Novel column chemistry raises the bar on sensitivity and data accuracy in the analysis of Semivolatile organic compounds

Jennifer Sanderson, Vanessa Abercrombie, Frans Biermans, Anastasia Andrianova, Joel Ferrer, Ashlee Gerardi

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POSTER

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Introduction

The evolution of ionization sources continues to push the limits of detection in Gas Chromatography Mass Spectrometry. As source technologies advance, column technology must advance as well. An increase in detection of analytes also results in unwanted increase detection of background noise. Mass spec GC column technology that lowers bleed can enhance sensitivity and data accuracy. In this poster, we will examine how column attributes, like column bleed, thermal stability and inertness can further benefit the sensitivity and accuracy of a mass spectrometer. When used in conjunction with the state-of-the-art Agilent 7010 triple quadrupole GC/MS system, we will illustrate achievable data parameters, like sensitivity limits at trace levels, retention time consistencies and data accuracy for active analytes.

Experimental

An Agilent 8890 GC with a 7010D TQ system upgraded with the HES 2.0 was used for the analysis. Agilent Semi-volatiles Standard (p/n: SVM-8270-1) was prepared in Dichloromethane for calibration standards to be analyzed as 10 pg on column to 1000 pg on column.

8890	
Inlet	300 °C, Pulsed Splitless Mode
Injection Volume	0.5 µL
Inlet Liner	Ultra Inert, split, low pressure drop (p/n 5190-2295)
Injection Pulse Pressure	30 psi Until 0.6 min
Purge Flow to Split Vent	50 mL/min at 0.6 min
Septum Purge Flow	3 mL/min
Oven	40 °C (0.5 min), ramp 10 °C/min to 100 °C, ramp 25 °C/min to 260 °C, ramp 5 °C/min to 280 °C, 15 °C/min to 320 °C (5 min), 10 °C/min to 330 °C (10 min), 10 °C/min to 340 °C (10 min)
Column	
Carrier Gas	Helium, 1.3 mL/min, Constant Flow
Column	DB-5Q 30m x 0.25 mm x 0.25 µm (p/n 122-5532Q) DB-5ms UI 30m x 0.25 mm x 0.25 µm (p/n 122-5532UI) 5MS Type Column Y 30m x 0.25 mm x 0.25 µm
Inlet Connection	Split/Splitless Inlet (S/SL)
Outlet Connection	MSD

Model	7010D
Source	HES
Mode	DMRM/Scan
Solvent Delay	2.5 min
Source Temperature	300 °C
Quad Temperature	175 °C
Gain	1.0

Increased Thermal Stability

Figure 1 demonstrates the impact of the increased thermal stability of the Agilent J&W DB-5Q in comparison to a commercially available 5ms Type GC column. The increased thermal stability allows for a perceived decrease in column bleed, which is ideal when analyzing low concentrations of compounds at high temperatures.

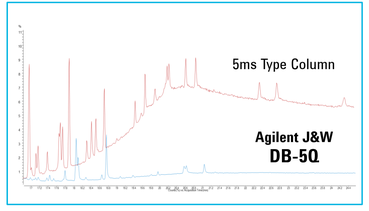

Figure 1. 50 pg on column Semi-volatile standard zoomed in on the end of the temperature cycle.

Easy Adoption

Figure 4 demonstrates a standard 8270 compounds, analyzed at 1000 pg on column, using the J&W DB-5Q and the J&W DB-5MS UI, using the same instrumentation method. The similar selectivity allows for upgrading analytical methods without need for re-developing the method.

Additionally, with the same selectivity, there is no need to update retention times, which makes the DB-5Q compatible with existing Retention Time Locking libraries.

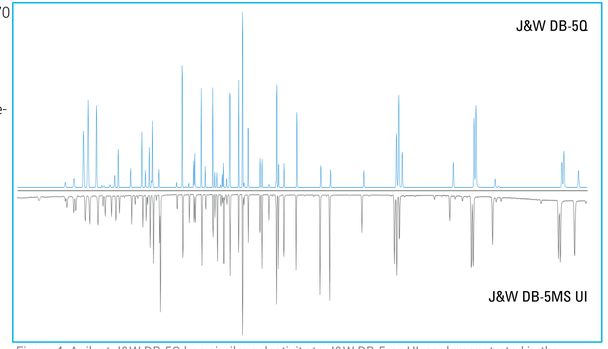

Figure 4. Agilent J&W DB-5Q has similar selectivity to J&W DB-5ms UI, as demonstrated in the analysis of 8270 compounds.

HES 2.0: Novel Electron Ionization Source Technology

The new HES 2.0 ion source, as seen in Figure 5 and Figure 6, is equipped with a novel dipolar RF lens that redirects carrier gas and low mass ions by >95%.

The deflected ions land on adjacent lenses and are pumped out prior to entering the mass analyzer, providing reduced noise and extended instrument robustness while maintaining sensitivity. A ramped RF amplitude vs mass is implemented to avoid spectra tilt. The reduction of noise allows for a further increase of sensitivity to Attogram-level detection limits.

Built-in intelligence features such as SWARM autotune and early maintenance feedback further enhance instrument performance and diagnostic capabilities.

The HES 2.0 will be available in Agilent's 7010D GC/TQ or as part of an upgrade kit to an existing 7010 or 7000 GC/TQ system.

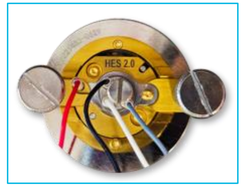

Figure 5. Front view of new HES 2.0

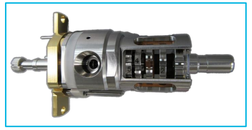

Figure 6. Side view of HES 2.0.

Robustness

A heavy matrix of soil diluted in DCM was used to stress the robustness of the GC Column along with thermal cycling. The tuning standard of DFTTP Tune was analyzed every five matrix injections, with %DDT breakdown indicating when to replace the inlet liner and peak shape of Pentachlorophenol and Benzidine being indicators of column activity. The inlet liner and septum were replaced every 20 matrix injections, as indicated by %DDT breakdown. Figure 2 demonstrates that after 200 matrix injections. After 200 matrix injections retention times and peak shape for all compounds are consistent, demonstrating the robustness of the DB-5Q column phase.

Figure 3 demonstrates the comparison of the peak shape of Pentachlorophenol on the DB-5Q to a commercially available 5ms Type Column. The tailing factor of Pentachlorophenol has increased at the same concentration, and the poor peak shape leads to a decrease in Signal to Noise response. When working with difficult methods, such as EPA 8270 it is important to use a column phase, such as DB-5Q, which are robust enough to maintain retention times and peak shape even when working with difficult matrices, such as soil extracts.

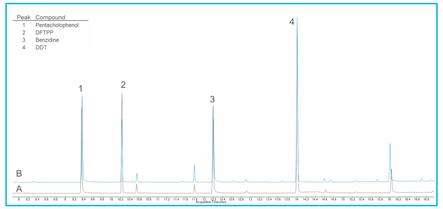

Figure 2. DFTPP Tune mix. A) Initial B) After 200 Matrix Injections (RT Stability and peak shape) on DB-5Q.

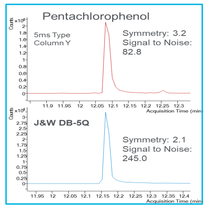

Figure 3. Comparison of peak shape of 250 pg on column of Semi-volatile compounds

Expand Limits of Detection by DMRM and Scan Simultaneously

With the improved sensitivity of the ion source along with the triple off axis detector configuration allows for fast MRM speeds, which lead to the ability to acquire data in Dynamic Multiple Reaction Mode (dMRM) as well as Scan Mode at the same time. This improvement allows for the possibility of performing a targeted analysis and untargeted at the same time. dMRM is very useful in setting up Multi Reaction Mode methods, as once the retention time is inputted into the Mass Hunter Acquisition software, the dwell time is calculated automatically. While this streamlines the method set up process, if retention times shift due to column maintenance, or poor thermal stability it can be helpful to have the ability to collect scan data and dMRM in the same acquisition method. In Figure 7 and Figure 8 a 10 pg on column standard was analyzed using dMRM/Scan collection mode. Figure 7 demonstrates the extracted Scan chromatogram, along with zoomed in on later eluting compounds displaying their signal to noise ratios. The combination of the improved HES 2.0 along with the improved thermal stability of the DB-5Q GC Column make it possible to not only analyze them in selective methods such as dMRM, but also by Scan Mode.

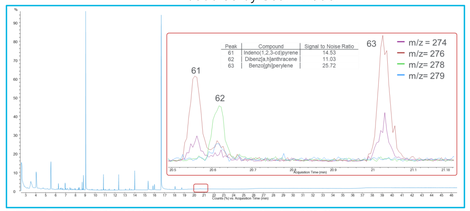

Figure 7. Scan TIC of 10 pg on column standard of 8270 compounds analyzed on a DB-5Q GC column collected in DMRM/Scan Mode.

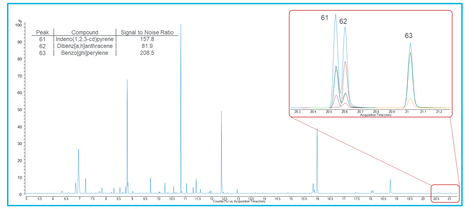

Figure 8. DMRM TIC of 10 pg on column standard of 8270 compounds analyzed on a DB-5Q GC column collected in DMRM/Scan Mode..

Conclusions

- The improved thermal stability of the Agilent J&W DB-5Q GC Column leads to less spectral interference and lower perceived column bleed.
- The DB-5Q has similar selectivity to the DB-5MS UI GC Column, making adoption easier and does not require additional method validation.
- The DB-5Q has superior inertness in comparison even when working with heavy matrix samples, helping to increase robustness.
- The combination of the DB-5Q and the HES 2.0 allow for greater sensitivity of 8270 compounds.

References

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