PHTHALATES:
LONG-TERM RELIABILITY

Technology Advantage: Agilent Intuvo 9000 GC with MSD-HES

Introduction

The need for a robust analytical method for the identification and quantitation of phthalate esters (phthalates) has increased over the past several years. Their common use in plasticizers has increased potential exposure (either accidental or intentional) from food, housewares, and even children's toys. This drives the need for a reliable method of analysis. Phthalates can be difficult to analyze using GC/MS due to a variety of reasons including poor peak shape, loss of sensitivity, and signal loss over time.

Issues with analyses such as those listed above can be mitigated with a conventional gas chromatograph/mass spectrometer system, but the Agilent Intuvo 9000 Gas Chromatograph, coupled to a mass spectrometer equipped with a high efficiency source (HES) provides additional advantages:

- Simplified column installation
- Innovative inert flow path
- Smaller footprint

A redesigned modular flow path simplifies column installation, while the innovative inert flow path maintains chromatographic integrity through the course of the analysis. Lastly, the Intuvo 9000 GC is only 27 cm wide affording additional flexibility, especially in laboratories with limited bench space.

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Table 1. Analytes and SIM ions for the phthalate standard.

	Analyte	Target ion
1	Dimethyl phthalate	163
2	Diethyl phthalate	149
3	Diisobutyl phthalate	149
4	Di-*n*-butyl phthalate	149
5	*Bis*(2-methyoxyethyl) phthalate	59
6	*Bis*(4-methyl-2-pentyl) phthalate	149
7	*Bis*(2-ethoxyethyl) phthalate	72
8	Dipentyl phthalate	149
9	Dihexyl phthalate	149
10	Butyl benzyl phthalate	149
11	*Bis*(2-*n*-butoxyethyl) phthalate	149
12	Dicyclohexyl phthalate	149
13	*Bis*(2-ethylhexyl) phthalate	149
14	Di-*n*-octyl phthalate	149
15	Dinonyl phthalate	149

Experimental

An Intuvo 9000 GC was coupled to an MS-HES. A 30 m Intuvo HP-5ms Ultra Inert column was installed and run at 1 mL/min. A variable oven program was used to separate 14 phthalates in a standard obtained from Ultra Scientific. The standard was diluted to 200 ppb in isooctane, and analyzed in Selected Ion Monitoring mode (SIM). Table 1 lists the peak identification and target ions.

Results and Discussion

Phthalate response was monitored over time by making approximately 120 consecutive injections on to the 30 m Intuvo HP-5ms UI column. After 10–15 injections, the phthalate response was remarkably consistent with a standard instrument configuration (Figure 1).

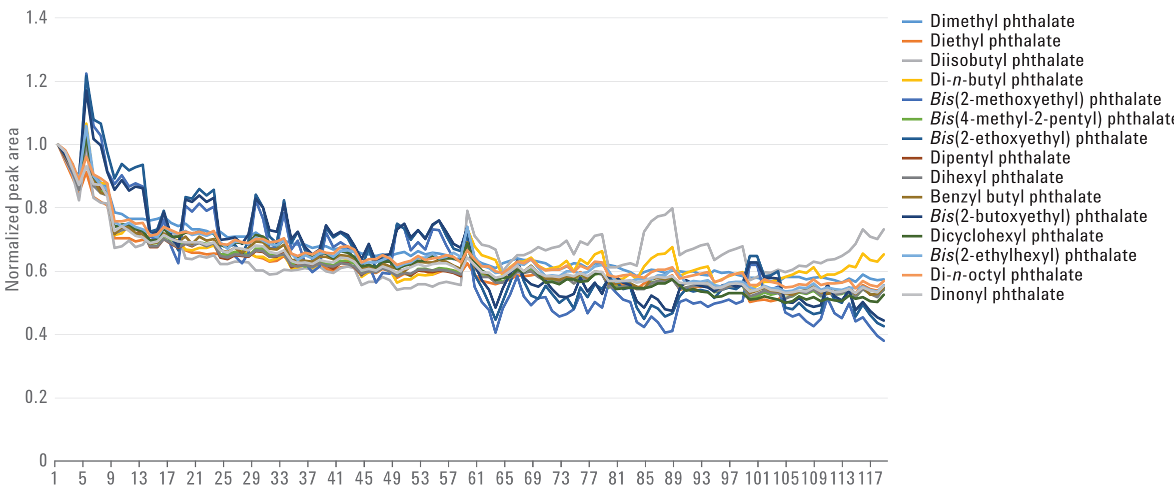

Figure 1. Phthalate normalized area response for 117 injections.

The initial decrease in phthalate response was not unexpected, as the analytical column was installed without preconditioning. In addition to demonstrating a consistent response, the peak shape was unchanged over the course of the analysis. Sharp, symmetric peaks were maintained over 117 injections (Figure 2). Peaks 14 and 15 (di-*n*-octyl phthalate and dinonyl phthatlate) are highlighted in the inset chromatogram.

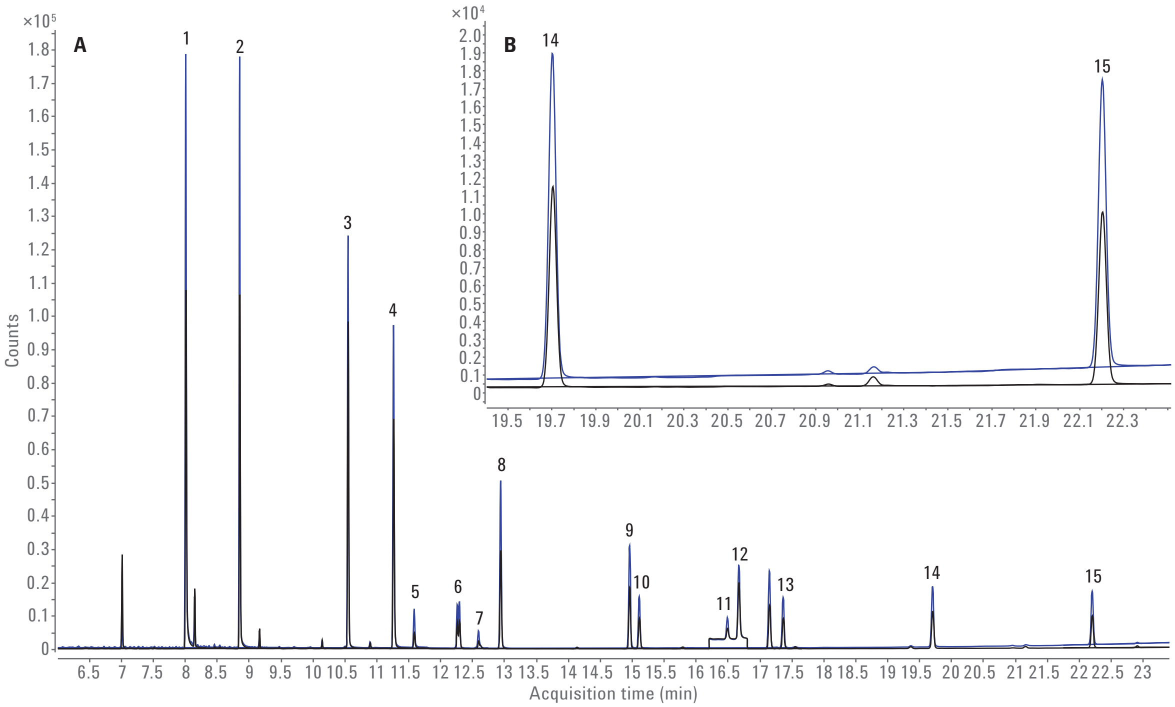

Figure 2. The phthalate standard diluted to 200 ppb at initial injection (blue) and after 117 injections (black). Aside from the difference in response due to initial column bleed, there is no change in peak shape over time.

Conclusion

The Agilent Intuvo 9000 GC, coupled with an MS-HES, and a 30 m Intuvo HP-5ms Ultra Inert column delivers consistent peak shape and area response for approximately 120 injections of a phthalate standard at 200 ppb. This demonstrates a robust and reliable method for analyzing a range of phthalates at a relatively low concentration. The innovative flow path simplifies method development and analysis with the inertness required to maintain acceptable peak shape and response over multiple analyses.

Tel: (+34) 93.590.28.50 Fax: (+34) 93.675.05.16 www.ingenieria-analitica.com
inf@ingenieria-analitica.com

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