PBDE Analysis Using an Agilent J&W DB-5ms Ultra Inert GC Column

Application Brief

Environmental

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Introduction

Polybrominated diphenyl ether (PBDE) analyses are important tools for understanding food supply and environmental quality worldwide. In this application, trace-level PBDE analysis is demonstrated using electron impact GC–MS. For these challenging separations, knowing that each GC column has been thoroughly tested for column inertness gives the analyst higher confidence in the accuracy of the results.

Agilent has implemented new testing procedures to more effectively evaluate GC column inertness performance. These new testing procedures employ deliberately aggressive probes to thoroughly investigate column inertness and quality. These extremely active probes, including 1-propionic acid, 4-picoline and trimethyl phosphate, are used to verify each column's inertness performance. Capillary GC column activity as a potential source of result uncertainty has been virtually eliminated with the Ultra Inert series of columns.

PBDE Analyses

PBDE-209 is a particularly challenging analyte because of its long retention and tendency to degrade with high-temperature exposure. High-temperature thermal stability is an issue for this class of compounds, but is more pronounced for BDE-209, as it is highly brominated and well retained. One key to successful BDE analysis is to limit the time that these compounds are exposed to high temperatures. A 15-m long column, as opposed to a typical 30-m long column was used in this instance to limit residence time for BDE-209. Fortunately, the BDEs resolve well, with symmetrical peak shapes, when using Agilent J&W DB-5ms ultra inert phase, enabling successful separation on the shorter column. Figure 1 shows a total ion chromatogram of the eight BDEs investigated in this study.

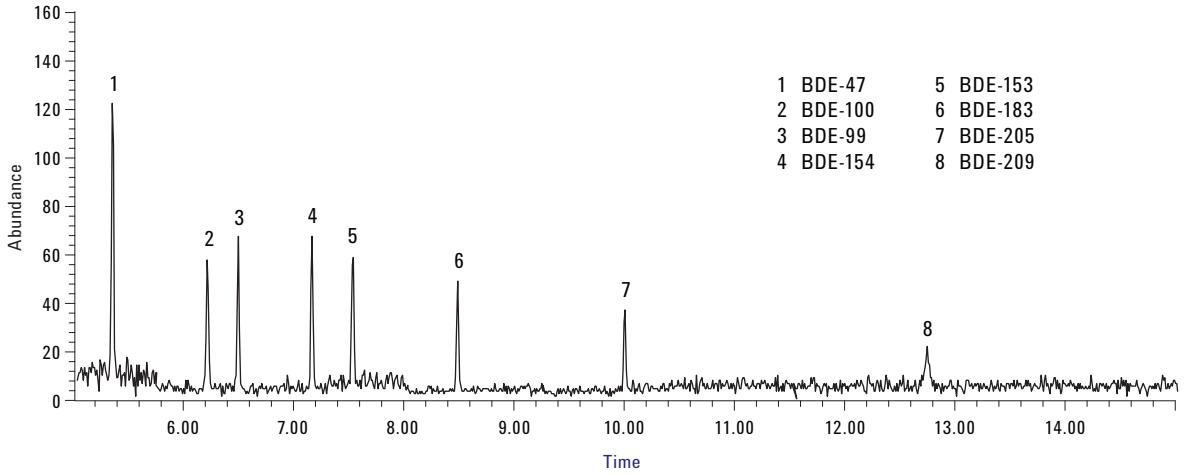

Figure 1. Total ion chromatogram (SIM mode) of a 0.005 ng (BDEs -47, -100, -99, -154, -153, -183 and -205) and 0.025 ng (BDE-209) on-column loading on an Agilent J&W DB-5ms Ultra Inert 15 m x 0.25 mm, 0.25 µm capillary GC column (p/n 122-5512UI).

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 72 cm/s, constant flow
Inlet	Pulsed splitless; 325 °C, 20 psi until 1.5 min, purge flow 50 mL/min at 2.0 min
Inlet liner	Deactivated dual taper direct connect (Agilent p/n G1544-80700)
Column	Agilent J&W DB-5ms Ultra Inert 15 m × 0.25 mm, 0.25 µm (Agilent p/n 122-5512UI)
Oven	150 to 325 °C (17 °C/min), hold 5 min
Detection	MSD source at 300 °C, quadrupole at 150 °C, transfer line at 300 °C, scan range 200–1000 amu

This application successfully demonstrates the use of a 15 m Agilent J&W DB-5ms Ultra Inert capillary GC column for trace-level BDEs in a 15-min analysis. Linearity was excellent for all eight BDEs studied, yielding 0.997 or greater R^2 values down to a 0.005 ng (0.025 ng for BDE-209) on-column loading of each component. One of the reasons for the 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.

The Agilent 6890/5975B GC/MSD (SIM mode) equipped with an inert electron impact source had excellent sensitivity with even the most challenging BDE in this set, BDE-209. The signal-to-noise ratio for a 0.025 ng on-column loading of BDE-209 was greater than three to one with this system. This result shows clearly the power of using an Agilent J&W DB-5ms Ultra Inert capillary GC column for trace-level BDE analysis. Lower limits of quantification are expected when using one of Agilent's latest GC/MS offerings, such as the 6890N/5975C GC/MSD Triple-Axis Detector coupled with an Agilent J&W DB-5ms Ultra Inert GC capillary column.

Access the full application note at:
http://www.chem.agilent.com/Library/applications/5989-9571EN.pdf

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