

A new High Temperature Carbowax Column stable up to 300°C for FAST-GC and GCxGC use.

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Introduction

Carbowax polar stationary phases are the most used in GC separations in conjunction with 5% phenyl apolar columns. For example PEG columns are very often used in Essential Oil, FAMES and Aromatic Compounds analyses. In recent years growth and development of techniques such as Fast-GC and GCxGC showed the need to have polar phases that can reach high temperatures. In fact, using Fast-GC systems with high temperature rates, the elution temperature of the compounds analyzed rises accordingly. In the same way, in the GCxGC, having a second dimension column such polar as a carbowax one that in the meantime allows to reach higher temperatures greatly expands the application possibilities of the technique.

For these reasons MEGA has developed a new Carbowax based column called MEGA-Wax HT, able to reach over 300°C and maintain the temperature of 300°C in isothermal mode. The MEGA-Wax columns have a special deactivation treatment of the tubing surface that ensures a better coating of the phase. Beyond that a particular treatment on the PEG polymer is designed to allow the phase to reach high temperatures with a low bleeding and keeping the performances in time. The temperature range of this new column is extremely wide compared to other Carbowax columns, starting from 40°C reaching up to 300°C.

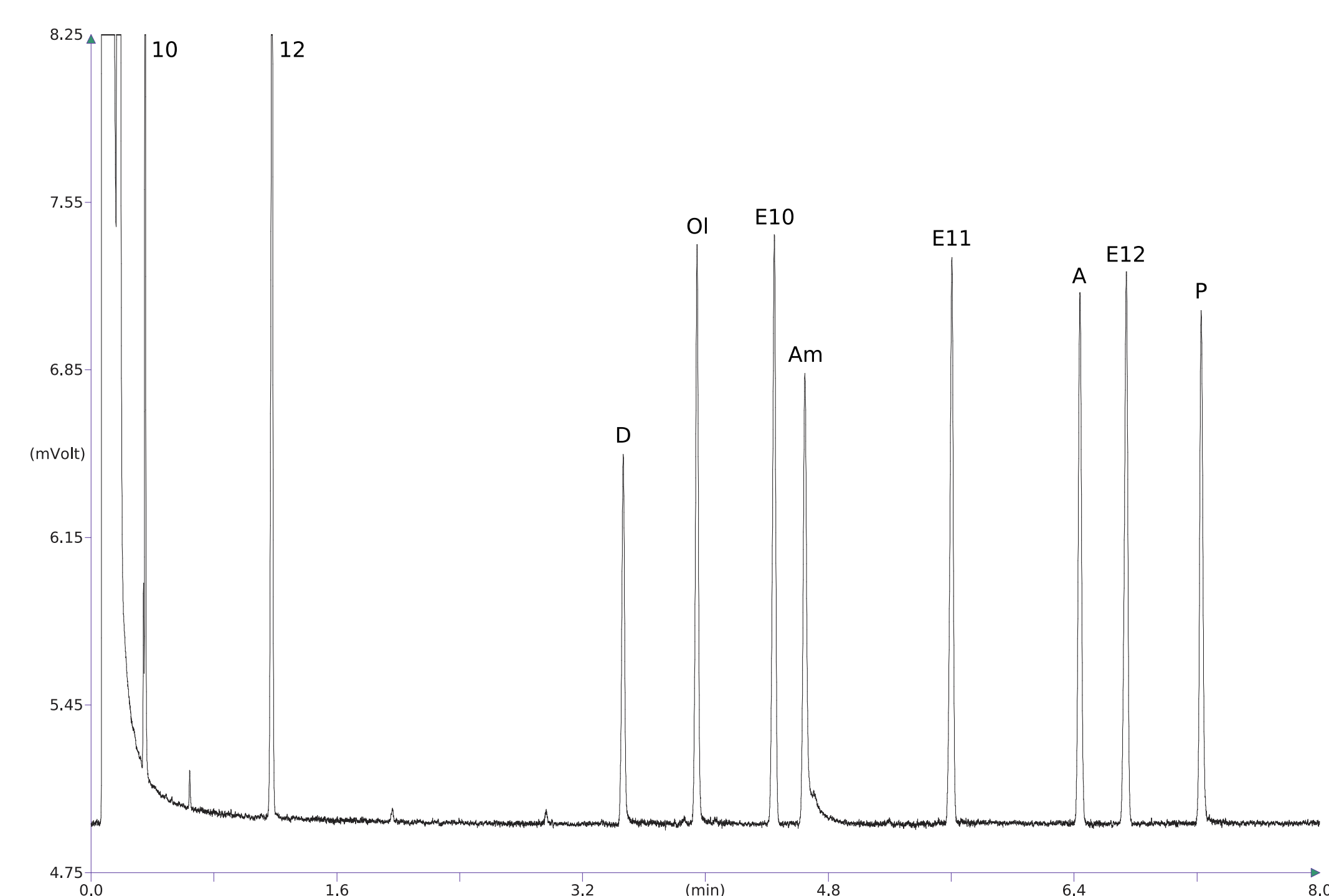


Figure 1. Grob Test (Test Mixture n. 2, Fluka Cat. # 86501) on a 0.10 mm ID x 4.5 m L (0.5 m incorporated retention gap) x 0.10 µm FT, MEGA-Wax HT Fast column. Conditions: 40°C to 200°C @ 15° C/min, Hydrogen carrier gas @ 150 kPa (constant pressure), SSL 250°C, FID 310°C. Identification: decane (10), dodecane (12), 2,3-butanediol (D), 1-octanol (Ol), methyl decanoate (E10), dicyclohexylamine (Am), methyl undecanoate (E11), methyl laurate (E12), 2,6-dimethylaniline, 2,6-dimethylphenol.

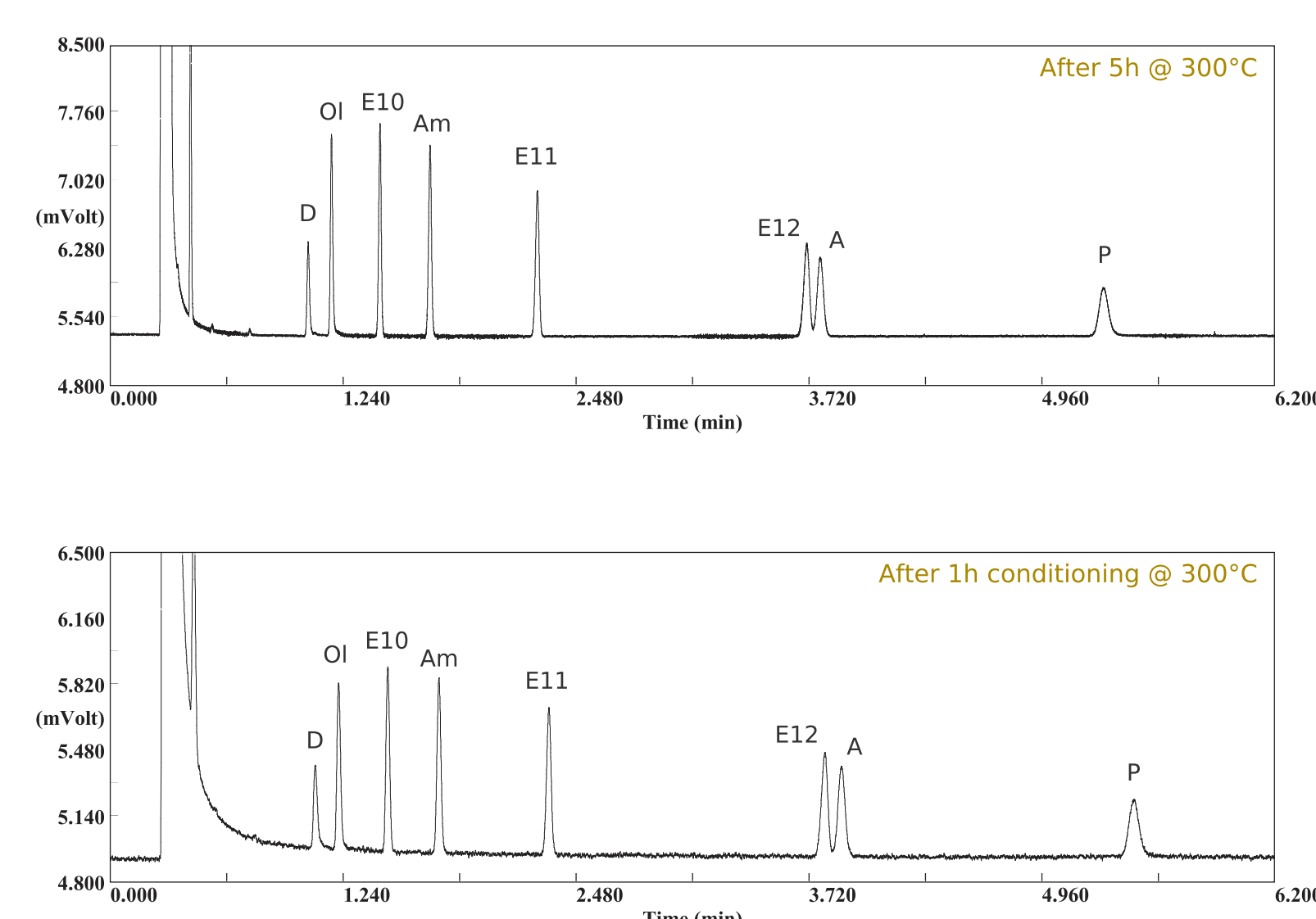


Figure 2.

Two isothermal runs to calculate the theoretical plates number (N) are here shown. All our narrow-bore Fast-GC 100µm ID columns have N/m > 9000, even for this new Carbowax stationary phase. The first value is obtained after one hour of initial column conditioning, while after keeping the column 5h @ 300°C a loss of less 5% than previous N value was noted, thus showing a very good resistance at high temperatures. The N values were calculated at 110°C in isothermal mode on a 0.10 mm ID x 8 m L x 0.10 µm FT, MEGA-Wax HT Fast.

Experimental Results

1. BIODIESEL Analysis on MEGA-Wax HT.

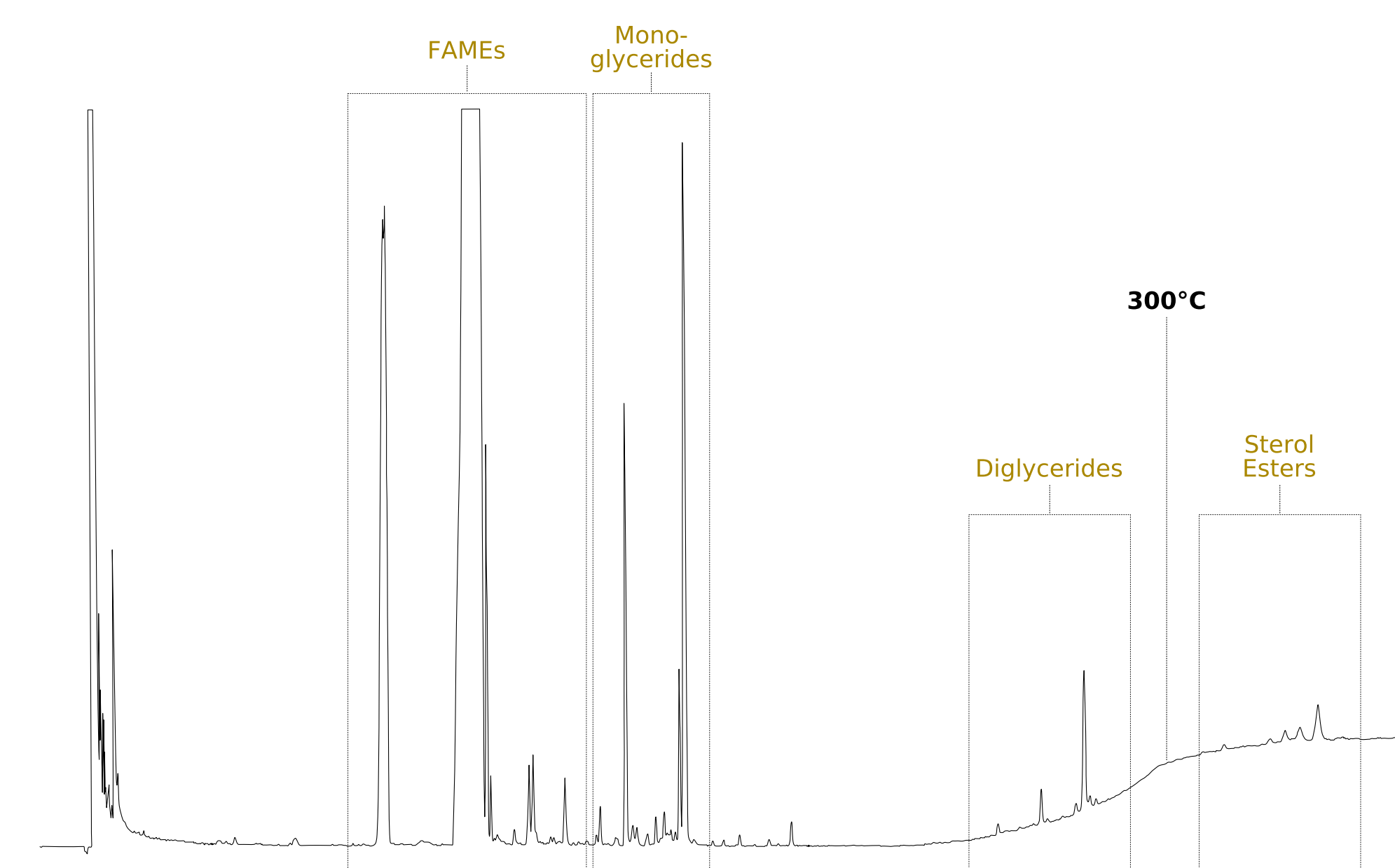


Figure 3. A real Biodiesel sample analysis is shown above. The Biodiesel sample was prepared according to the EN-14105 procedure. The column used is a 0.32 mm ID x 10 m L (1 m incorporated retention gap) x 0.05 µm FT, MEGA-Wax HT column. Conditions: 60°C to 300°C @ 10°C/min, Hydrogen carrier gas @ 35 kPa (constant pressure), On-Column Injection.

Biodiesel presents a significant challenge from the analytical point of view. There are many implemented methods regarding the GC Biodiesel analysis to determine free and total Glycerine, ester and linoleic acid methyl esters and Mono, Di and Tri-glycerides content. We injected a Biodiesel real sample on the new MEGA-Wax HT column. The results in Figure 3 show the possibility to extend the use of a polar Carbowax phase also for this kind of high temperature analysis. The MEGA-Wax HT works very well at high temperature, resolving sterol esters at 300°C in isothermal mode, while keeping an high polarity necessary to solve a typical FAMES separation that is made on a Carbowax column according to the EN-14103 method (see the Figure 4 below). The aim of this example is to demonstrate the possible use of the MEGA-Wax HT for high temperature Gas-Chromatography. The column could of course be applied to the GCxGC technique in order to have a second dimension column that allows to maintain an high orthogonality while reaching high temperatures needed for these applications.

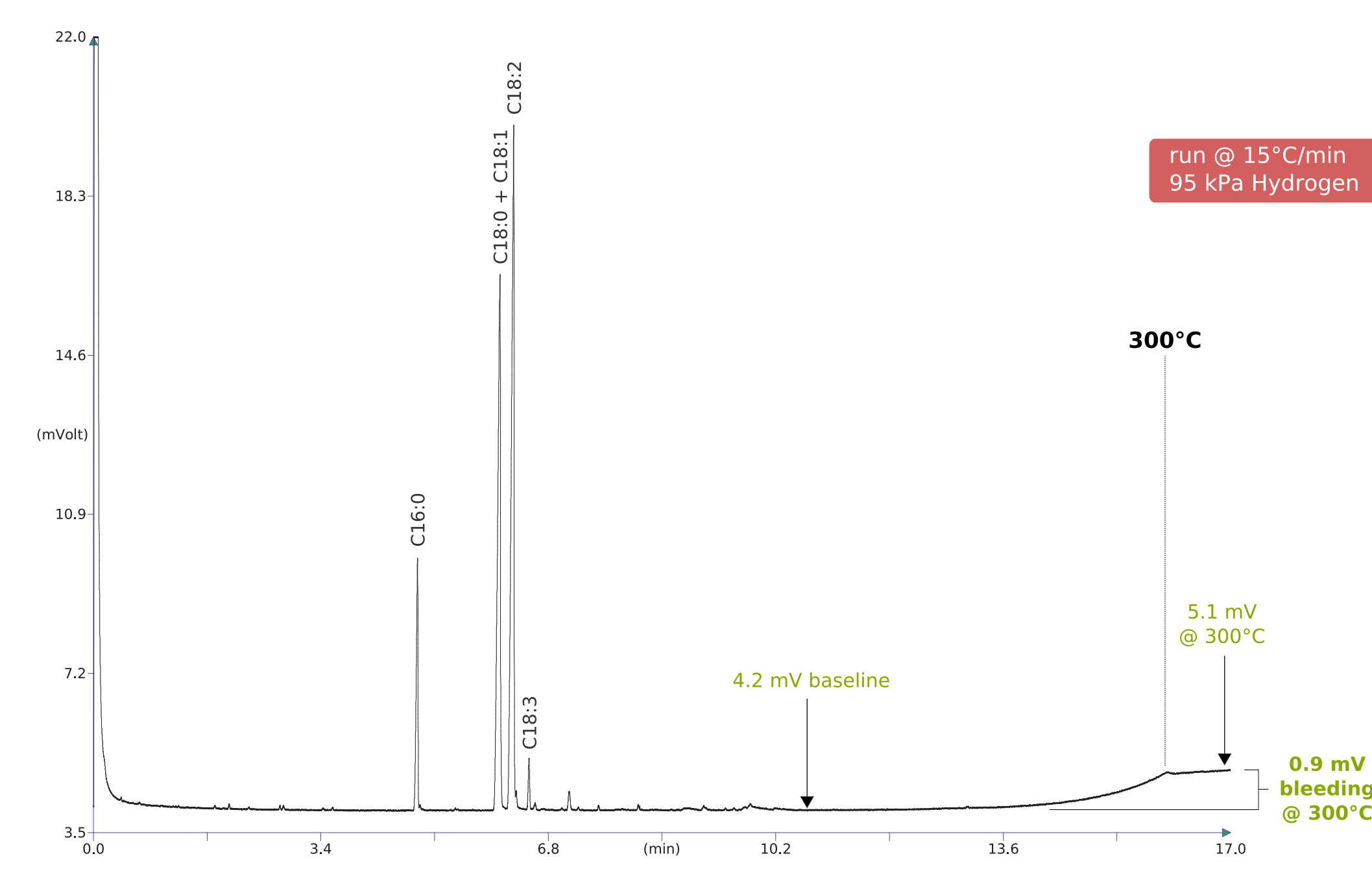


Figure 4. A FAST-GC separation example of Biodiesel FAMES performed on 0.10 mm ID x 2.5 m L (0.5 m incorporated retention gap) x 0.10 µm FT, MEGA-Wax HT Fast column. The extreme low bleeding is here also displayed: 0.9 mV bleeding only even under "aggressive" run conditions (15°C/min and 95 kPa Hydrogen constant pressure).

2. POLYWAX 500 Analysis on MEGA-Wax HT.

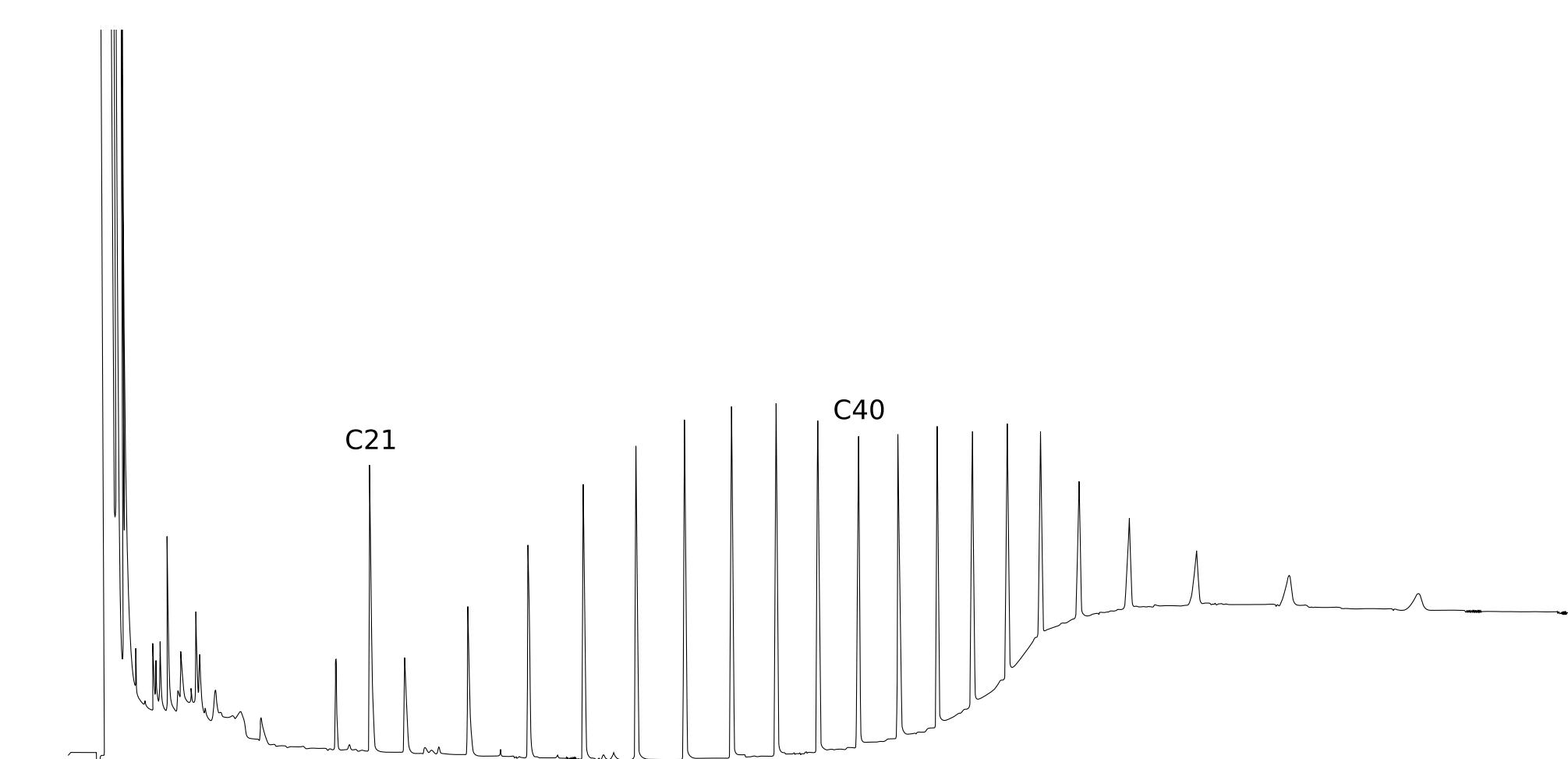


Figure 5. A Polywax 500 analysis on a 0.32 mm ID x 10 m L (1 m incorporated retention gap) x 0.05 µm FT, MEGA-Wax HT column. Another example of the use of the new Wax HT stationary phase for high temperature works. The sample introduction was made in On-Column mode.

Conclusions

The new MEGA-Wax column HT represents a novelty in the panorama of high polarity phases Carbowax. We think that the examples show the suitability of this new phase to meet the needs of having polar phases with an extended temperature range, especially to meet the modern needs of FAST-GC and GCxGC. At the present time in the Comprehensive 2DGC you are forced to find the best compromise between temperature limit of the column system and orthogonality between the two dimensions because of temperature limits given by more polar (and used) phases like Carbowax. We can move these limits a bit higher with the new Wax-HT column. Furthermore, this column may be well used for conventional GC e.g. in combination with an apolar phase for a classic double-column configuration for MS systems. These systems, very often used in F&F labs for example, currently have big limitations because the usable temperature range of the apolar columns (like 5% phenyl phase) is reduced by the temperature limit of the normal Carbowax based phases.

We are still performing applications on this new stationary phase to find the maximum effective operating limits in temperature programmed mode with the best compromise of column lifetime. We are going on with the investigation on the possibility to use the Wax-HT for triglycerides analysis with the advantage to elute these heavy compounds below their decomposition temperature.

You can see other performances of the new MEGA-Wax HT columns on the "LC-GC Europe" (February 2010 Issue) magazine, GC Connections section. Here applications of MEGA-Wax HT are shown both on Fast-GC and GCxGC use for Essential Oil analysis. We wish to thank the authors of this paper for their work and their cooperation.

[Click here and read the LCGC article about the new MEGA-Wax HT column.](#)

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