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Benzene, Toluene, Xylene and Ethylbenzene in the Air of Workplace: Validation of Solvent Desorption Gas Chromatography

Pan Yu*
Nanjing Qixia District Center for Disease Control and Prevention, Nanjing 210000, Jiangsu, China

**Author to whom correspondence should be addressed.*

Abstract: To ensure the applicability and reliability of the detection methods for benzene, toluene, xylene and ethylbenzene in the air of workplace under the conditions of our laboratory, the linear range, detection limit, quantification limit, desorption efficiency, precision and accuracy of the method were systematically verified according to the solvent desorption gas chromatography method in GBZ/T 300.66-2017 (5) determination of toxic substances in the air of Workplace Part 66: benzene, toluene, xylene and ethylbenzene. The results showed that the linear range of the benzene series was 0–25 μg/mL, and the correlation coefficients were ≥ 0.9993. The detection limits were 0.05–0.08 μg/mL, and the limits of quantification were 0.16–0.25 μg/mL. The average desorption efficiency of the activated carbon tube for the target compound is between 96.1% and 98.8%. The relative standard deviations (RSDs) within and between batches were less than 7.00% and 7.80%, respectively. The recovery was 96.7%–106.3%. The validation results showed that the performance indicators of the method met the technical requirements of GBZ/T 210.4-2008 guidelines for the formulation of Occupational Health Standards Part 4: methods for the determination of chemical substances in the air of workplaces, and could meet the actual needs of the laboratory for batch and efficient daily monitoring of benzene, toluene, xylene and ethylbenzene in the air of workplaces.
Keywords: Benzene series; Workplace; Gas chromatography; Method validation

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1. Introduction

Benzene series, such as benzene, toluene, xylene, and ethylbenzene, are widely used as typical volatile organic compounds (VOCs) in the chemical industry, painting, printing, furniture manufacturing and many other industries [1,2]. Long term or high-concentration exposure to BTEX will cause clear damage to human health. Among them, benzene has been listed as a class I human carcinogen by the international agency for research on cancer (IARC), which mainly damages the hematopoietic system and can lead to leukemia; Toluene, xylene and ethylbenzene have significant anesthetic and stimulating effects on the central nervous system, and may cause functional damage to the liver and kidney [3–6]. In order to strengthen occupational health protection, China revised and implemented GBZ 2.1-2019 occupational exposure limits

for hazardous factors in workplace air in 2022 [7], and put forward stricter requirements for the exposure limits of some BTEX, which reflects the continuous tightening of national control over occupational hazard factors. In recent years, workplace air monitoring results carried out in many regions show that benzene, toluene, xylene, ethylbenzene and other exposure concentrations have large occupational health risks [8–11], and its potential threat to the health of operators cannot be ignored. Therefore, systematic and accurate monitoring is the premise of effective risk intervention.

There are more than 2000 occupational health monitoring target enterprises involving leather, furniture, chemical and other industries in the jurisdiction, with a large number of employees, and the potential benzene series exposure risk points are widely distributed. In the face of such a huge monitoring demand and increasingly stringent standard requirements, the establishment and possession of a set of reliable, accurate and efficient benzene series determination methods that are convenient for the daily detection work can provide solid and efficient technical support for the large-scale and normalized occupational health benzene series monitoring work in this area, which is of urgent practical significance for performing the technical support responsibility of occupational health and protecting the health rights and interests of workers.

2. Materials and methods

2.1. Instruments and reagents

Agilent 8890 gas chromatograph (equipped with FID detector) on db-ffap column (30 m × 0.32 mm × 0.25 μm). Reagent: Standard Solution of 7 benzene series in carbon disulfide (a240500611000 mg/l, quality inspection by Tanmo); Carbon disulfide (benzene free, chromatographically pure); Activated carbon tube (solvent desorption type, 100 mg/50 mg); High purity nitrogen (purity ≥ 99.999%), hydrogen and air.

2.2. Instrument working conditions

The column temperature was 40 ℃, the injection port temperature was 150 ℃, and the detector temperature was 200 ℃. The column flow rate was 2 μL/min, and the split ratio was 5:1; Hydrogen flow: 40 mL/min, air flow: 400 ml/min; The injection volume was 2.0 μL.

2.3. Sample handling

Transfer the front and rear sections of the activated carbon tube to the solvent desorption bottle, respectively, add 1.0 mL of carbon disulfide, seal, and desorb for 30 min (shake from time to time), and the desorption liquid is analyzed by gas chromatography.

2.4. Standard curve drawing

Dilute the standard solution step by step with carbon disulfide to prepare a standard series with concentrations of 1.0, 2.5, 5.0, 10.0, 15.0, 25.0 μg/mL, and draw the standard curve according to the above instrument conditions (**Table 1**).

Table 1. Standard series of benzene, toluene, xylene and ethylbenzene

Serial number	Std_1	Std_2	Std_3	Std_4	Std_5	Std_6
Volume of standard solution added (μL)	10	25	50	100	150	250
Volume of carbon disulfide added (μL)	990	970	950	900	850	750
Concentration of 7 benzene series (μg/mL)	1.0	2.5	5.0	10.0	15.0	25.0

2.5. Validation indicators

The linear range, detection limit (LOD), limit of quantification (LOQ), desorption efficiency, intra batch and inter batch

precision, and spiked recovery of the method were evaluated with reference to gbz/t 210.4-2008 guidelines for the formulation of Occupational Health Standards Part 4: Determination of chemical substances in the air of workplaces [12] and gbz/t 300.66-2017 determination of toxic substances in the air of workplaces part 66: benzene, toluene, xylene, and ethylbenzene solvent analysis gas chromatography [13].

3. Results and discussion

3.1. Detection limit and quantification limit

According to the chromatographic conditions, the standard series was injected three times at each point, and the standard curve was drawn with the peak area of benzene, toluene, xylene and ethylbenzene. The detection limit (μg/mL) is calculated by 3 times the baseline noise, and the quantification limit (μg/mL) is calculated by 10 times the baseline noise. The minimum detection concentration (mg/m^3) and the minimum quantitative concentration (mg/m^3) of this method are calculated by collecting 1.5 l of air. The results are shown in **Table 2**. The linearity of the benzene series was good in the range of 0–25 μg/mL, and the correlation coefficients were greater than 0.999. The minimum detectable concentration of each component is 0.03–0.05 mg/m³, which meets the requirements of occupational health monitoring.

Table 2. Linear equation, correlation coefficient and detection limit

Component	Linear equation	r	LOD (μg/mL)	LOQ (μg/mL)	Minimum detectable concentration (mg/m^3)
Benzene	y = 4.3704x + 0.3693	0.9998	0.05	0.16	0.03
Toluene	y = 5.4225x-0.3098	0.9998	0.05	0.18	0.03
Ethylbenzene	y = 5.2385x-0.6398	0.9996	0.07	0.22	0.05
Paraxylene	y = 4.9544x-0.4587	0.9996	0.07	0.23	0.05
M-xylene	y = 5.7052x-0.8899	0.9993	0.07	0.24	0.05
O-xylene	y = 5.6364x-1.2948	0.9997	0.08	0.25	0.05

3.2. Desorption efficiency

Three gradient spiked concentrations (4.5 μg, 9.0 μg and 18.0 μg) were designed, and 6 samples were determined in parallel at each concentration level for a total of 18 times. It can be seen from **Table 3** that the average desorption efficiency of all BTEX is between 96.1% and 98.8%. The results showed that the target compounds adsorbed on the activated carbon could be eluted efficiently and stably under the pretreatment conditions of 1.0 mL carbon disulfide and ultrasonic desorption for 30 min, with minimal system loss.

Table 3. Average resolution of benzene, toluene, xylene and ethylbenzene

Component	Average desorption efficiency (%)	Range (%)	RSD (%)
Benzene	98.0	90.7–102.9	3.8–6.1
Toluene	98.8	94.6–102.8	3.8–5.6
Ethylbenzene	98.2	94.6–101.8	4.0–6.0
Paraxylene	97.3	93.4–101.1	4.1–5.9
M-xylene	96.1	92.6–97.8	3.3–5.3
O-xylene	97.1	92.5–100.8	3.4–5.9

3.3. Precision and accuracy

The precision and accuracy of the method are the core indicators to evaluate its stability, reproducibility and reliability, which directly determine whether the detection results can truly reflect the content level of the target in the sample.

3.3.1. Intra-batch precision

In the intra-batch precision experiment, six parallel determinations were performed on the simulated samples with three gradient spiked concentrations (4.5 μg, 9.0 μg and 18.0 μg) on the same day. As shown in **Table 4**, the within-batch relative standard deviation (RSD) of all components ranges from 4.16% to 7.00%, indicating that the method has excellent repeatability in short-term operation.

Table 4. Intra-batch precision and accuracy of benzene, toluene, xylene and ethylbenzene

Component	Scalar addition (μg)	Blank (μg)	Measured value (μg)						Mean (μg)	RSD (%)	Recovery rate (%)
Benzene	4.5	0.0	4.3	4.9	4.7	4.8	4.6	4.8	4.6	4.56	102.2
	9.0	0.0	9.6	9.2	9.7	9.5	9.9	9.8	9.4	4.16	105.0
	18.0	0.0	20.2	18.2	17.2	18.6	18.8	19.9	18.8	5.08	104.4
Toluene	4.5	0.0	4.1	4.8	4.5	4.6	4.6	4.8	4.5	5.06	101.3
	9.0	0.0	9.9	8.9	9.5	9.4	9.6	8.4	9.3	5.63	103.1
	18.0	0.0	19.5	17.6	16.4	18.4	18.1	19.6	18.3	5.60	101.4
Ethylbenzene	4.5	0.0	4.2	4.8	4.3	4.5	4.8	4.9	4.6	6.38	102.2
	9.0	0.0	10	8.8	9.5	9.2	9.7	9.4	9.4	4.37	104.7
	18.0	0.0	19.5	17.8	17.0	19.1	18.9	20.5	18.8	6.60	104.4
Paraxylene	4.5	0.0	4.2	4.8	4.2	4.4	4.8	4.9	4.5	7.00	101.1
	9.0	0.0	10.1	8.8	9.5	9.0	9.8	8.4	9.2	6.94	102.8
	18.0	0.0	19.7	17.9	17.5	19.3	19.5	21.0	19.15	6.66	106.3
M-xylene	4.5	0.0	4.2	4.7	4.3	4.5	4.7	5.0	4.5	6.44	101.3
	9.0	0.0	9.8	8.6	9.5	9.4	9.4	8.5	9.2	5.71	102.2
	18.0	0.0	19.3	17.7	18.3	18.8	19.4	19.5	18.8	3.79	104.6
O-xylene	4.5	0.0	4.1	4.9	4.5	4.5	4.7	4.8	4.6	6.23	101.7
	9.0	0.0	9.9	8.8	9.4	9.5	9.5	8.5	9.3	5.57	102.8
	18.0	0.0	19.1	17.9	16.9	19.1	18.0	19.5	18.4	5.34	102.2

3.3.2. Precision between batches

In the inter-batch precision experiment, the simulated samples with three gradient spiked concentrations (4.5 μg, 9.0 μg, and 18.0 μg) were determined for six times in parallel for three consecutive days, and the samples with the same spiked concentration were determined using the newly prepared standard curve every day. As shown in **Table 5**, the relative standard deviation (RSD) between batches of all components is between 2.18% and 7.80%, indicating that under the influence of variable factors such as different time and different reagent batches, this method can still maintain a high degree of stability and consistency, and fully meet the reproducibility requirements of daily batch testing in the laboratory.

Table 5. Inter batch precision and accuracy of benzene, toluene, xylene and ethylbenzene

Component	Scalar addition (μg)	Blank (μg)	Measured value (μg)						Mean (μg)	RSD (%)	Recovery rate (%)
			First day		The next day		Day 3				
Benzene	4.5	0.0	4.3	4.6	4.1	4.3	4.8	4.1	4.4	6.42	96.9
	9.0	0.0	9.2	8.8	8.0	8.6	9.9	9.7	9.03	7.80	100.4
	18.0	0.0	18.6	18.8	19.6	19.0	19.2	18.4	18.9	2.28	105.0
Toluene	4.5	0.0	4.5	4.6	4.2	4.5	4.8	4.2	4.5	5.23	99.3
	9.0	0.0	8.9	9.4	8.3	8.6	9.4	9.8	9.1	6.20	100.7
	18.0	0.0	19.5	18.4	19.6	18.7	19.0	18.5	18.9	2.69	105.3
Ethylbenzene	4.5	0.0	4.3	4.5	4.3	4.5	4.6	4.1	4.38	4.19	97.3
	9.0	0.0	8.8	9.2	8.5	8.5	8.8	9.7	8.9	5.18	99.1
	18.0	0.0	19.1	18.9	19.3	18.4	18.5	18.3	18.8	2.18	104.2
Paraxylene	4.5	0.0	4.2	4.4	4.3	4.5	4.6	4.1	4.4	4.30	96.7
	9.0	0.0	8.8	9.0	8.5	8.5	8.7	9.7	8.9	5.07	98.5
	18.0	0.0	19.3	19.5	19.2	18.3	18.5	18.2	18.8	3.00	104.4
M-xylene	4.5	0.0	4.3	4.5	4.2	4.4	4.6	4.1	4.4	4.30	96.7
	9.0	0.0	8.6	9.4	8.2	8.3	8.8	9.6	8.8	6.50	98.0
	18.0	0.0	19.3	18.8	18.5	17.7	18.4	18.2	18.5	2.92	102.7
O-xylene	4.5	0.0	4.5	4.5	4.2	4.4	4.6	4.2	4.4	3.80	97.8
	9.0	0.0	8.8	9.4	8.5	8.6	8.8	9.6	8.9	4.98	99.4
	18.0	0.0	19.1	19.1	19.1	18.3	18.5	18.2	18.7	2.30	103.9

3.3.3. Accuracy

In the accuracy experiment, it is verified by the standard addition recovery experiment. As shown in **Table 4**. The recoveries of all BTEX spiked at three concentration levels in the intra-batch and inter-batch experiments were 101.3%–105.0% and 96.7%–105.3%, respectively, which showed that the method had a small systematic error and ensured the reliability of the final detection results.

4. Conclusion

The validation data of this method showed that the linear relationship of benzene, toluene, xylene and ethylbenzene was good in the concentration range of 0–25 μg/mL, and the correlation coefficient $R \geq 0.9993$; The sensitivity of the method is practical Detection requirements, the detection limit is 0.05-0.08 μg/mL; The desorption efficiency of activated carbon tube is 96.1%–98.8%,; The intra assay RSD and inter assay RSD of the method precision were less than 7.0% and 7.8%, respectively; The recovery was 96.7%–106.3%. All technical indicators meet the evaluation requirements.

In conclusion, the laboratory has the technical ability to carry out the detection of the project, and the relevant instruments and equipment, personnel qualifications and quality control measures have been in place. It can provide standardized and accurate detection services of Benzene Series in the air of the workplace for enterprises within its jurisdiction according to the verified methods and procedures, and provide strong technical support for the monitoring and risk assessment of occupational hazards.

Disclosure statement

The author declares no conflict of interest.

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