8	Styrene	$y = 13\,013.5x$	0.999 6

From Table 2, it can be seen that within the set concentration range, benzene, toluene, ethylbenzene, p-xylene, m-xylene, isopropylbenzene, o-xylene, and styrene all had good linear relationships, with correlation coefficients R of 0.999 1, 0.999 7, 0.999 5, 0.999 8, 0.999 0, 0.999 8, 0.999 3, and 0.999 6, respectively, meeting the requirements of the standard (HJ 1261 – 2022). The working curve can be used for subsequent analysis.

2.2 Detection limit According to the standard (HJ 168 – 2010), the standard gas with a measured concentration of 0.125 μmol/mol was selected to calculate the detection limit of the method. According to the analysis method, 0.125 μmol/mol of parallel standard samples were measured for 7 times, and the standard deviation was calculated. Then, the formula (1) was used to calculate the method detection limit (MDL)[8].

$$MDL = t_{(n-1,0.99)} \times S \tag{1}$$

According to the table, when $n = 7$, $t_{(n-1,0.99)} = 3.143$. The measurement and calculation results were shown in Table 3.

Table 3 Determination and calculation results of method detection limits μmol/mol

Target object		Benzene	Toluene	Ethylbenzene	p-xylene	m-xylene	Isopropylbenzene	o-xylene	Styrene
Parallel sample	1	0.140	0.143	0.139	0.143	0.137	0.149	0.134	0.104
	2	0.139	0.150	0.154	0.155	0.143	0.156	0.149	0.087
	3	0.146	0.147	0.159	0.178	0.171	0.164	0.158	0.110
	4	0.148	0.141	0.144	0.155	0.156	0.154	0.156	0.104
	5	0.143	0.139	0.147	0.151	0.146	0.156	0.140	0.099
	6	0.144	0.140	0.151	0.165	0.158	0.161	0.153	0.130
	7	0.139	0.140	0.162	0.172	0.155	0.144	0.134	0.110
Mean		0.143	0.143	0.151	0.160	0.152	0.155	0.146	0.106
Standard deviation		0.004	0.004	0.008	0.012	0.011	0.007	0.010	0.013
MDL		0.011	0.013	0.026	0.039	0.035	0.021	0.032	0.041
Lower limit of determination		0.045	0.052	0.103	0.155	0.142	0.085	0.128	0.164

The results showed that the average concentrations of benzene, toluene, ethylbenzene, p-xylene, m-xylene, isopropylbenzene, o-xylene, and styrene were 0.143, 0.143, 0.151, 0.160, 0.152, 0.155, 0.146, and 0.106 μmol/mol, respectively. Based on this result, the standard deviations of the 8 benzene derivatives mentioned above were calculated, between 0.004 and 0.013 μmol/mol, and lower determination limits were 0.045, 0.052, 0.103, 0.155, 0.142, 0.085 , 0.128, and 0.164 μmol/mol, respectively. The detection limits of the 8 benzene derivatives verified in this experiment all met the method requirements of the standard (HJ 1261 – 2022).

2.3 Precision and accuracy Low, medium, and high concentration points within the working curve range were selected for 6 parallel measurements, and the relative standard deviation and relative error were calculated. The experimental and calculated results were shown in Table 4 – 6.

Seen from Table 4 – 6, in the low concentration range, the relative standard deviations of 8 benzene derivatives were between 0.530% and 5.324%, and the relative errors were between –3.833% and 6.611%. In the medium concentration range, the standard deviation of 8 benzene derivatives ranged from 0.606% to 3.441%, and the relative error ranged from –5.975% to –2.842%. In the high concentration range, the standard deviation of 8 benzene derivatives ranged from 1.116% to 3.654%, and the relative error ranged from 1.243% to 6.707%. Under the detection conditions set in this experiment, the results of measuring the standard gases of 8 benzene derivatives were relatively stable and accurate.

Table 4 Determination data of precision and accuracy for low concentration standard gases μmol/mol

Target object		Benzene	Toluene	Ethylbenzene	p-xylene	m-xylene	Isopropylbenzene	o-xylene	Styrene
Measurement results	1	0.275	0.283	0.285	0.320	0.291	0.270	0.269	0.263
	2	0.273	0.286	0.295	0.319	0.291	0.281	0.272	0.253
	3	0.278	0.285	0.303	0.334	0.304	0.295	0.283	0.261
	4	0.274	0.285	0.279	0.315	0.290	0.286	0.271	0.238
	5	0.284	0.282	0.287	0.310	0.284	0.288	0.273	0.242
	6	0.267	0.285	0.282	0.321	0.288	0.282	0.259	0.274
Mean		0.275	0.284	0.289	0.320	0.291	0.284	0.271	0.255
Standard gas concentration		0.280	0.280	0.300	0.300	0.290	0.290	0.280	0.260
Standard deviation		0.006	0.002	0.009	0.008	0.007	0.008	0.008	0.014
Relative standard deviation//%		2.048	0.530	3.098	2.512	2.314	2.947	2.841	5.324
Relative error//%		−1.726	1.548	−3.833	6.611	0.460	−2.184	−3.155	−1.859

Table 5 Measurement data of precision and accuracy for medium concentration standard gases μmol/mol

Target object		Benzene	Toluene	Ethylbenzene	p-xylene	m-xylene	Isopropylbenzene	o-xylene	Styrene
Measurement results	1	5.052	5.064	5.516	5.779	5.656	5.269	5.158	5.162
	2	5.077	5.038	5.216	5.321	5.188	5.143	5.050	5.153
	3	5.062	5.098	5.184	5.364	5.226	5.096	5.041	5.018
	4	5.130	5.145	5.390	5.614	5.312	5.230	5.018	5.183
	5	5.044	5.064	5.259	5.486	5.196	5.169	5.001	5.011
	6	5.065	5.020	5.172	5.361	5.198	5.177	5.027	5.132
Mean		5.072	5.072	5.290	5.488	5.296	5.181	5.049	5.110
Standard gas concentration		5.220	5.190	5.510	5.630	5.570	5.440	5.370	5.370
Standard deviation		0.031	0.045	0.136	0.179	0.182	0.062	0.056	0.076
Relative standard deviation//%		0.606	0.881	2.574	3.259	3.441	1.189	1.110	1.481
Relative error//%		−2.842	−2.283	−4.002	−2.531	−4.919	−4.767	−5.975	−4.845

Table 6 Measurement data of precision and accuracy for high concentration standard gases μmol/mol

Target object		Benzene	Toluene	Ethylbenzene	p-xylene	m-xylene	Isopropylbenzene	o-xylene	Styrene
Measurement results	1	13.334	13.027	13.253	14.178	12.802	12.478	12.039	13.879
	2	13.558	13.103	13.421	14.470	13.124	12.995	12.483	14.223
	3	13.629	13.374	13.696	14.720	13.294	13.263	12.664	14.238
	4	13.579	13.376	13.655	14.674	13.250	13.356	12.747	14.238
	5	13.443	12.901	12.934	13.780	12.480	12.581	11.850	13.318
	6	13.249	13.087	13.071	13.655	12.875	12.326	11.956	13.105
Mean		13.465	13.145	13.338	14.246	12.971	12.833	12.290	13.834
Standard gas concentration		13.300	12.900	12.500	13.400	12.400	12.200	12.000	13.000
Standard deviation		0.150	0.192	0.309	0.454	0.311	0.432	0.388	0.505
Relative standard deviation//%		1.116	1.461	2.316	3.185	2.400	3.363	3.160	3.654
Relative error//%		1.243	1.897	6.707	6.315	4.603	5.190	2.415	6.412

3 Conclusions

The experimental results showed that under the conditions set in this experiment, the working curve of determining 8 benzene derivatives by Shimadzu Nexis GC—2030 gas chromatography had a good linear relationship, and the detection limit, precision, and accuracy can meet the requirements of the standard (HJ 1261 − 2022). Therefore, the laboratory has the ability to monitor 8 benzene derivatives in fixed pollution exhaust gas.

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