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Low Thermal Mass Technology, a New Approach to Accelerated Gas Chromatography that Provides More Efficient Dual Column Confirmation

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Abstract
Low Thermal Mass (LTM) technology was developed by RVM Scientific and is now offered as a retrofit to existing Agilent 5890 and 6890 GCs by GERSTEL as the Modular Accelerated Column Heater (MACH). The technology involves combining any length standard capillary GC column, a Resistance Temperature Detector (RTD) based thermal measurement system, and a precision controlled heating element in a bundle over the full length of a column. The bundle is wrapped with a ceramic twine and coiled to a 5” diameter torus that is covered with a metal foil. These column modules can then be heated outside the GC oven with heated transfer lines to the GC’s injector and detector going through the oven door and the GC oven. This technology enables fast heating (up to 1800°C per minute), fast cooling (350°C to 35°C in 2 minutes or less), as well as independent temperature control of up to 4 column modules on one GC platform. A mixture of C_5 through C_{44} n-alkanes can be separated in less than 4 minutes. The combination of fast GC capability and independent temperature control of multiple column modules on one GC enables efficient dual column separation. For

example, the separation of 20 standard pesticides can be accomplished in about 3 minutes. This note will review the LTM technology, the MACH hardware, and present data to demonstrate the increased speed of analysis.

Introduction

A number of techniques have been used in the past to achieve faster temperature ramping than that obtained with a standard convection GC oven. These include the use of a 220 Volt powered GC, oven inserts to decrease the volume of the GC oven, oven inserts which can add extra heating capability to the standard oven, and resistively heated tubes which are installed inside the GC oven and used to rapidly heat GC columns [1]. These approaches have suffered from limitations such as slow oven cool down, inability to use a guard column, restriction of the dimensions of the GC column used, and leaks from problematic hardware used to connect the GC column to the specific injector and detector.

Low Thermal Mass (LTM) technology for fast column heating was developed by RVM Scientific Inc. [2] and is now offered as a retrofit to Agilent 5890 and 6890 GCs by GERSTEL as the Modular Accelerated Column Heater (MACH). The practical issues of leaks and column fouling seen in earlier Fast GC designs are addressed by making column connections with standard Agilent and Valco fittings and by using deactivated fused silica capillaries that protect the analytical column. In addition to fast heating and cooling to shorten GC cycle times this technology also enables the configuration of up to 4 columns on the same GC, each with independent temperature control.

The MACH offers advantages for dual column confirmation analyses, since each column can be independently heated and optimized rather than using the same temperature ramp for both columns. In the following overview, the MACH instrumentation is described and data is presented which shows the benefits of its unique design when configured for dual column confirmation analyses.

Experimental

Instrumentation (System-1). Analyses were performed on a GC (6890, Agilent Technologies) equipped with a micro ECD detector, split/splitless injector, and a dual column MACH system (GERSTEL).

Analysis conditions.

Injection:	1 µL, manual
GC Inlet:	split 20:1; 250°C
GC Oven:	275°C, held for duration
MACH Module 1:	10 m Rtx®-CLPesticides1 (Restek), MACH format d_i = 0.18 mm d_f = 0.18 µm He, P_i = 31.1 psi 140°C (0.17 min); 50°C/min; 160°C; 300°C/min; 225°C; 50°C/min; 325°C (1 min)
MACH Module 2:	10 m Rtx®-CLPesticides2 (Restek), MACH format d_i = 0.18 mm d_f = 0.14 µm He, P_i = 29.4 psi 120°C (0.17 min); 300°C/min; 225°C; 50°C/min; 325°C (1 min)

Instrumentation (System-2). Analyses were performed on a GC (6890, Agilent Technologies) equipped with a 5975 MSD, split/splitless injector, MPS 2 Autosampler (GERSTEL), and a single column MACH system (GERSTEL).

Analysis conditions.

Injection:	1 µL, MPS 2
GC Inlet:	split 25:1; 250°C
GC Oven:	280°C, held for duration
MACH Module:	10 m Rtx®-5 (Restek), MACH format d_i = 0.18 mm d_f = 0.20 µm He, P_i = 18.0 psi 30°C (0.08 min); 200°C/min; 330°C
MSD:	scan, 35-350 amu, 19.22 scans/s

Instrument Design

Low Thermal Mass (LTM) technology involves combining any length standard capillary GC column, an RTD based thermal measurement system, and a precision controlled heating element in a bundle over the full length of a column. The bundle is wrapped with a ceramic twine and coiled to a 5” diameter torus that is covered with a metal foil (Figure 1). The average power consumption to heat an LTM assembly at a programmed rate is approximately 1% of the power required to heat a GC oven [2].

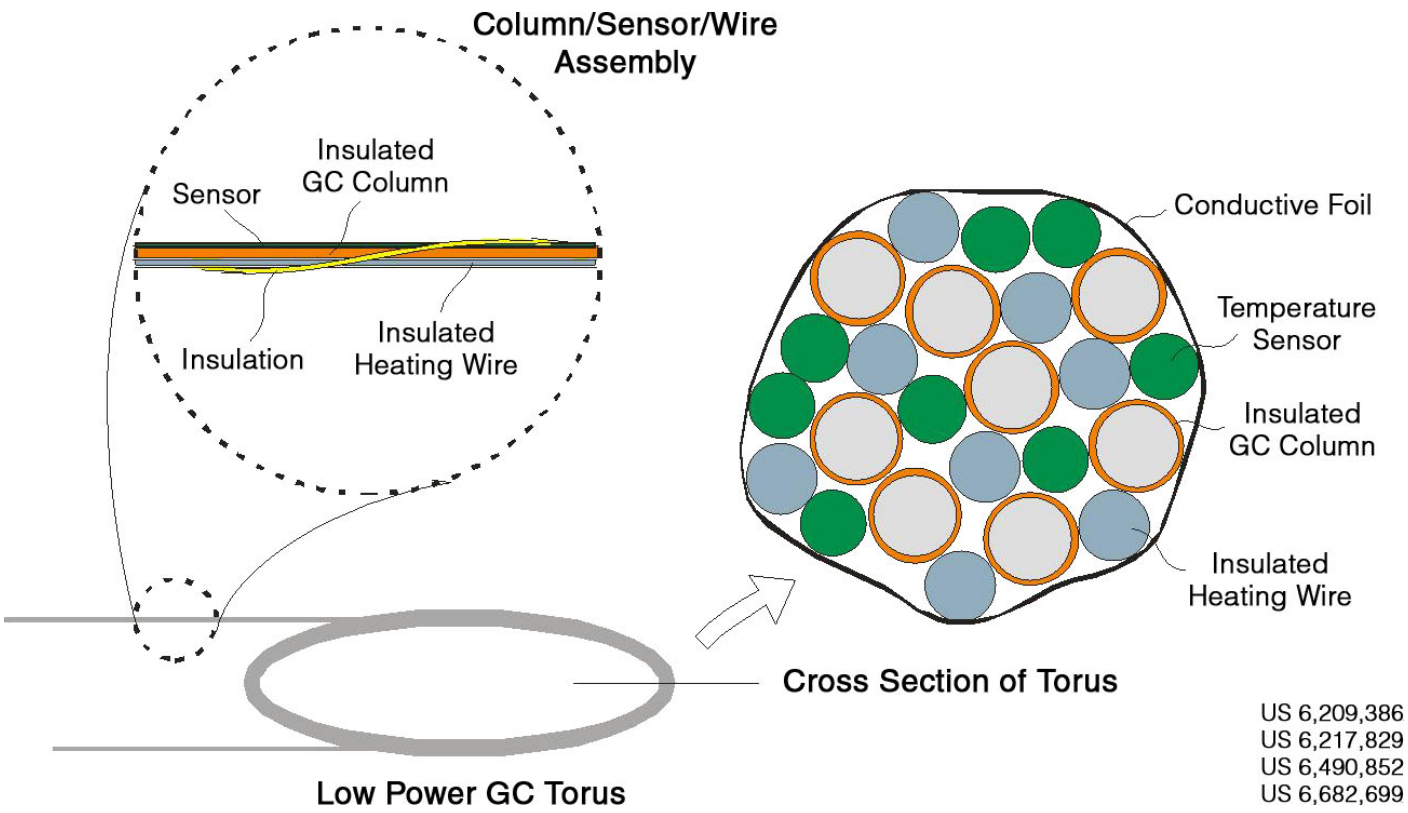

Figure 1. Low thermal mass column module diagram.

The MACH column modules can be independently heated outside the GC oven using heated transfer lines to the GC's injector and detector going through the GC oven door (Figures 2 and 3). Connections to the injector and detector are identical to standard GC column connections. Programming of the temperature ramps can be performed either using a key pad on the front of the MACH door or through GERSTEL software integrated into the Agilent Chemstation control software.

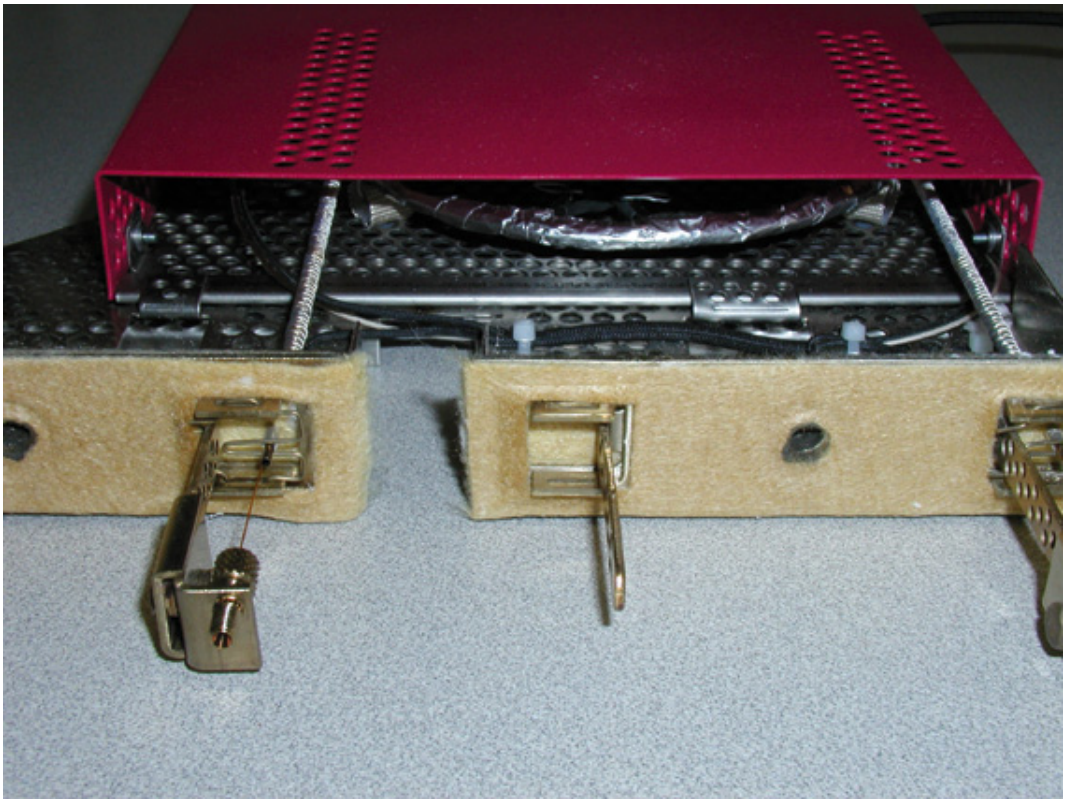

Figure 2. Column module connection to transfer line module.

Figure 3. Dual column MACH connected to 6890 GC system.

Results and Discussion

Figure 4 shows a chromatogram of a DRO standard mix of 16 aliphatic hydrocarbons from Restek (Cat. # 31459) separated under conditions described above for System 2. The entire separation is completed in less than 1.6 minutes, with an injection to injection cycle time of approximately four minutes.

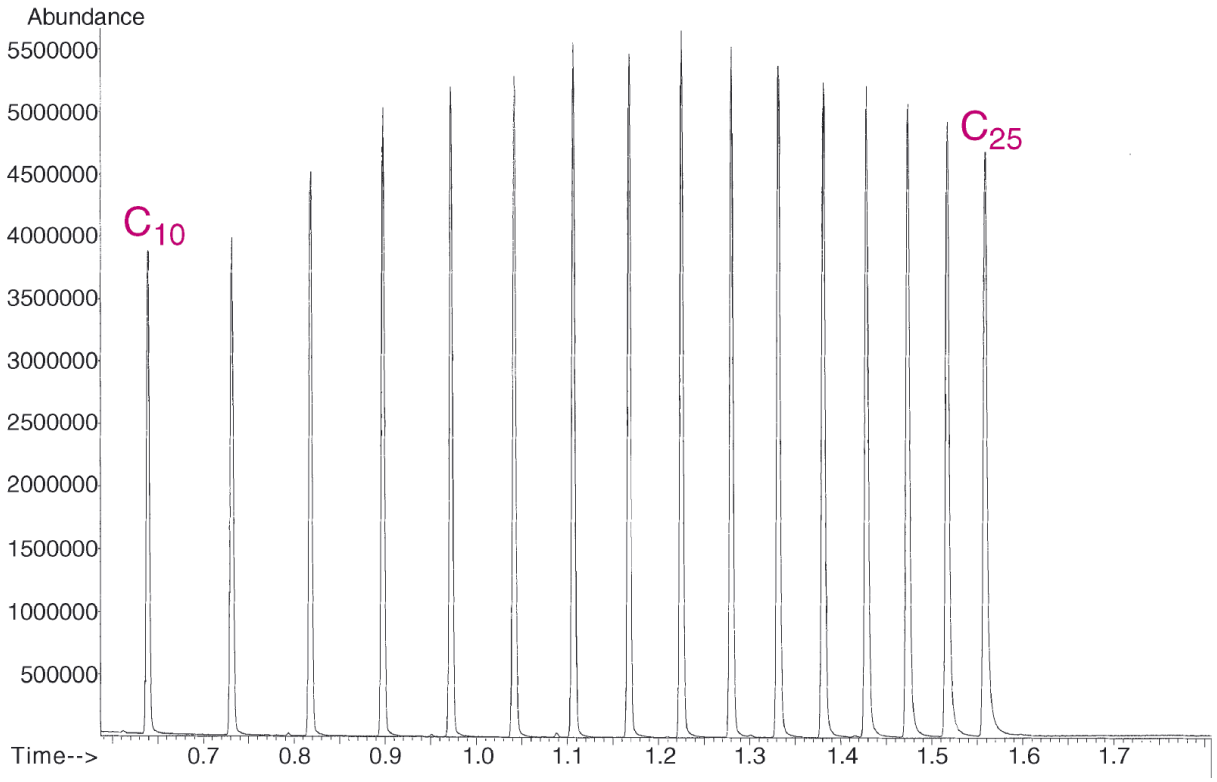

Figure 4. Diesel range organic (DRO) chromatogram.

Figures 5 and 6 show chromatograms of a 20 pesticide standard mix from Restek (Cat. # 32292) analyzed on the MACH system under conditions described above as System 1. Please note that the temperature ramps of the Rtx®-CLPesticides1 and Rtx®-CLPesticides2 columns are different and were optimized independently to provide maximum resolution in under 2 minutes on each column.

1. α-BHC
2. γ-BHC
3. β-BHC
4. δ-BHC
5. Heptachlor
6. Aldrin
7. Heptachlor epoxide
8. trans-Chlordane
9. cis-Chlordane
10. 4,4'-DDE
11. Endosulfan I
12. Dieldrin
13. Endrin
14. 4,4'-DDD
15. Endosulfan II
16. 4,4'-DDT
17. Endrin aldehyde
18. Methoxychlor
19. Endrin sulfate
20. Endrin ketone

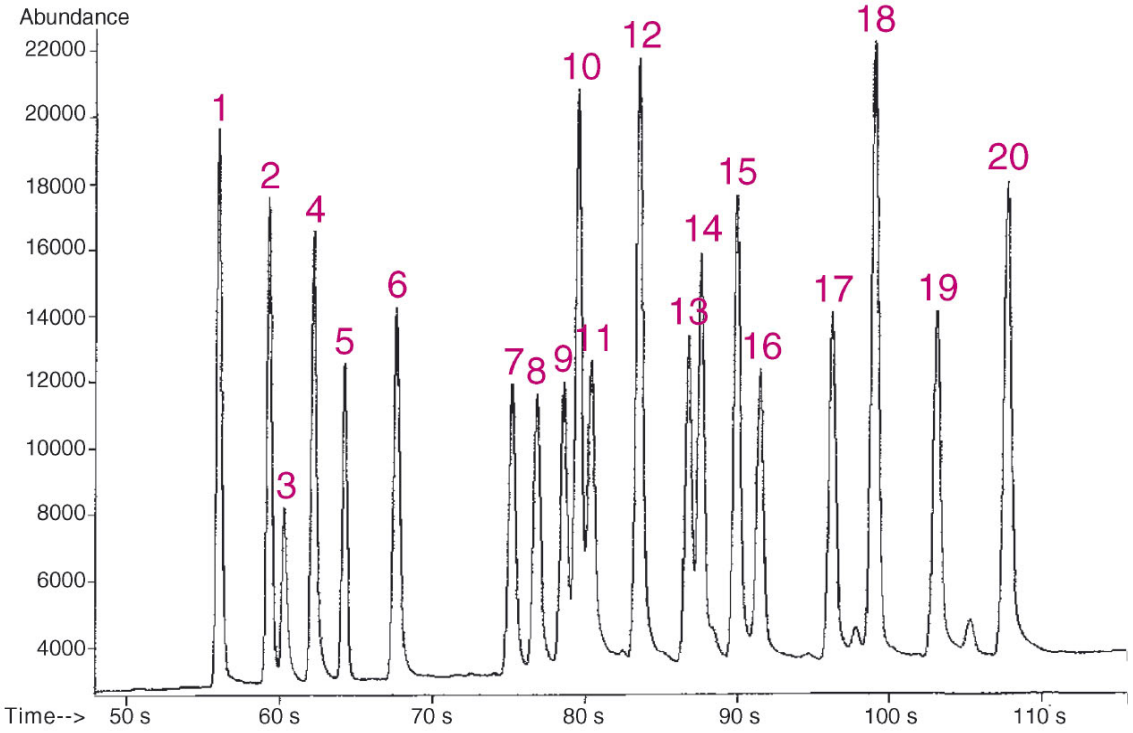

Figure 5. Pesticide separation on Rtx®-CLPesticides1 column.

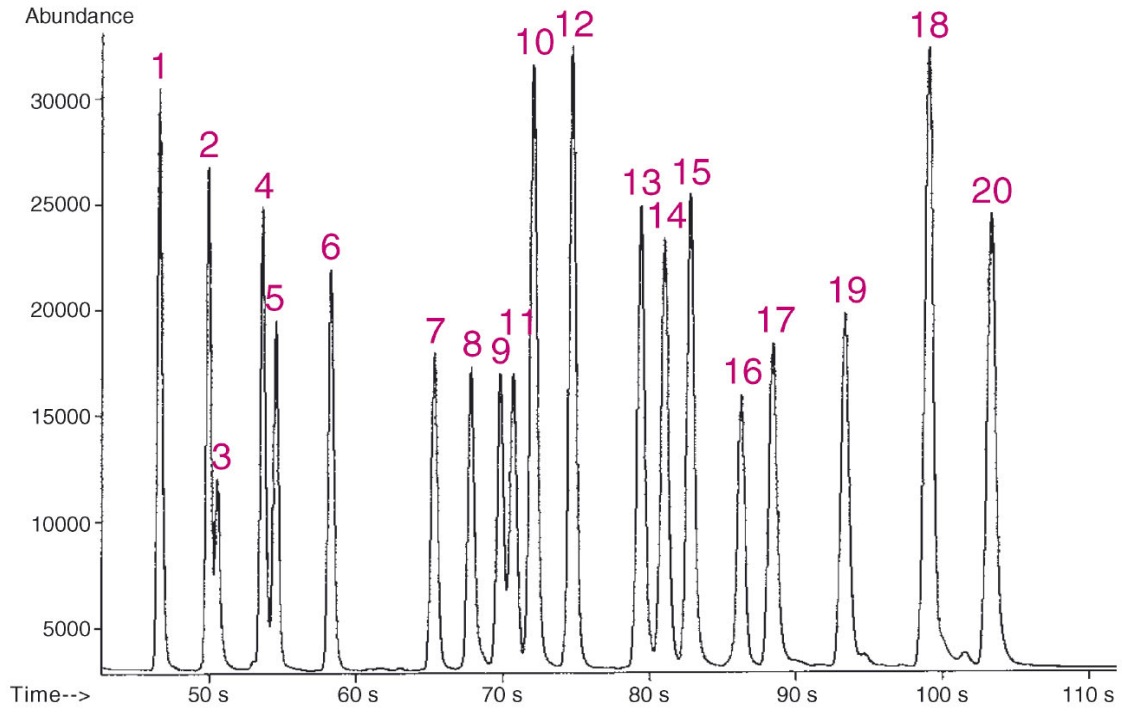

Figure 6. Pesticide separation on Rtx®-CLPesticides2 column.

LTM technology is used commercially for efficient dual column confirmation analysis of PCBs as shown in Figures 7 and 8. PCB standards were run with simultaneous injection onto DB-608 and DB-1701 GC columns which were in modules heated independently with LTM technology.

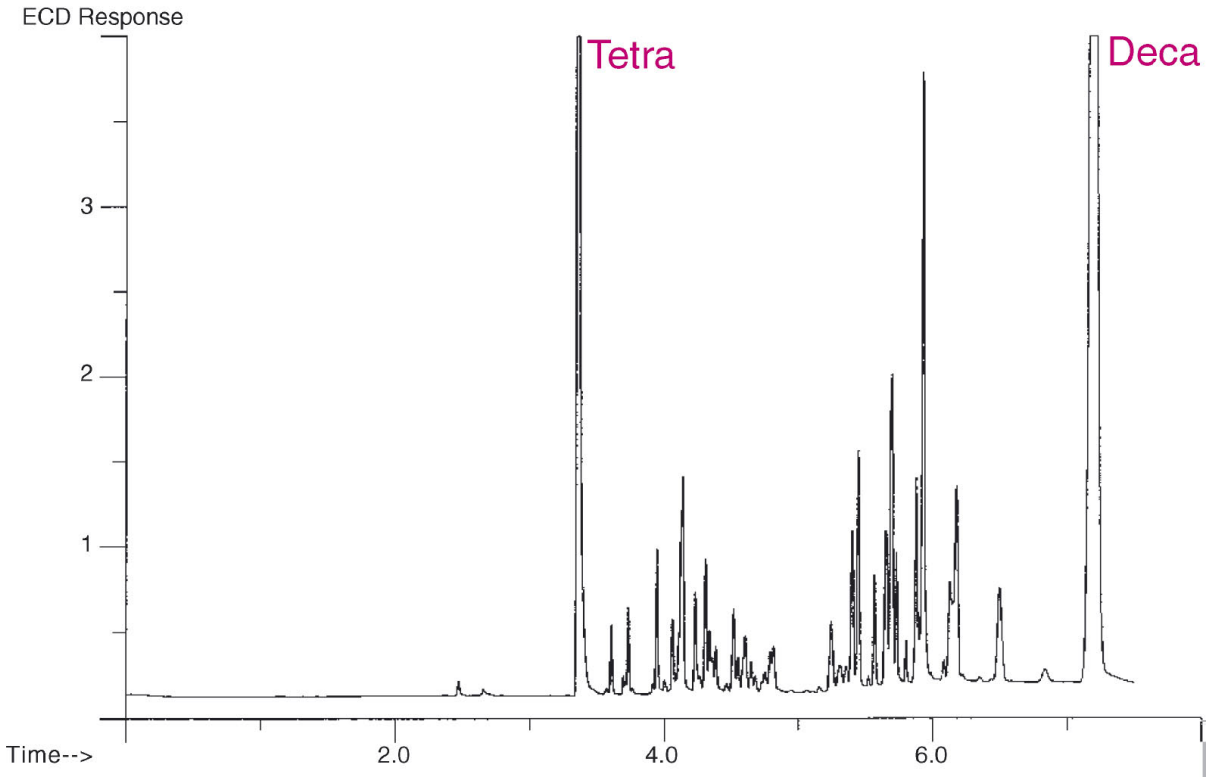

Figure 7. PCB separation on DB-608 column, courtesy of Kevin Maher, SGS Environmental, Anchorage, Alaska.

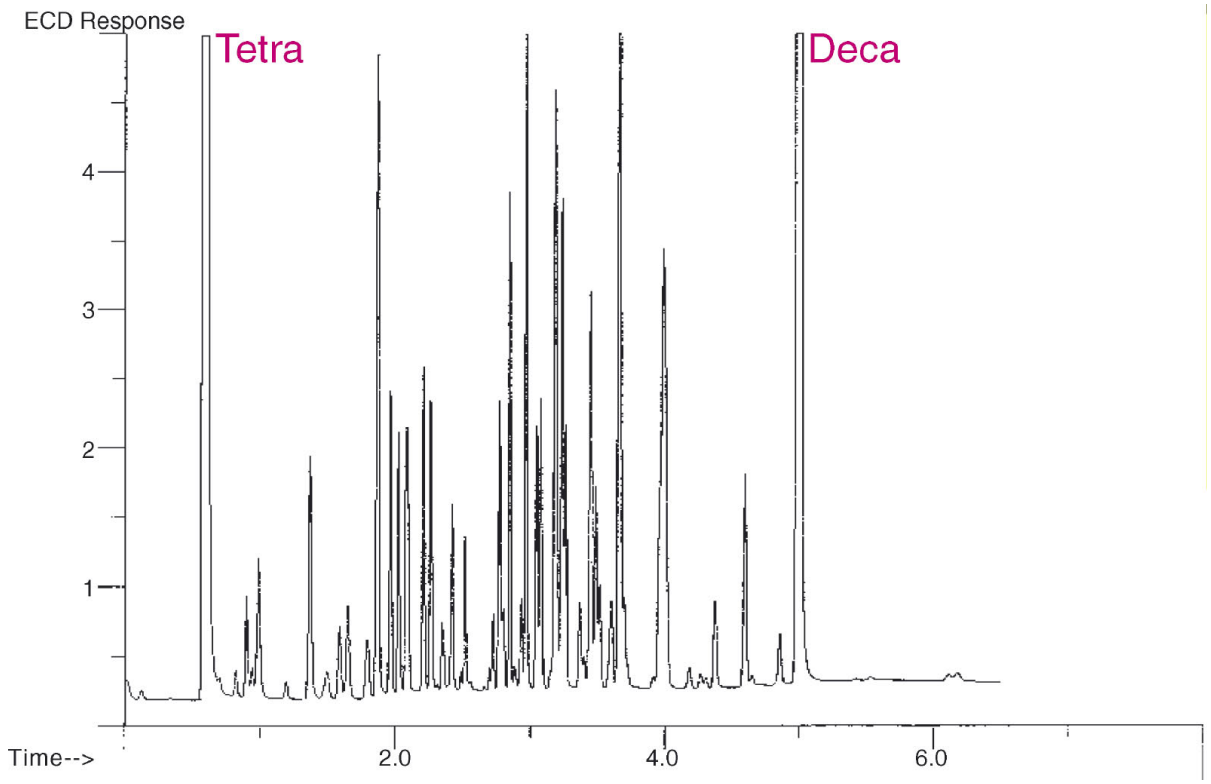

Figure 8. PCB separation on DB-1701 column, courtesy of Kevin Maher, SGS Environmental, Anchorage, Alaska.

Conclusions

In this study LTM technology in GERSTEL MACH modules was used to separate C_{10} through C_{25} diesel range hydrocarbons in less than 1.6 minutes with a cool down time of approximately 2 minutes. With short chromatographic run times, the cool down time becomes very important when trying to minimize the overall injection to injection time. Compared to fast GC methodologies where the GC oven must cool to the starting temperature before another run can begin, LTM technology offers a great advantage in overall sample throughput.

The pesticide separations on the Rtx®-CLPesticides1 and Rtx®-CLPesticides confirmation analysis could be obtained with an injection to injection time of approximately 4 minutes.

The MACH system provides the ability to independently optimize column dimensions and operation for dual column Fast GC methods which should enable extending existing methods to even more complex sample types.

References

[1] Macdonald, SJ, Wheeler D. Fast temperature programming by resistive heating with conventional GCs. Am Lab 1998; 30(22):27-40

[2] Robert Mustacich, James Everson, and John Richards. Fast GC: Thinking outside the box. American Laboratory, March 2003, pp 38-41

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