

Analysis of Drinking Water with the Agilent 8860 GC and 7697A Headspace Sampler

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Abstract

The flexibility of the Agilent 8860 GC is demonstrated by an analysis requiring multiple types of detection and sample introduction. Volatile halogenated and aromatic hydrocarbons in water are sampled by headspace for detection by FID and ECD, and organophosphorus pesticides are detected by FPD. A single 8860 GC can be configured to run both analyses.

Introduction

Volatile halogenated and aromatic hydrocarbons can enter the water table through various sources. Benzene, for example, can leach into groundwater from petroleum production and storage sites. Headspace sampling is well suited for the detection of these pollutants in water as it requires minimal sample preparation.

Organophosphorus pesticides are the most widely used pesticides because of their high efficacy and short residual period. However, increasing attention has been paid to the pollution of organophosphorus pesticides in water. Organophosphorus pesticide detection is an important part of water quality detection. Because of the high efficiency, accuracy, and sensitivity of GC analysis, it is widely used in the analysis of organophosphorus pesticide residues.

Experimental

Instrumentation

The volatile halogenated and aromatic hydrocarbon analysis was performed using an Agilent 7697A headspace sampler, allowing the direct analysis of water samples (Figure 1). The headspace transfer line was installed into a split/splitless inlet on the Agilent 8860 GC. An unpurged capillary flow technology (CFT) splitter was used to split the sample between two columns. Column 1, an Agilent J&W DB-624 Ultra Inert, was connected to a flame ionization detector (FID) for the detection of aromatic hydrocarbons. Column 2, an Agilent HP-5ms Ultra Inert, was connected to an electron capture detector (ECD) for the detection of halogenated hydrocarbons. Table 1 shows the instrument conditions.

The organophosphorus pesticides analysis was performed using an 8860 GC equipped with an Agilent J&W DB-1701 column and flame photometric detector (FPD). Sample introduction was done using an Agilent 7650A automatic liquid sampler with a 5 μL syringe and a split/splitless injection port. Table 2 shows the instrument conditions.

Chemicals and standards

Standards for three halogenated and five aromatic hydrocarbons were made from aliquots of neat solvents (>99.99% purity, Sigma-Aldrich) in 99.9% methanol. Six calibration levels were made in headspace vials by adding stock standard to 5 mL water. Final concentrations were approximately 250, 1,000, 1,500, 2,000, 5,000, and 10,000 $\mu\text{g/L}$ for the aromatic hydrocarbons and 20, 50, 100, 200, 500, and 1,000 $\mu\text{g/L}$ for the halogenated hydrocarbons.

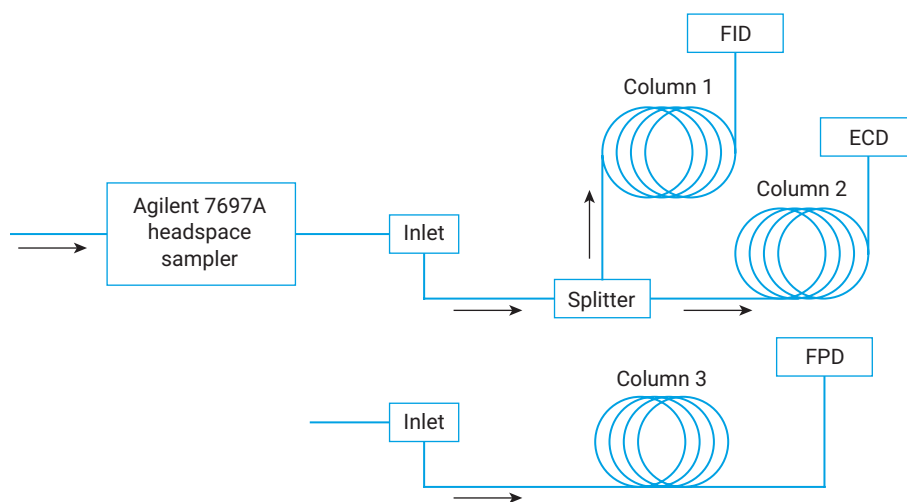


Figure 1. Configuration diagram for the Agilent 8860 GC with an Agilent 7697A headspace sampler.

Table 1. Volatile halogenated and aromatic hydrocarbon method conditions.

Headspace	
Oven	70 °C
Loop	70 °C
Transfer Line	100 °C
Vial Equilibration	40.00 minutes
Injection Duration	0.50 minutes
Vial Shaking	Level 1
Vial Fill Mode	Default
Vial Fill Pressure	15 psi
Loop Fill Mode	Default
Inlet (SSL)	
Mode	Split
Heater	On, 250 °C
Pressure	10.795 psi
Carrier	Nitrogen
Split Ratio	50:1
Split Flow	50 mL/min
Septum Purge	3 mL/min
Liner	Split, Straight, 2 mm id, Ultra Inert (p/n 5190-6168)
Columns	
Column 1	Agilent DB-624 UI (p/n 122-1334UI)
Dimensions	30 m × 250 µm, 1.40 µm
Column 2	Agilent HP-5ms UI (p/n 19091S-433UI)
Dimensions	30 m × 250 µm, 0.25 µm
Flow	1 mL/min, constant flow
Retention Gap	0.2 m × 250 µm
CFT Device	2-way Tee, p/n G3184-60065
Oven	
Initial	40 °C hold for 2 minutes
	Rate (°C/min) Value (°C) Hold time (min)
	6 120 4
Column 1 Detector (FID)	
Heater	250 °C
Air	400 mL/min
H ₂	30 mL/min
Makeup	N ₂ at 25 mL/min
Column 2 Detector (ECD)	
Heater	300 °C
Makeup	N ₂ at 60 mL/min

Table 2. Organophosphorus pesticides method conditions.

Inlet (SSL)			
Mode	Splitless		
Heater	On, 230 °C		
Pressure	13.5		
Carrier	Nitrogen		
Purge Time	0.75 minutes		
Purge Flow	60 mL/min		
Septum Purge	Off		
Liner	Splitless (p/n 5190-2293)		
Injection Volume	1 µL		
Column			
Column 3	Agilent DB-1701 (p/n 122-0732)		
Dimensions	30 m × 250 µm, 0.25 µm		
Flow	1 mL/min, constant flow		
Oven			
Initial	100 °C hold for 0 minutes		
	Rate (°C/min)	Value (°C)	Hold time (min)
	25	170	0
	15	210	1
	10	220	0
	15	240	5
Column 1 Detector (FPD+)			
Transferline	270 °C		
Emission Block	150 °C		
Air	60 mL/min		
H ₂	60 mL/min		
Makeup	N ₂ at 60 mL/min		

Standards for six organophosphorus pesticides were purchased from ANPEL Scientific Instrument Co. Ltd. (Shanghai, China). The concentration of each component was 100 µg/mL. A stock solution of the six compounds was prepared in methylene chloride for a final concentration of 1,000 µg/L. Six vials were made at each calibration level by spiking varying amounts of stock solution to achieve the required levels. The calibration standards were prepared at standard concentrations of 20, 50, 100, 200, 500, and 1,000 µg/L.

Results and discussion

Volatile halogenated and aromatic hydrocarbons

The volatile halogenated and aromatic hydrocarbons were well separated on their respective channels (Figure 2). Excellent area and retention time repeatability were achieved on this configuration. Area %RSD for all compounds were below 2.15% with retention time %RSD less than 0.017% for 10 spiked water samples (Table 3). Calibration coefficients (R^2) for each component were above 0.9994.

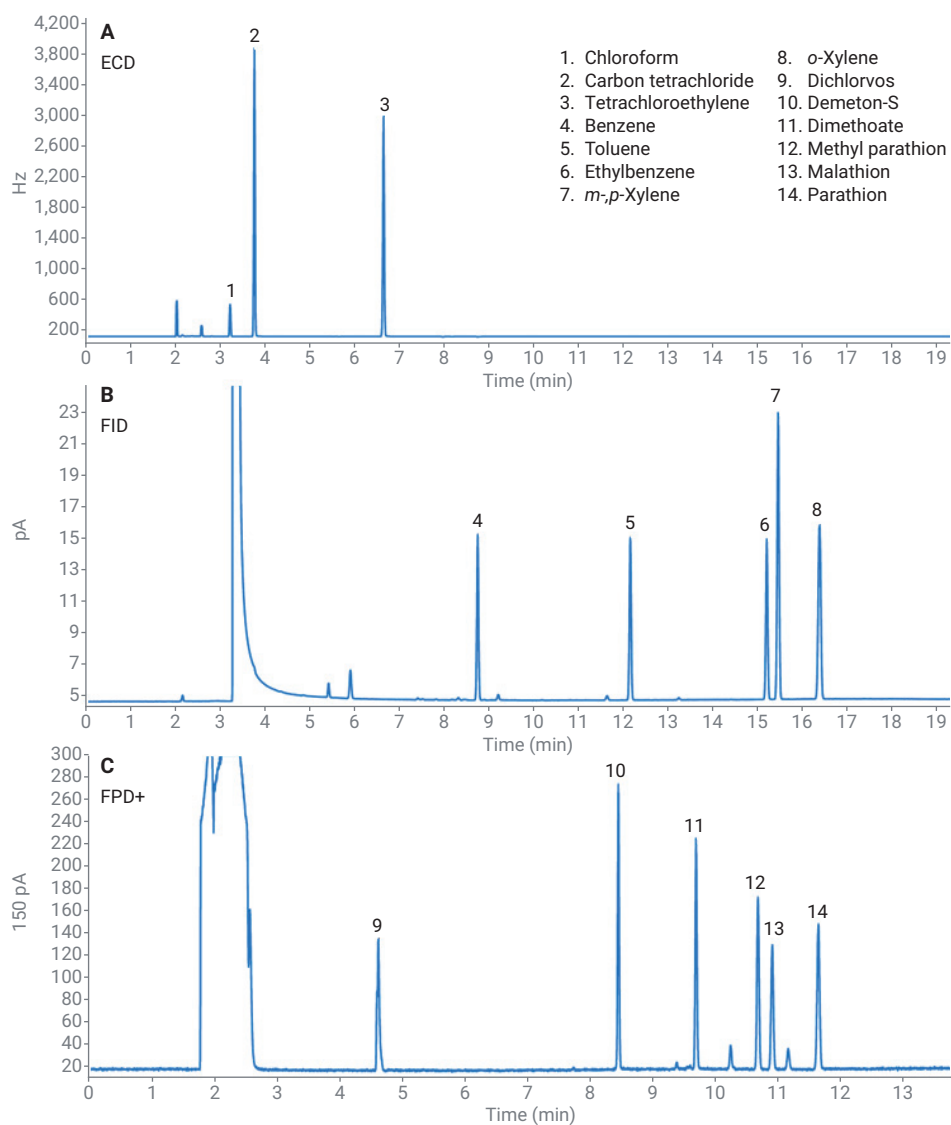


Figure 2. ECD (A) and FID (B) chromatograms of volatile halogenated and aromatic hydrocarbons spiked in water. (C) FPD+ chromatogram of organophosphorus pesticide standard.

Organophosphorus pesticides

Figure 2 shows an example chromatogram of the six organophosphorus pesticides at a concentration of 100 µg/L. The 8860 GC system demonstrates good peak shape and separation for these compounds, indicating a highly inert flowpath from inlet to detector. Table 4 shows that the correlation coefficients for all these compounds were better than 0.9995. Repeatability was tested using six injections of the standard mixture at 100 µg/L. Table 4 illustrates that the retention time and area repeatability are quite good. For all compounds, the retention time %RSD is below 0.019% and the area %RSD is well below 1.83%.

Conclusion

The flexibility of the Agilent 8860 GC allows multiple sampling techniques and three different detectors to be configured on a single system. The system delivered excellent results across various analyte classes. The inclusion of an Agilent 7697A headspace sampler makes this system a versatile tool for the analysis of drinking water.

Table 3. Volatile halogenated and aromatic hydrocarbon repeatability and calibration performance.

Compound	Area %RSD (n = 10)	Retention Time %RSD (n = 10)	Calibration R ²
Chloroform	1.54	0.017	0.9999
Carbon Tetrachloride	2.46	0.014	0.9995
Tetrachloroethylene	2.15	0.010	0.9998
Benzene	1.85	0.008	0.9997
Toluene	1.88	0.006	0.9996
Ethylbenzene	1.79	0.008	0.9995
<i>m,p</i> -Xylene	1.75	0.007	0.9995
<i>o</i> -Xylene	1.46	0.009	0.9994

Table 4. Organophosphorus pesticide repeatability and calibration performance.

Compound	Area %RSD (n = 6)	Retention Time %RSD (n = 6)	Calibration R ²
Dichlorvos	1.36	0.017	0.9999
Demeton-S	1.44	0.004	0.9998
Dimethoate	1.56	0.009	0.9997
Methyl Parathion	1.48	0.015	0.9996
Malathion	1.34	0.013	0.9999
Parathion	1.83	0.019	0.9996

References

1. Wang, C. X.; Zhang, J. Q.; Na, S. Analysis of Volatile Halogenated and Aromatic Hydrocarbons and Organophosphorus Pesticides in Water with a Versatile Agilent 7890B Gas Chromatography System and an Agilent 7697A Headspace Sampler, *Agilent Technologies Application Note*, publication number 5991-2787EN.
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