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CUTTING DOWNTIME, NOT COLUMNS: PERFORMANCE OF A NEXT-GENERATION INERT CAPILLARY COLUMN FOR MULTIRESIDUE GC-MSMS ANALYSIS

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Background

The analysis of pesticide residues in complex botanical matrices is often complicated by high levels of co-extracted starches, oils, water, and other naturally occurring compounds that persist even after extensive sample cleanup. Achieving reliable detection and quantitation of pesticides at trace levels demands a chromatographic column with exceptional inertness, resolution, and long-term stability. We present the evaluation of a novel GC capillary column, engineered for outstanding inertness and robustness in GC-MS/MS multiresidue pesticide analysis. Compared to conventional columns, this new design delivers markedly improved peak shape, signal-to-noise, and resolution across a wide variety of challenging botanical matrices, including ashwagandha root, lavender oil, and ginseng powder. A standout feature is its integrated fused silica retention gap at the inlet and fused silica transfer line at the outlet. This innovative construction ensures that retention times remain consistent, even after trimming, allowing for fast and reliable maintenance with no impact on chromatographic performance.

Now Foods

Established in 1968 by Elwood Richard, Now Foods is a family-owned company, committed to promoting health and wellness offering over 1500 products, and testing for over 400 unique pesticide residues.

Mission: Provide value in products and services that empower people to lead healthier lives.

Pesticide Testing Program at Now Foods: Sample load consists of plant-based extracts, powders, liquids, organic-certified ingredients, and oils.

Instrumentation & Conditions

GC analysis was performed using Thermo Scientific TRACE 1610 GC equipped with a TSQ 9610 Triple Quadrupole Mass Spectrometer. Data acquisition and processing performed using Thermo Scientific Chromeleon Software.

RMX-5Sil MS 30 m, 0.25 mm ID, 0.25 µm, with 5 m Integra-Guard & Integra-Transfer Line, Liner Topaz 4mm ID single taper liner with wool. 1µL splitless injection 0.8 minute hold @ 260°C using helium carrier gas 1.4mL/min (32 cm/sec). Oven program:40 °C (hold 1.5 min) to 90 °C at 40 °C/min (hold 1.5 min) to 180 °C at 40 °C/min to 250 °C at 10 °C/min to 280 °C at 5 °C/min to 320 °C at 10 °C/min (hold 5 min)

Sample Preparation

Initial Preparation
- Homogenize samples as needed.
- Weigh 0.5 g sample into 50 mL centrifuge tube.
- Add 10 mL water and 10 mL acetonitrile with internal standards.

QuEChERS Extraction
- Add QuEChERS salts (AOAC buffered or original unbuffered as appropriate).
- Vortex for 30 minutes.
- Centrifuge at 9000 rpm for 5 minutes.
- Combine 2.5 mL extract with 0.25 mL 1% of a formic acid solution.

Sample Clean Up
- Load entire extract on an appropriate cartridge and allow it to pass through into a collection vessel.
- Transfer 1 mL (for GC) or 0.5 mL (for LC) to a glass test tube.
- Evaporate to ~100 µL, reconstitute with an appropriate solution for GC or LC, and transfer to vial for analysis. For GC samples, reconstitute in toluene and add $MgSO_4$ to remove any residual water.

Analytical Performance

The performance of RMX-5Sil MS columns was compared to a traditional 5sil type column. All columns tested were 30 m x 0.25 mm ID x 0.25 µm. The traditional column was installed with a 10 m integrated guard column to protect the analytical column from matrix contamination. For the RMX-5Sil MS column, two formats were tested: an Integra-Guard column and a dual Integra-Guard and Integra-transfer line column. Both formats are a continuous piece of tubing that houses the 5Sil MS analytical stationary phase as well as uncoated sections of deactivated tubing at the inlet side (guard) and/or detector side (transfer line). These formats were selected because they both provide a high degree of protection from matrix contamination without the need for manual connections that can leak and require downtime for maintenance. The dual format provides an additional benefit because the lack of stationary phase in the Integra-transfer line going to the detector means less bleed at high temperatures, so the MS stays cleaner and signal-to-noise ratios can be improved.

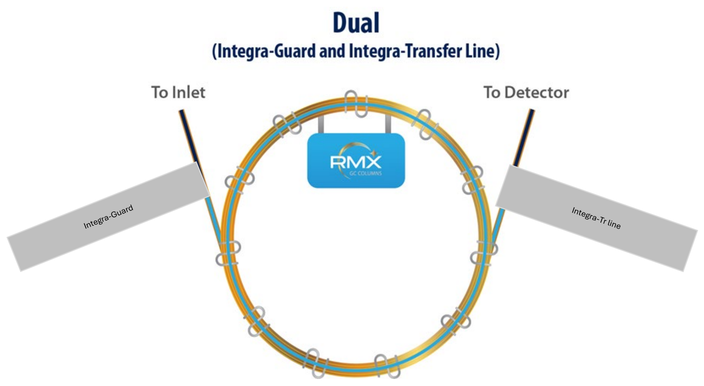

Figure 1: Diagram of the GC-MS/MS system with a 5-meter guard column connected to the analytical column. The RMX-5Sil MS column has an integrated transfer line which is deactivated but not coated with stationary phase which reduces conditioning time and bleed.

Observations

As shown in the chromatographic comparisons, even these sensitive compounds respond well in the presence of difficult matrices on RMX-5Sil MS columns. For example, as seen in Figure 3, metalaxyl and chlorfenapyr could not be properly analyzed on the traditional 5sil column because only one product ion was measurable, whereas SANTE guidelines require two for MS/MS detection. In contrast, on the RMX-5Sil MS column, the highly inert surface produced two or three signals with excellent peak shapes and intensities that allowed for definitive identification and integration.

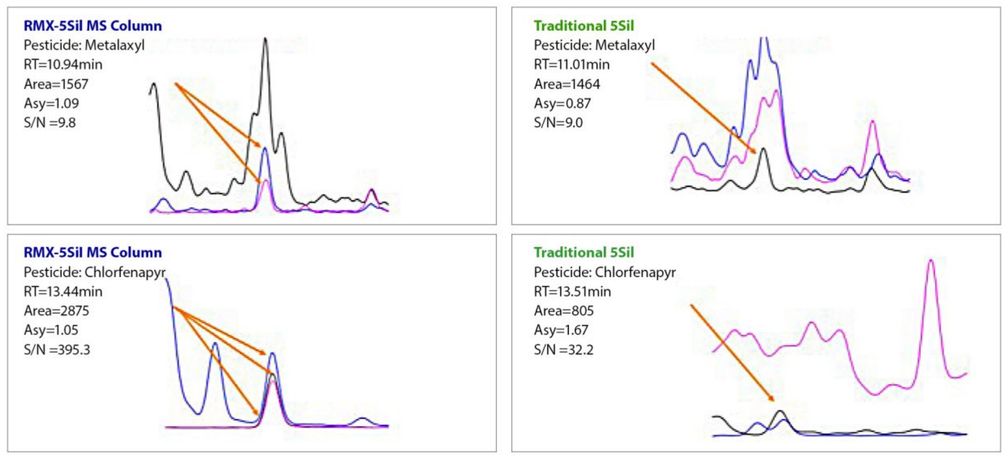

Figure 2: Compared to traditional 5sil columns, highly inert RMX-5Sil MS columns produce clear peaks for multiple ions, allowing metalaxyl and chlorfenapyr (50 ppb) to be accurately analyzed in ashwagandha.

Recovery Evaluation

In addition to comparing the chromatographic performance of RMX-5Sil MS columns to traditional 5sil columns for GC-MS/MS multiresidue pesticides analysis, recovery experiments were also conducted. For this assessment, 35 pesticides representing a range of different chemistries were fortified at 50 ppb in curcumin and cinnamon-honey coated almonds. Curcumin is an extremely complex sample, and the high levels of curcuminoids (phenolic pigments) and volatile oils are known to cause matrix interference, which can compromise reporting accuracy. Overall, acceptable recoveries (70-120%) were obtained on both columns, but the RMX-5Sil MS column delivered better performance with an average percent recovery across all pesticides of 94% vs. 88% in curcumin (Figure 3)

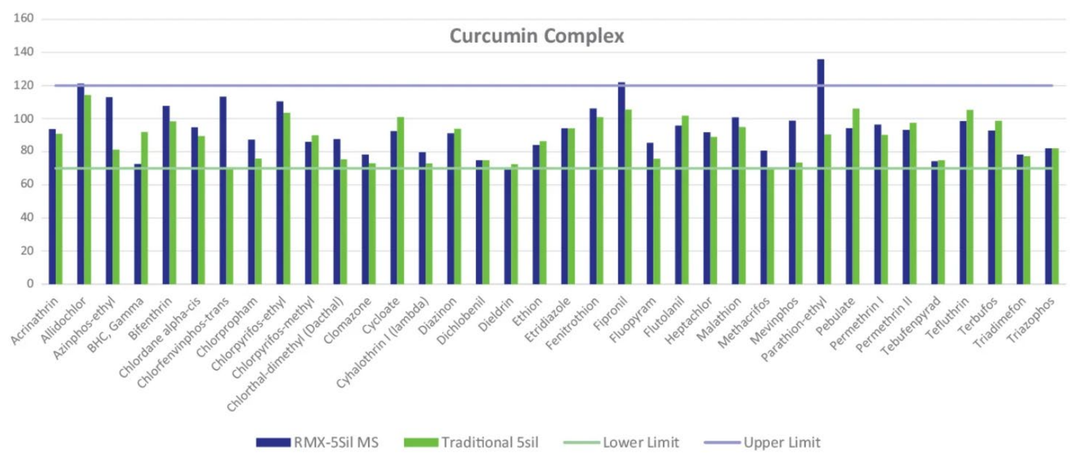

Figure 3: Recovery Data for 35 Pesticides in Curcumin at 50 ppb

Column Lifetime

As shown in Table I, the traditional column required more frequent trims and ultimately failed to meet performance requirements after 1131 injections and 659 hours of use. In contrast, both RMX-5Sil MS column formats needed only two or three trims and met performance requirements for much longer. At the end of the study, both RMX-5Sil MS columns continued to deliver good performance and were still suitable for use after 1800 injections (1050 hours) for the Integra-Guard format and 1995 injections (1164 hours) for the dual Integra-Guard and Integra-transfer line format. It should be noted that this study was conducted during routine sample testing, and results were accumulated for each column during the period of time when it was installed, so the lower numbers for guard-only format reflect the fact it was not installed in the instrument as long. For both formats, the inertness of the RMX-5Sil MS column significantly reduced downtime for maintenance and sample analysis could continue even after running challenging matrices, such as oils, that usually require that the column be trimmed before another matrix can be run.

	RMX-5Sil MS (Integra-Guard Format)	RMX-5Sil MS (Dual Integra-Guard and Integra Transfer Line Format)	Traditional 5sil with Guard
Number of injections	1800	1995	1131
Run time (hours)	1005	1164	659
Number of column trims	2	3	7
Status at end of experiment	still meets performance requirements	still meets performance requirements	end of lifetime

Table I: Lifetime of the RMX-5Sil MS column in both formats exceeded the traditional 5sil column, and more samples could be analyzed with fewer column trims.

Conclusion

This comparative study demonstrates that GC-MS/MS multiresidue pesticides analysis is significantly influenced by the inertness of the chromatographic system, particularly the GC column. Across a broad range of 230 different pesticides and more than 100 challenging botanical matrices, RMX-5Sil MS columns consistently delivered superior chromatographic performance compared to a traditional 5sil column. The enhanced inertness provided by TriMax deactivation technology resulted in improved peak shapes, higher signal-to-noise ratios, and more accurate results at both 10 ppb and 50 ppb.

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