Determination of Benzene and its Derivatives in Water with the Agilent 8697 Headspace Sampler and 8890 GC

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Introduction

The analysis of benzene and its derivatives in environmental water samples usually proceeds by either headspace or purge and trap methods. This is typically accompanied by separation by gas chromatography (GC) and detection by a flame ionization detector (FID) or a mass spectrometric detector (MSD). HJ 1067-2019, issued by Chinese Ministry of Environmental Protection, is a method that describes the determination of benzene and its analogues in water by headspace GC with FID. Sample extraction, analysis, identification, and quantitation are detailed in this method.



Response (pA)

Results and discussion

1. Benzene 2. Toluene 3. Ethylbenzene 4. *p*-Xylene 5. *m*-Xylene 6. Cumene 7. o-Xylene 8. Styrene

This poster demonstrates that the Agilent 8697 headspace sampler, coupled with the Agilent 8890 GC, delivers accurate and reliable analysis of benzene and certain derivatives in water. This system can easily achieve the performance specification for the compounds detailed in method HJ 1067-2019. The calibration curves determined for those target compounds were found to be within method requirements and the correlation coefficients were well above 0.999. The relative standard deviation (RSD) was determined for each compound. The area %RSD was 1.3% – 2.4% and the retention time %RSD was less than 0.045%. For all the compounds, the MDLs were $\leq 0.2 \ \mu g/L$. Satisfactory recoveries were achieved at around 99.1% to 101.7%.



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Retention time (min)

8

Chromatogram of the eight target compounds at a concentration of 200 μ g/L.

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Agilient 8697 Headspace Sampler		

No.	Name	RT	Formula	R ²
1	Benzene	4.839	y = 0.7283x - 7.2099	0.9998
2	Toluene	7.807	y = 0.7677x - 8.5950	0.9998
3	Ethylbenzene	10.519	y = 0.7666x - 7.9522	0.9999
4	<i>p</i> -Xylene	10.771	y = 0.7541x - 7.7485	0.9999
5	<i>m</i> -Xylene	10.994	y = 0.7561x - 7.7873	0.9999
6	Cumene	12.068	y = 0.7571x - 5.4705	0.9999
7	o-Xylene	12.463	y = 0.7416x - 7.6819	0.9998
8	Styrene	15.173	y = 0.7033x - 7.0938	0.9998

R² values for benzene and its derivatives in the calibration standard over the 10 to 2,000 μ g/L range of this study.

Experimental

Separation was carried out using the Agilent 8697 headspace sampler coupled with an Agilent 8890 GC/FID. Agilent OpenLab CDS 2.5 software was used for data acquisition and analysis. The instrument conditions are shown below.

Parameter	Value			
Agilent 8697 Headspace Sampler				
Loop Size	1 mL			
Pressurization Gas	Nitrogen			
Oven Temperature	80 °C			
Loop Temperature	80 °C			
Transfer Line Temperature	100 °C			
Vial Equilibration Time	40 min			
Injection Duration	0.5 min			
Vial Size	20 mL			
Fill Pressure	15 psi			
Loop Fill Mode	Default			
Vial Shaking	Level 8			
Agilent 8890 GC				
Inlet	Split/splitless 200 °C, split ratio 10:1 Liner: Straight, deactivated, 2 mm id (p/n 5181-8818)			





(A) Calibration of benzene from 10 to 2,000 μ g/L. (B) Calibration of ethylbenzene from 10 to 2,000 μ g/L.

		RT %RSD	Area %RSD (n = 8)		MDL	Average % Recovery (n = 6	
No.	Name	(n = 8)	20 µg/L	200 µg/L	(µg/L)	200 µg/L	
1	Benzene	0.045	1.77	1.74	0.16	101.7	
2	Toluene	0.034	1.66	1.71	0.14	100.5	
3	Ethylbenzene	0.022	1.69	1.62	0.16	99.9	ব
4	<i>p</i> -Xylene	0.030	2.13	1.92	0.16	99.1	onse[p,
5	<i>m</i> -Xylene	0.026	1.82	1.73	0.17	99.7	Respi
6	Cumene	0.025	1.30	1.51	0.16	100.2	
7	o-Xylene	0.023	1.80	1.74	0.16	100.7	
8	Styrene	0.021	2.32	2.40	0.20	100.3	

RSD, MDL, and recovery percentages for benzene and its derivatives.



Overlaid GC/FID chromatograms of eight repeat

Oven	40 °C (5 min), then 5 °C/min to 80 °C (5 min), then 30 °C/min to 200 °C (5 min)
FID	250 °C, hydrogen: 30 mL/min, air: 300 mL/min





Reference

1. HJ 1067-2019. Water Quality— **Determination of Benzene and its** Analogies—Headspace/Gas Chromatography. China National **Environmental Monitoring Station**, **Chinese Ministry of Ecology and Environment** (date of issue: 24 **December 2019**).

Conclusions

This poster demonstrates that the 8697 headspace sampler configured with an 8890 GC and an FID can provide a reliable and economical solution for the analysis of benzene and its analogues in water. The inert flow path from headspace to detector results in a reliable inertness level that provides excellent peak shape, resolution, and great repeatability.