Agilent Inert Flow Path Enhancements: Impact on Semivolatile Analysis

Application Note

Environmental

Author

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Abstract

A comparison between an Agilent 7890/5975C GC/MS equipped with Agilent Inert Flow Path components and an alternative vendor's offering shows superior results for the Agilent flow path for semivolatile analysis. The Agilent system delivers higher area responses, sharper peaks, and improved resolution for semivolatile analytes such as pentachlorophenol, terbuphos, chlorthalonil, simazine, and atrazine.

Introduction

The semivolatile analyte set includes a number of active substances that readily find and adsorb onto active sites anywhere in the flow path of a modern GC/MS system. In gas phase analysis, analytes must survive the trip from injection to detection. This is a difficult process when compounds of interest are active analytes, tracked at low concentration levels, and the flow path has surface activity. As laboratories process heavy matrix samples using minimal sample preparation and detection limits for GC/MS, the need for a suite of inert flow path components that line the road analytes travel becomes a necessity. Activity anywhere in the flow path can lead to poor peak shapes, poor quantitation, or missed analytes.

Previous efforts to eliminate active sites throughout the flow path have focused on column and liner deactivation strategies [1,2]. These strategies have succeeded to the point where a keen focus on additional components in the flow path is the next logical step toward achieving a totally inert flow path. Inlet weldments, gold seals, and ferrules used to make gas tight connections are all flow path components that come in contact with analytes of interest [3]. Lessons learned developing surface deactivation strategies for Agilent J&W Ultra Inert columns, liners, and inert MS source component designs serve as an excellent foundation for taking inertness to the next level. These strategies were employed to expand the inert flow path to include split/splitless top and shell inlet weldments, Ultra Inert Gold Seals, and UltiMetal Plus Flexible Metal ferrules. This application note illustrates a preliminary evaluation of the latest version of the Agilent Inert Flow Path (Agilent IFP) solution for semivolatiles testing.

Example chromatograms of semivolatile analyte mixes injected on an Agilent IFP and an alternative vendor's similarly equipped split/splitless inlet highlight the impact that the Agilent IFP has on chemically active semivolatiles. For the sake of comparison, the alternative vendor's flow path was assembled from their premium components, consisting of a 5% Sil MS column, a liner with treated wool, treated inlet seal, and deactivated top and shell weldments.

Experimental

The GC/MS system used to compare inlet inertness consisted of an Agilent 7890 GC coupled to an Agilent 5975C MSD with a triple-axis detector. A single Agilent 7683B auto-injector was used and switched between front and back split/splitless inlets. The front inlet was set up as an Agilent Inert Flow Path inlet and the rear inlet was equipped with similar components from the alternative vendor. As much as possible, conditions for both front and rear inlet experiments were kept the same.

Standard preparation

A 29-component GC/MS semivolatiles analyzer checkout mix at a nominal concentration of 10 ng/µL was obtained from Agilent Technologies, Inc. (Santa Clara, CA) (p/n 5190-0473). This mixture was transferred and serially diluted using Class A volumetric glassware and positive displacement syringes. Ultra Resi-analyzed grade dichloromethane was purchased through VWR International.

Conditions – Agilent Inert Flow Path

Column:	Agilent J&W DB-UI 8270D, 20 m × 0.18 mm, 0.36 µm (p/n 121-9723)
Carrier:	Helium, 48.5 cm/s (1.2 mL/min) set at 32 °C, EPC constant flow
Oven:	32 °C 2.5 min hold, 25 °C/min to 320 °C (4.8 min hold)
Injection:	0.5 µL Splitless 230 °C, purge flow on at 1.42 min
Detector:	MSD SCAN Mode 40 to 450 amu, 300 °C source temperature, 150 °C quad temperature, 320 °C transfer line
Gas purifier:	Gas Clean GC/MS 1/8 inch kit (p/n CP17974)
Inlet:	Split/splitless with inert shell and top weldments (p/n G3452-60570 and G3452-60586)
Inlet liner:	Agilent Ultra Inert single taper with wool (p/n 5190-2293)
Gold seal:	Agilent Ultra Inert Gold Seal (p/n 5190-6144 UI)
Syringe:	5 µL Blue line (p/n G4513-80206)
Ferrules:	UltiMetal Plus Flexible Metal ferrule at inlet (p/n G3188-27501), MS (p/n 5188-5361)
Column nut:	Universal column nut, 1/16 inch hex, 2 pk (p/n 5181-8830) for inlet
MS nut:	MS interface column nut (p/n 05988-20066)
MSD ferrule:	85/15 Vespel/graphite (p/n 5062-3508)

Conditions - alternative vendor flow path

Column:	Rxi-5Sil MS, 20 m × 0.18 mm, 0.36 µm
Carrier:	Helium, 48.5 cm/s (1.2 mL/min) set at 32 °C, EPC constant flow
Oven:	32 °C 2.5 min hold, 25 °C/min to 320 °C (4.8 min hold)
Injection:	0.5 µL Splitless 230 °C, purge flow on at 1.42 min
Detector:	MSD SCAN Mode 40 to 450 amu, 300 °C source temperature, 150 °C quad temperature, 320 °C transfer line
Gas purifier:	Gas Clean GC/MS, 1/8 inch kit (p/n CP17974)
Inlet:	Split/splitless with passivated shell and top weldments
Inlet liner:	Sky single taper with wool
Gold seal:	Siltek-treated inlet seal
Syringe:	5 µL Blue line (p/n G4513-80206)
Ferrules:	Agilent UltiMetal Plus Flexible Metal ferrule at inlet (p/n G3188-27501), MS (p/n 5188-5361)
Column nut:	Universal column nut, 1/16 inch hex, 2 pk (p/n 5181-8830) for inlet
MS nut:	MS interface column nut (p/n 05988-20066)
MSD ferrule:	85/15 Vespel/graphite, 0.4 mm preconditioned (p/n 5062-3508)

Additional Agilent supplies

Vials:	Amber silanized screw top vials (p/n 5183-2072)
Vial caps:	Blue screw caps (p/n 5185-5820)
Vial inserts:	250 µL Glass/polymer feet (p/n 5181-8872)
Septa:	11 mm Non-Stick Advanced Green (p/n 5183-4759)
Inlet ferrule:	Agilent UltiMetal Plus Flexible Metal ferrule, 10 pk (p/n G3188-27501)
Magnifier:	20x Magnifier loop (p/n 430-1020)
Syringes:	Replaceable needle, PTFE plunger, 1 mL (p/n 5190-1539), 0.5 mL (p/n 5190-1525)

Results and Discussion

Figure 1 shows example chromatograms of the semivolatiles check out mix at the 0.25 ng/component on-column level, to compare Agilent and non-Agilent flow paths. Considering the active acidic phenols, bases and active pesticides in this mix, peak shapes and responses are excellent.

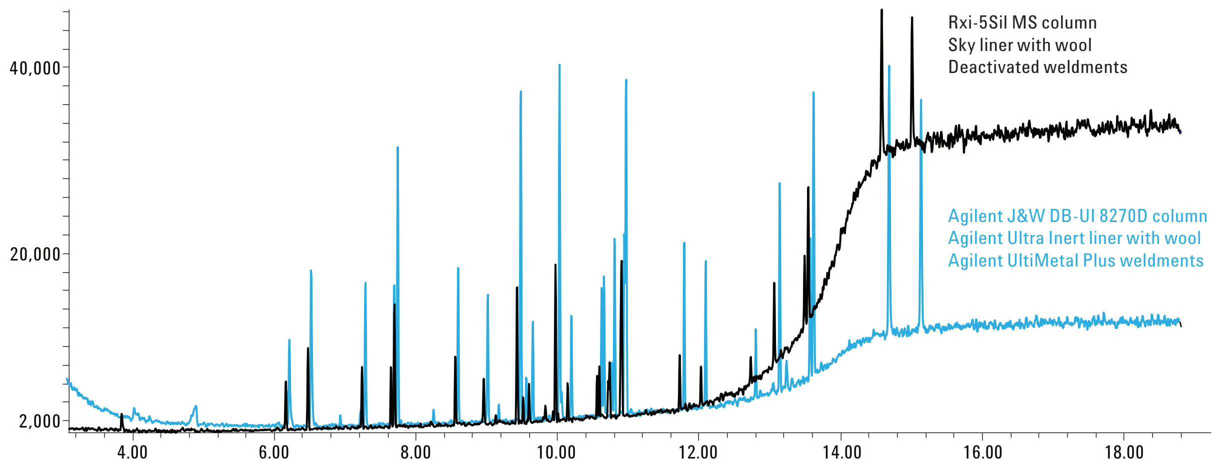

Figure 1. Total ion chromatogram overlay of Agilent IFP (blue trace) versus an alternative vendor flow path (black trace).

Figure 2 is a chromatographic overlay of total ion chromatogram traces of analyte response using Agilent IFP components in blue and alternative vendor components in black. More signal strength and sharper peaks were observed for the acidic phenols using the Agilent IFP.

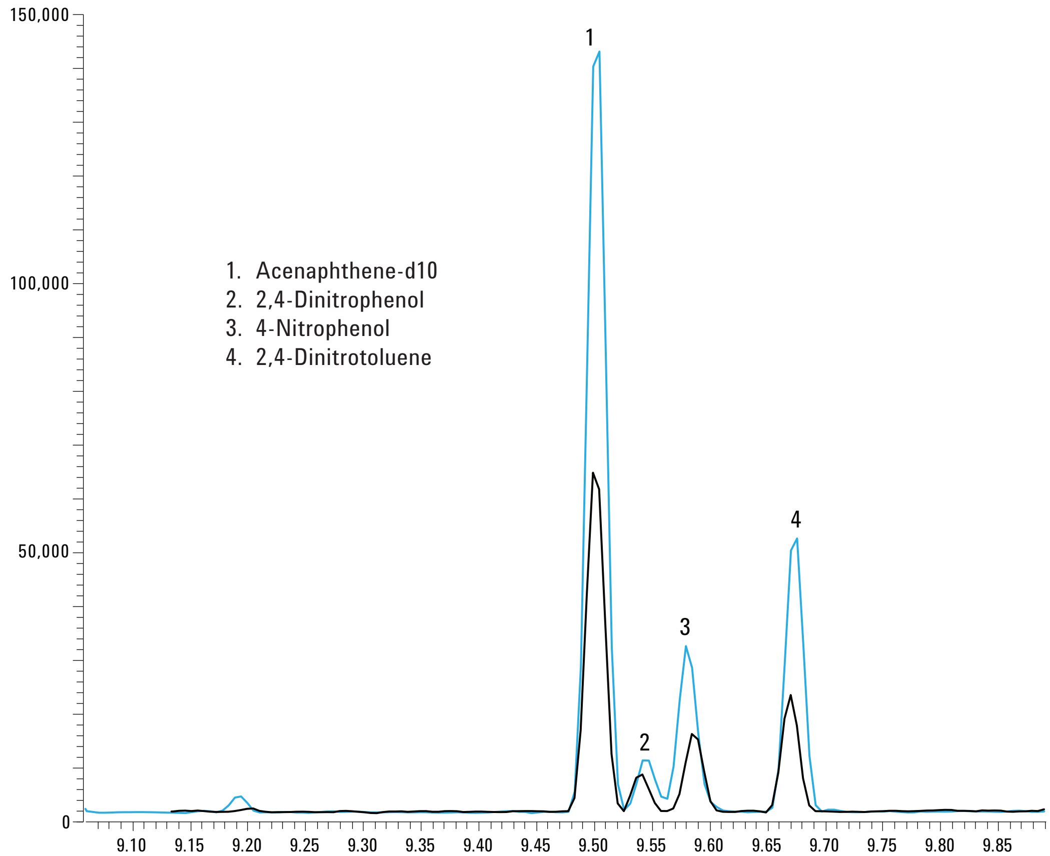

Figure 2. Agilent IFP (blue trace) shows improved performance for acidic phenols, with more signal and sharper peaks.

Figure 3 shows a total ion chromatogram overlay highlighting the impact of having more signal and sharper peaks on chromatographic resolution. Pentachlorophenol was resolved from terbuphos only on the Agilent IFP inlet (blue trace). Better peak resolution was also seen between chlorthalonil and phenanthrene-d10 using the Agilent IFP equipped inlet and DB-8270D UI column. More signal and sharper peaks were also observed for the triazine herbicides, simazine and atrazine.

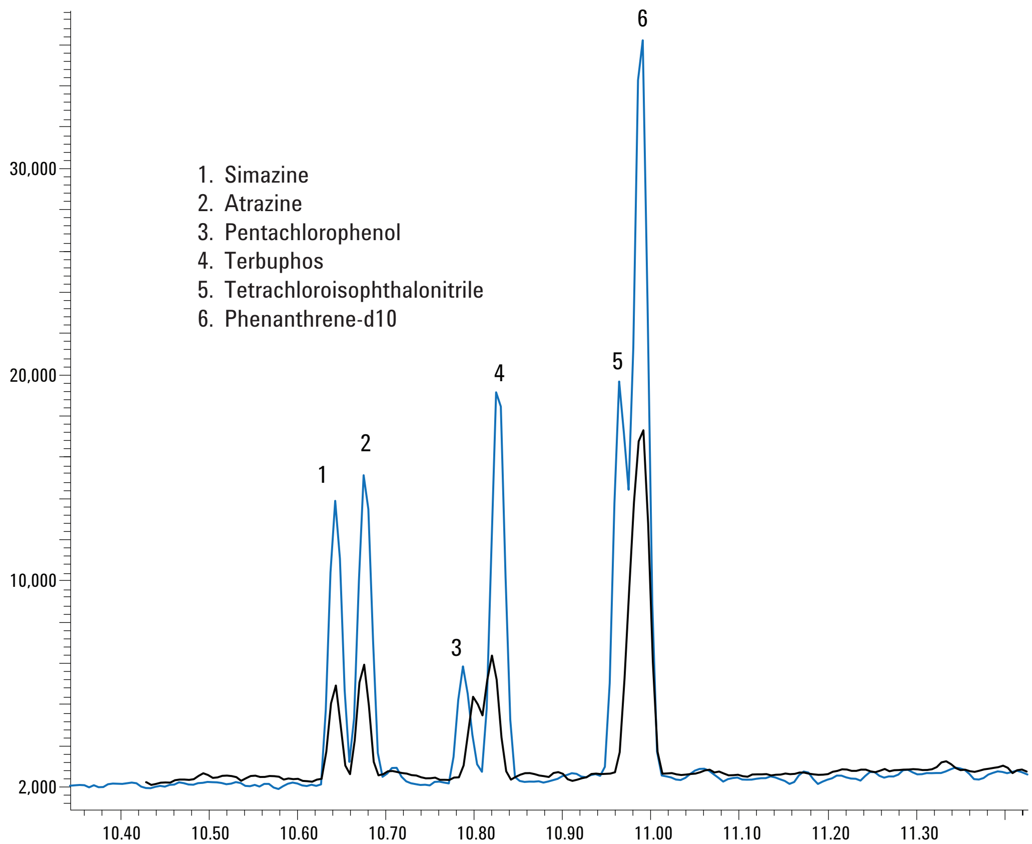

Figure 3. Agilent IFP (blue trace) yields more pentachlorophenol signal and resolution enhancement for challenging pesticides.

Delivery of inertness performance must go hand-in-hand with keeping siloxane column bleed to a minimum at elevated temperature. Figure 4 shows that the Agilent IFP-equipped inlet gave higher analyte signal for late eluting compounds, with lower bleed up to 320 °C, than the alternative vendor inlet.

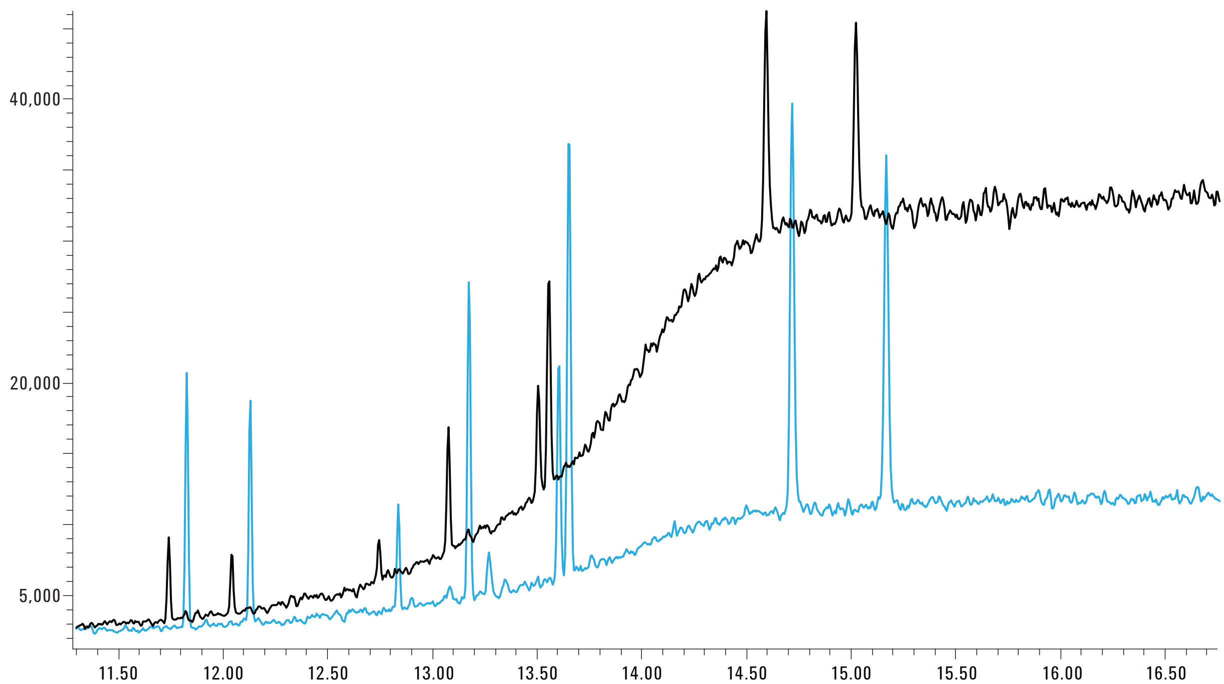

Figure 4. Agilent IFP (blue trace) delivers more signal and less column bleed at elevated temperature.

Conclusions

The Agilent 7890/5975C GC/MS equipped with an Agilent Inert Flow Path inlet showed higher signal response, sharper peaks, and lower column bleed than the alternative vendor's offering. More signal strength and better shape had a significant impact on the separation of pentachlorophenol and terbuphos. These peaks were resolved only on the Agilent IFP-equipped inlet and coeluted on the inlet from the alternative vendor.

These results suggest that better detection and quantitation of semivolatiles is readily achievable through the use of an Agilent GC/MS equipped with Agilent IFP components. Thoroughly deactivated Ultra Inert liners, Ultra Inert Gold Seals, inlet weldments, Ultra Inert columns, and inert MS source components all work in harmony to deliver optimal performance for active analytes.

References

1. K. Lynam. *Semivolatile Analysis Using an Inertness Performance Tested Agilent J&W DB-5ms Ultra Inert Column.* Application note, Agilent Technologies, Inc. Publication number 5989-8616EN (2008).
2. L. Zhao, *et al.*, *Evaluation of the Ultra Inert Liner Deactivation for Active Compounds by GC.* Application note, Agilent Technologies, Inc. Publication number 5990-7380EN (2011).
3. Anon. *Optimizing Your GC Flow Path for Inertness.* Poster, Agilent Technologies, Inc. www.agilent.com/chem/uiorder.

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