# Polycyclic Aromatic Hydrocarbon (PAH) Analysis Using an Agilent J&W DB-5ms Ultra Inert Capillary GC Column

Application

**Environmental and Food Quality**


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## Abstract

**Agilent Technologies Inc. has implemented new testing procedures to more effectively evaluate GC column inertness performance. This new testing procedure employs deliberately aggressive probes to thoroughly investigate column inertness and quality. These aggressive probes, including 1-propionic acid, 4-picoline, and trimethyl phosphate, are used to verify each column's inertness performance.**

**Trace- and ultra trace-level polycyclic aromatic hydrocarbon (PAH) analyses are important tools for accessing environmental quality and foodstuff purity worldwide. In this application, trace-level PAH analyses are demonstrated using electron impact single quadrupole scanning mass spectrometry. In these challenging separations, knowing that the GC column has been thoroughly investigated for column inertness gives the analyst higher confidence in the accuracy of the results.**


## Introduction

Polycyclic aromatic hydrocarbons (PAHs) are both persistent and common in the environment, being found primarily in soil and sediment. These molecules are often associated with the burning of fossil fuels and have been used to fingerprint pollution sources for a given area. Temporal studies in soil are also conducted to evaluate potential natural sources for PAHs and to establish a regional background. [1-2]

Toxicities for this class of molecules range from relatively nontoxic to highly carcinogenic. PAHs have been indentified in edible oils, in roasted and smoked meat products, and in human tissues. Human exposure by ingestion of PAH-containing food substances is of major concern. The need for reliable, sensitive, and robust analytical methods for the analysis of PAHs has been well established globally. [3-5]

PAHs tend to adsorb onto active sites or cold spots within a given chromatographic system, which can lead to false negative analytical results. This application demonstrates PAH analysis using GC capillary columns whose inertness performance has been verified. Capillary GC column activity as a potential source of result uncertainty has been all but eliminated.

## Experimental

An Agilent 6890N GC/5975B MSD equipped with a 7683B autosampler was used for this series of experiments. Table 1 lists the chromatographic conditions used for these analyses. Table 2 lists the flow path consumable supplies used in these experiments.

**Table 1. Chromatographic Conditions**

| | |
|---|---|
| GC: | Agilent 6890N/5973B MSD |
| Sampler: | Agilent 7683B, 5.0 µL syringe (Agilent p/n 5188-5246), 1.0 µL splitless injection, 5 ng each component on column |
| Carrier: | Helium 45 cm/s, constant flow |
| Inlet: | Pulsed splitless; 300 °C, 40 psi until 0.2 min, purge flow 30 mL/min at 0.75 min |
| Inlet liner: | Deactivated dual taper direct connect (Agilent p/n G1544-80700) |
| Column: | Agilent J&W DB-5ms Ultra Inert 30 m x 0.25 mm x 0.25 µm (Agilent p/n 122-5532UI) |
| Oven: | 55 °C (1 min) to 320 °C (25 °C/min), hold 3 min |
| Detection: | MSD source at 300 °C, quadrupole at 180 °C, transfer line at 280 °C, scan range 45 to 450 AMU |

**Table 2. Flow Path Supplies**

| | |
|---|---|
| Vials: | Amber screw cap (Agilent p/n 5182-0716) |
| Vial caps: | Blue screw cap (Agilent p/n 5282-0723) |
| Vial inserts: | 100 µL glass/polymer feet (Agilent p/n 5181-1270) |
| Syringe: | 5 µL (Agilent p/n 5181-1273) |
| Septum: | Advanced Green (Agilent p/n 5183-4759) |
| Inlet liners: | Deactivated dual taper direct connect (Agilent p/n G1544-80700) |
| Ferrules: | 0.4 mm id short; 85/15 Vespel/graphite (Agilent p/n 5181-3323) |
| 20x magnifier : | 20x magnifier loupe (Agilent p/n 430-1020) |

### Sample Preparation

The 16-component PAH standard mix was supplied by Agilent (p/n 8500-6035). Acetone used was Burdick and Jackson Ultra Resi Grade purchased through VWR International, West Chester, PA 19380, USA. The stock PAH solution as delivered had a nominal concentration of 500 µg/mL. The stock was diluted 1:50 and then serially diluted to prepare standard solutions with concentrations of 5, 2, 1, 0.5, 0.1, and 0.05 µg/mL. All solutions were prepared in acetone using class A volumetric pipettes and flasks.

## Results and Discussion

### Baseline Inertness Profile for Ultra Inert Columns

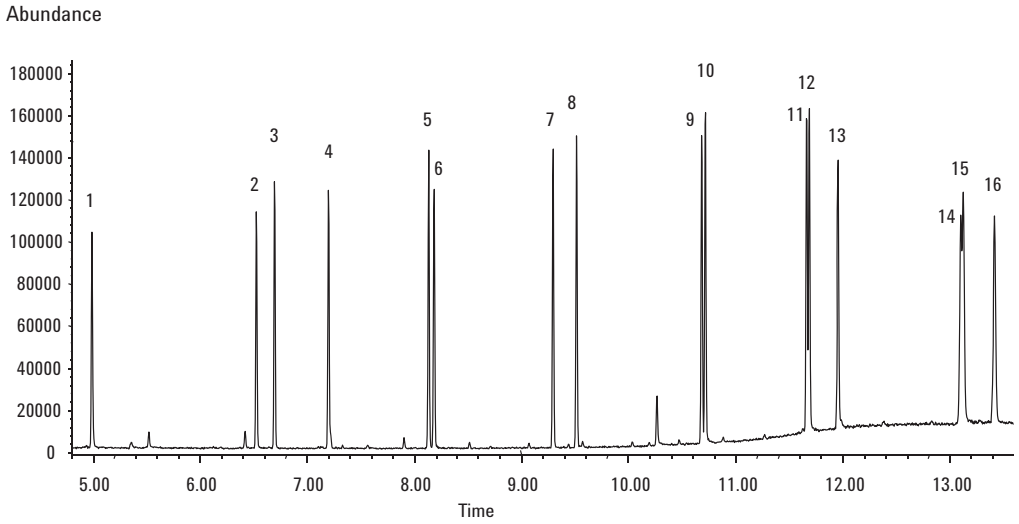
The basic approach for inertness verification for the Agilent J&W Ultra Inert series of capillary GC columns is testing with aggressive active probes at low concentration and low temperature. [6] This is a rigorous approach that establishes consistent baseline inertness profiles for each column in the Agilent J&W Ultra Inert GC column series. The baseline inertness profile then serves as a predictor for successful analysis of chemically active species that tend to adsorb onto active sites particularly at trace levels, like the PAHs in this application. A more detailed description of the test mix and an additional application can be found in references 7 and 8, respectively.

### PAH Analysis

In this application, a 16-component PAH standard mixture was evaluated over a concentration range of 0.05 µg/mL to 5 µg/mL on an Agilent J&W DB-5ms Ultra Inert 30 m x 0.25 mm x 0.25 µm (p/n 122-5532UI). Excellent sensitivity was observed for each of these components across the range studied. Good resolution was obtained in a 15-minute analysis for each of the PAHs with the exception of indeno [1,2,3-c,d]pyrene and dibenz[a,h]anthrancene, which were only partially resolved. Figure 1 shows the total ion chromatogram for a standard injection at the 0.5 µg/mL level; conditions are described in Tables 1 and 2.

Benzo[a]pyrene is a PAH of particular interest due to its toxicity. Figure 2 focuses on this specific analyte with an expanded section of the total ion chromatogram from an injection at the lowest standard level studied. The signal-to-noise ratio indicated was greater than 9:1. Scanning mode was used exclusively throughout these experiments on an Agilent 5975B MSD equipped with an inert electron impact source.

Linearity was excellent across the range studied, giving $R^2$ values of 0.995 or greater in all cases. Figure 3 indicated the correlation coefficients for each of the individual analytes and shows an example linear regression plot for benzo[a]pyrene.




1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benz[a]anthracene
10. Chrysene
11. Benzo[b]fluoranthene
12. Benzo[k]fluoranthene
13. Benzo[a]pyrene
14. Indeno[1,2,3-cd]pyrene
15. Dibenz[a,h]anthracene
16. Benzo[g,h,i]perylene

**Figure 1. Total ion chromatogram (scanning mode) of a 1-µL injection of the 0.5 µg/mL standard solution on an Agilent J&W DB-5ms Ultra Inert 30 m x 0.25 mm x 0.25 µm capillary GC column (p/n 122-5532UI). This injection represents an on-column loading of 0.5 ng per component.**

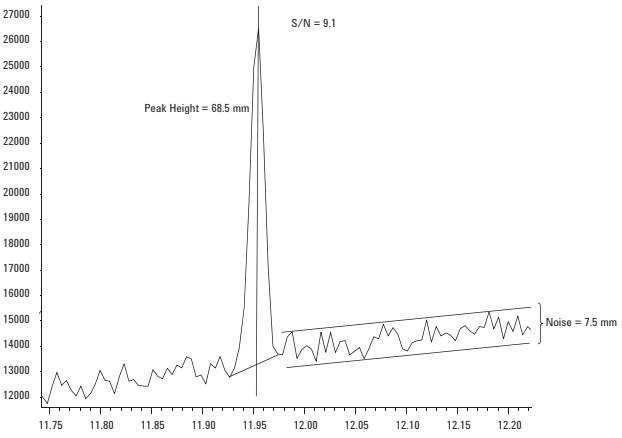



**Figure 2. Enlarged section of the total ion chromatogram for a 1-µL injection of 0.05 µg/mL standard PAH mix. The peak in the figure is the benzo[a]pyrene peak, a PAH of particular interest due to its toxicity. This injection represents an on-column loading of 0.05 ng per component.**

## Conclusions

This application successfully demonstrates the use of an Agilent J&W DB-5ms Ultra Inert capillary GC column for trace-level PAHs in a 15-minute analysis. Linearity was excellent for all 16 PAHs studied, yielding 0.995 or greater $R^2$ values down to a 0.05 ng column loading of each component. One of the reasons for excellent linearity and high $R^2$ values is the highly inert surface of the column. The lack of chemically active sites makes these columns an excellent choice for trace-level applications.

This study was done exclusively using scanning mode on an Agilent 6890/5975B GC/MSD equipped with an inert electron impact source. The signal-to-noise ratio for a 0.05 ng on-column loading of benzo[a]pyrene was greater than 9 to 1 with this system. This result shows clearly the power of using an Agilent J&W DB-5ms Ultra Inert column for trace-level PAH analysis. Excellent sensitivity was observed for the PAHs in this study even in scanning mode. Lower limits of quantification are expected from using either combined SIM/scan or SIM modes, or one of Agilent's newest GC/MS offerings, such as the 7890A/5975C GC/MSD Triple-Axis Detector coupled with an Agilent J&W DB-5ms Ultra Inert GC capillary column.

| | $R^2$ |
|---|---|
| Naphthalene | 1.000 |
| Acenaphthylene | 0.999 |
| Acenaphthene | 0.999 |
| Fluorene | 0.999 |
| Phenanthrene | 0.999 |
| Anthracene | 0.999 |
| Fluoranthene | 0.999 |
| Pyrene | 0.999 |
| Benz[a]anthracene | 0.998 |
| Chrysene | 0.999 |
| Benzo[b]fluoranthene | 0.998 |
| Benzo[k]fluoranthene | 0.998 |
| Benzo[a]pyrene | 0.998 |
| Indeno[1,2,3-cd]pyrene | 0.995 |
| Dibenz[a,h]anthracene | 0.998 |
| Benzo[g,h,i]perylene | 0.997 |

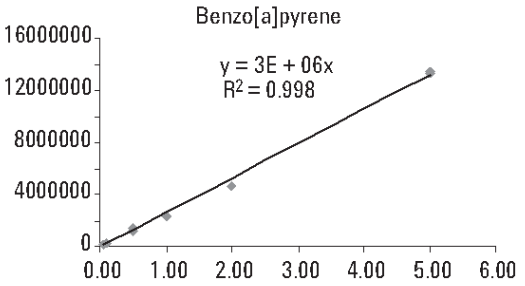



**Figure 3. Correlation coefficients for the 16 components over the 0.05 µg/mL to 5.0 µg/mL range of this study and an example linear regression plot.**

## References


1. Wolfgang Wilcke, Martin Krauss, Grigorij Safronov, Alexej D. Fokin, and Martin Kaupenjohann, "Poylcyclic Aromatic Hydrocarbons (PAHs) in Soils of the Moscow Region – Concentrations, Temporal Trends, and Small-Scale Distribution," *Journal of Environmental Quality* 34: 1581–1590 (2005)
2. Mai Bixian, Fu Jiamo, Zhang Gan, Lin Zheng, Min Yushun, Sheng Guoying, and Wang Xingmin, "Polycyclic Aromatic Hydrocarbons in Sediments from the Pearl River and Estuary, China: Spatial and Temporal Distribution and Sources," *Applied Geochemistry,* volume 16, issues 11–12, August-September 2001, pages 1429–1445
3. Mike Szelewski, "Synchronous SIM/Scan Low-Level PAH Analysis Using Agilent Technologies 6890/5975 Inert GC/MSD," Agilent Technologies publication 5989-4184EN, November 9, 2005
4. U.S. EPA Method 8270D, Revision 4, February 2007, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)"
5. Thomas Wenzl, Rupert Simon, Juliane Kleiner, and Elke Anklam, "Analytical Methods for Polycyclic Aromatic Hydrocarbons (PAHs) in Food and the Environment Needed for New Food Legislation in the European Union," *Trends in Analytical Chemistry*, Vol. 25, No. 7, 2006
6. Mitch Hastings, Allen K. Vickers, and Cameron George "Inertness Comparison of Sample of 5% Phenyldimethylpolysiloxane Columns," Poster Presentation, 54th Annual Pittsburg Conference, Orlando, FL, March 2003
7. "Agilent J&W Ultra Inert GC Columns: A New Tool to Battle Challenging Active Analytes," Agilent Technologies publication 5989-8685EN, May 29, 2008
8. Kenneth Lynam "Semivolatile Analysis Using an Inertness Performance Tested Agilent J&W Ultra Inert DB-5ms Column," Agilent Technologies publication 5989-8616EN, May 13, 2008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