Sensitive and Reproducible Detection of PAHs Using the Agilent 5977A Series GC/MSD

Application Note

Environmental

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Abstract

A method has been developed on the Agilent 5977A Series GC/MSD for the detection of 16 PAHs at levels as low as 5 parts per billion (ppb), with excellent linearity (R^2>0.995) and reproducibility (RSDs $\leq$ 2%).

Introduction

Polycyclic Aromatic Hydrocarbons (PAHs) comprise a large group of chemical compounds that are found in oil, coal, and tar deposits, and create pollution in air, water and soil. They can occur in food either by uptake from the environment or as a result of food processing. Amongst the PAHs are some of the most toxic compounds known, including carcinogens, mutagens, and teratogens. While the list of priority PAHs varies in different countries, the United States Environmental Protection Agency (USEPA) and the European Union (EU) have identified 16 priority PAHs that require monitoring.

This application note demonstrates the ability of the 5977A Series GC/MSD to enable sensitive and reproducible detection of PAHs in 15 minutes, with relative standard deviations (RSDs) at or below 2% at 5 ppb for all 16 monitored PAHs.

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Experimental

Standards and Reagents

PAH standards containing the 16 monitored compounds and the two internal standards were commercially obtained. The working calibration standards were prepared in hexane using the Agilent 7696 Sample Prep WorkBench.

Instruments

The study was performed on an Agilent 7890B Series GC equipped with a Split/Splitless Inlet and coupled to a 5977A Series GC/MSD, using Selected Ion Monitoring (SIM) and Electron Ionization (EI) acquisition modes. The source was equipped with the optional 6 mm extractor lens (p/n G3870-20448). Table 1 lists the instrument conditions.

Table 1. Agilent 7890/5977 Gas Chromatograph and Mass Spectrometer Conditions

GC run conditions	
Analytical column	Agilent HP-5 ms 30 m × 0.25 mm, 0.25 µm (p/n 19191S-433UI)
Injection volume	1 µL
Injection mode	Pulsed splitless
Inlet temperature	290 °C
Liner	Dual-taper, 4 mm id liner, no wool (p/n 5181-3315)
Carrier gas	Helium, constant flow, 1.5 mL/min
Oven program	55 °C for 1 minute 25 °C/min to 320 °C, hold 3 minutes
Transfer line temperature	290 °C
MS conditions	
Solvent delay	4 minutes
Acquisition mode	EI, SIM
Tune	Etune.u
Gain factor	1
Source temperature	350 °C
Quadrupole temperature	150 °C

Acquisition Parameters

Table 2 shows the SIM ions used for acquisition.

Table 2. Acquisition Parameters

Internal standards	**Retention time**	**Quantifier Ion**	**Qualifier Ions**
Phenanthrene-d10	8.375	188	
Chrysene-d12	10.921	240	236,120
Target compounds			
Naphthalene	5.208	128	127,129
Acenaphthylene	6.75	152	153,151
Acenaphthene	6.93	153	154,152
Fluorene	7.419	166	165,167
Phenanthrene	8.349	178	176,179
Anthracene	8.393	178	176,179
Fluoranthene	9.518	202	200,101
Pyrene	9.736	202	200,101
Benzo(a)anthracene	10.902	228	226,229
Chrysene	10.943	228	226,229
Benzo(b)fluoranthene	12.222	252	263,126
Benzo(k)fluorathene	11.933	252	263,126
Benzo(a)pyrene	12.222	252	263,126
Indeno(1,2,3-cd)pyrene	13.481	276	138,277
Dibenzo(a,h)anthracene	13.494	278	139,279
Benzo(g,h,i)perylene	13.813	276	138,277

Results and Discussion

Linearity

Calibration curves were constructed from 5 to 200 ppb with two internal standards. Figure 1 shows the overlays of the total ion current (TIC) traces of the separations of all eight calibration concentrations of the PAH standard mix from 5 to 200 ppb, illustrating excellent reproducibility. Using Gain Factor 1 with a 350 °C source temperature, calibration curves with very high R^2 values were obtained (Table 3).

Reproducibility

Figure 2 illustrates the excellent reproducibility obtainable with the 5977A Series GC/MSD, even at low concentrations. A source temperature of 350 °C, Etune and Gain Factor 1 provided the lowest relative standard deviation values across all 16 PAH compounds, with all values lower than or equal to 2% across 10 injections (Table 4).

Table 3. Calibration Coefficient (R^2) Values Using Etune, Gain Factor 1 and a 350 °C Source Temperature

Compound name	R^2
Naphthalene	0.9999
Acenaphthylene	0.9997
Acenaphthene	0.9999
Fluorene	0.9999
Phenanthrene	0.9998
Anthracene	0.9999
Fluoranthene	0.9999
Pyrene Method	0.9999
Benzo(a)anthracene	0.9998
Chrysene	0.9999
Benzo(b)fluoranthene	0.9957
Benzo(k)fluorathene	0.9961
Benzo(a)pyrene	0.9994
Indeno(1,2,3-cd)pyrene	0.9985
Dibenzo(a,h)anthracene	0.9986
Benzo(g,h,i)perylene	0.9983

8 calibration concentrations: 5, 10, 20, 40, 40, 120, 160, and 200 ppb
Internal standard concentrations were 50 ppb

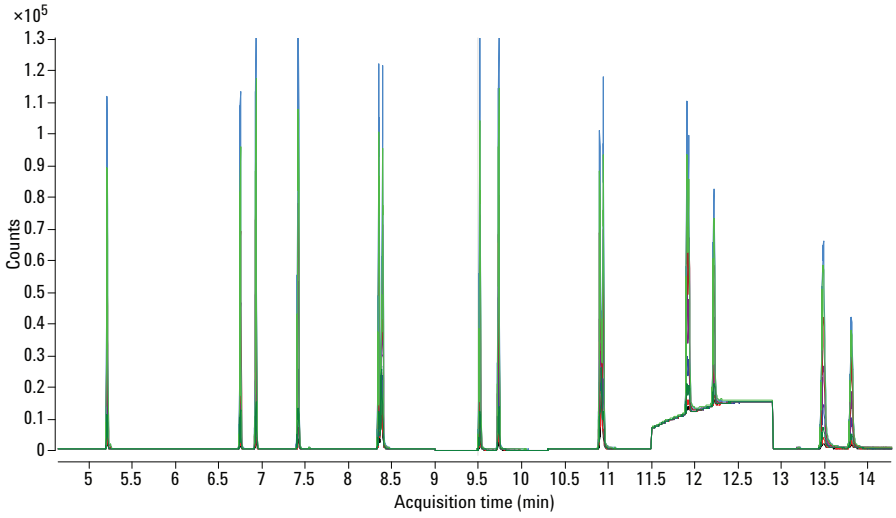

Figure 1. TIC overlays of the separations of all eight PAH standard mix calibration concentrations, from 5 to 200 ppb

Conclusion

The Agilent 5977A GC/MSD provides a stable platform for the sensitive, accurate and reproducible analysis of PAHs at levels as low as 5 ppb. Use of the new Extractor Source and 350 °C source temperature provides the highest relative standard deviations (RSDs), and use of automation like the 7696A Sample Prep WorkBench to prepare standards results in reproducible calibration coefficient (R^2) values.

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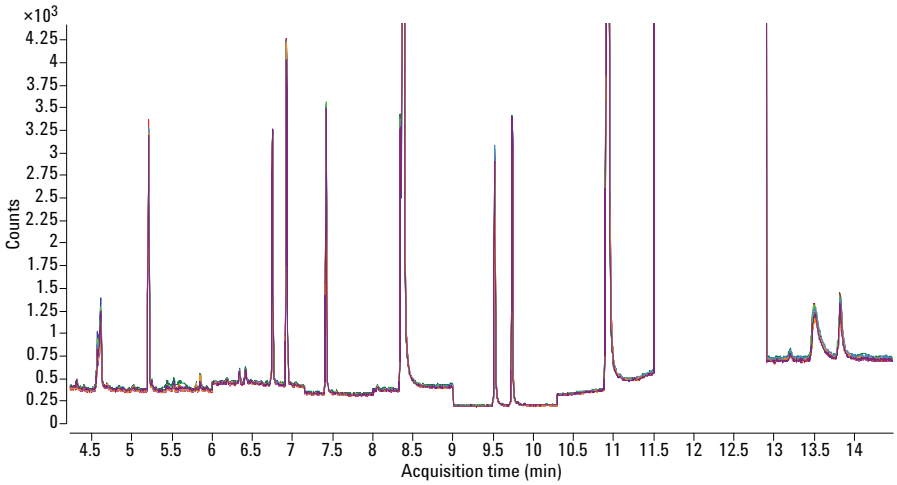

Figure 2. TIC overlays of 10 consecutive separations of PAH standard mix at 5 ppb, using Etune and a source temperature of 350 °C.

Table 4. Relative Standard Deviation Values (RSDs) Using Etune, Gain Factor 1 and a 350 °C Source Temperature

	RSD
Naphthalene	1.9%
Acenaphthylene	0.7
Acenaphthene	0.9
Fluorene	0.7
Phenanthrene	0.9
Anthracene	1.7
Fluoranthene	1.3
Pyrene Method	0.9
Benzo(a)anthracene	1.1
Chrysene	1.8
Benzo(b)fluoranthene	1.2
Benzo(k)fluorathene	1.1
Benzo(a)pyrene	2.0
Indeno(1,2,3-cd)pyrene	1.9
Dibenzo(a,h)anthracene	1.6
Benzo(g,h,i)perylene	1.9

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