Pro ezGC® and "Fast GC" Techniques

If you want to investigate "Fast GC" techniques, Pro ezGC can help you determine the columns and hardware you will need to reach your goals. Without changing your column or GC, Pro ezGC can normally reduce your analysis times by 15% to 50%.

AII presented the following poster at PITTCON'97.

Practical Fast GC Analyses

John Garrett, Joseph Solch, Daniel Wagel,Garrett VanNess and Thomas O. Tiernan

Analytical Innovations, Inc.,

Abstract

Significant reductions in analysis times (>90%), can be achieved for a wide variety of analyses using Fast GC. The modeling program, Pro ezGC, can be used to predict practical solutions for a specific analysis. The program calculates temperature programs, flow rates and column dimensions for resolving critical analytes and meeting peak width and analysis time requirements. For more complex analyses, the program can model dual columns and coupled columns.

The maximum temperature programming rates for some newer GCs have increased to 300 C/sec while reset times have decreased. This allows for fast analyses of mixtures that contain a wide boiling point range. When using very fast programming rates, the injection technique and detector volume are the main constraints for good results as they directly affect the peak width of the target analytes. With good injection techniques and well designed detectors, most GCs can achieve a 50% reduction in analysis time for complex mixtures on 15m or longer columns.

Raising the column temperature to shorten the retention time of late eluting components increases the column bleed. Pressure programming provides options for decreasing the analysis time without increasing the column bleed. Modeling both the pressure program and temperature program together often yields the best solution.

Short columns (<5m) or coupled columns are often the answer when there is a small targeted set of analytes. Fast programming rates in these instances are beneficial if there is a wide boiling point range of targeted compounds. Fast programming rapidly raises the column to its maximum temperature so strongly retained compounds are quickly eluted. In practice, a more conventional 10m or 15m column operated at a higher linear velocity can give results that are similar to the results obtained with a 5m column. When separations are optimized using a modeling program, conventional columns, short columns, and coupled columns are easily compared. Modeling is especially useful when evaluating the column length, diameter and film thickness with different temperature and pressure programs.

Modeling can help you take full advantage of your existing GC and columns. This often yields an acceptable result for a specific analysis without any additional expenditure of time and money. Modeling is also a practical way to explore Fast GC techniques and can aid in justifying the purchase of new GC hardware or columns.

Introduction

Significant reductions in analysis times (>90%) can be accurately predicted by a computer modeling program (Pro ezGC®). Using thermodynamic properties from a database, the program calculates the best combination of temperature program, carrier gas flow rate and column dimensions to resolve the critical analytes in the least amount of time.

The program can model a single column, dual columns or serially coupled columns in conjunction with uncoated precolumns and postcolumns. Additionally, the program supports the use of six levels of temperature and pressure programming.

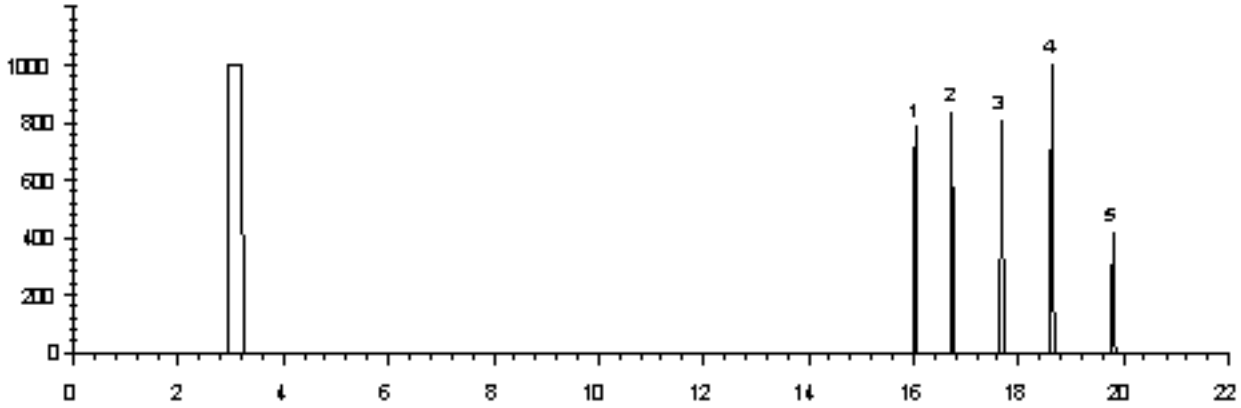
First Example

Fast GC often works best for a small number of compounds or for a larger number of compounds that are very similar in nature. This example will focus on the analysis of five barbiturates.

The first chromatogram shows an unoptimized separation. This chromatogram is similar to Alltech Associates, Inc. Chromatogram #1044 on an EC-5 column.

Barbiturates

5% phenyl / 95% methyl, 30 m x 0.25 mm x 0.25 µm
100°C (3) @ 10°C/min to 260°C (3), Inlet Pressure : 60.2 kPa



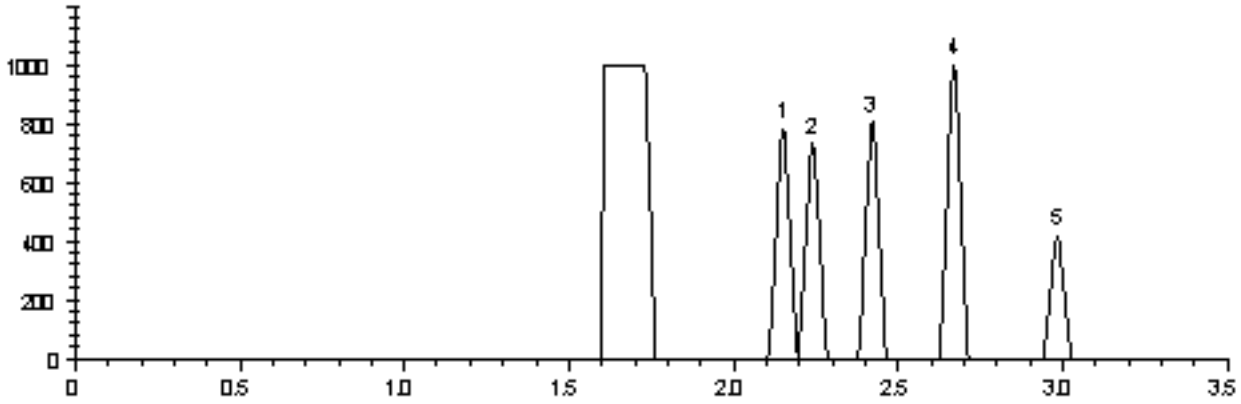
Run Time : 19.79 min	Min Rs Pair : 7.45
Carrier : Helium	Regulation : Constant Pressure
Flow : 0.650 ml/min	Temperature : 50°C
Dead Time : 2.685 min	Linear Velocity : 18.62 cm/sec

# Component Name	Retention Time (min)	Peak Width (min)	Resolution
1. Butabarbital	16.026	0.0701	7.45
2. Amobarbital	16.726	0.0696	7.45
3. Secobarbital	17.677	0.0704	11.57
4. Hexobarbital	18.633	0.0723	11.57
5. Phenobarbital	19.792	0.0769	13.00

The second chromatogram shows the same column optimized for the shortest run times while maintaining resolution of all the analytes. This optimized Normal-GC separation represents an 85% reduction in the run time compared to the original barbiturates separation.

Barbiturates - Optimized Normal-GC Separation

5% phenyl / 95% methy, 30 m x 0.25 mm x 0.25 µm
234°C (1) @ 30°C/min to 260°C (2), Inlet Pressure : 141.5 kPa

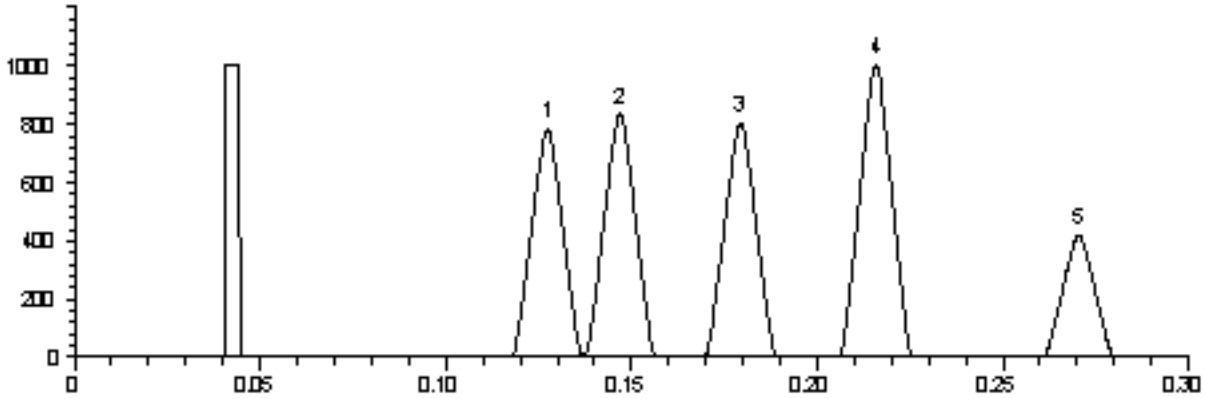


Run Time : 2.98 min	Min Rs Pair : 1.04
Carrier : Helium	Regulation : Constant Pressure
Flow : 0.943 ml/min	Temperature : 234°C
Dead Time : 1.603 min	Linear Velocity : 31.20 cm/sec

# Component Name	Retention Time (min)	Peak Width (min)	Resolution
1. Butabarbital	2.149	0.0874	1.04
2. Amobarbital	2.239	0.0865	1.04
3. Secobarbital	2.420	0.0857	2.10
4. Hexobarbital	2.669	0.0856	2.90
5. Phenobarbital	2.983	0.0853	3.68

The third chromatogram shows an optimized run that uses a short column, high linear velocities and fast ramp temperature program. This Fast GC separation represents a 90% reduction in run time from the Optimized Normal-GC separation.

Barbiturates - Fast GC Separation
5% phenyl / 95% methy, 3 m x 0.25 mm x 0.25 µm
180°C @120°C/min to 300°C Inlet Pressure : 49.7 kPa



Run Time : 0.27 min	Min Rs Pair : 1.09
Carrier : Helium	Regulation : Constant Pressure
Flow : 2.93 ml/min	Temperature : 180°C
Dead Time : 0.0406 min	Linear Velocity : 123.11 cm/sec

# Component Name	Retention Time (sec)	Peak Width (sec)	Resolution
1. Butabarbital	7.63	1.080	1.09
2. Amobarbital	8.81	1.087	1.09
3. Secobarbital	10.76	1.094	1.79
4. Hexobarbital	12.95	1.104	1.99
5. Phenobarbital	16.23	1.092	2.97

Second Example

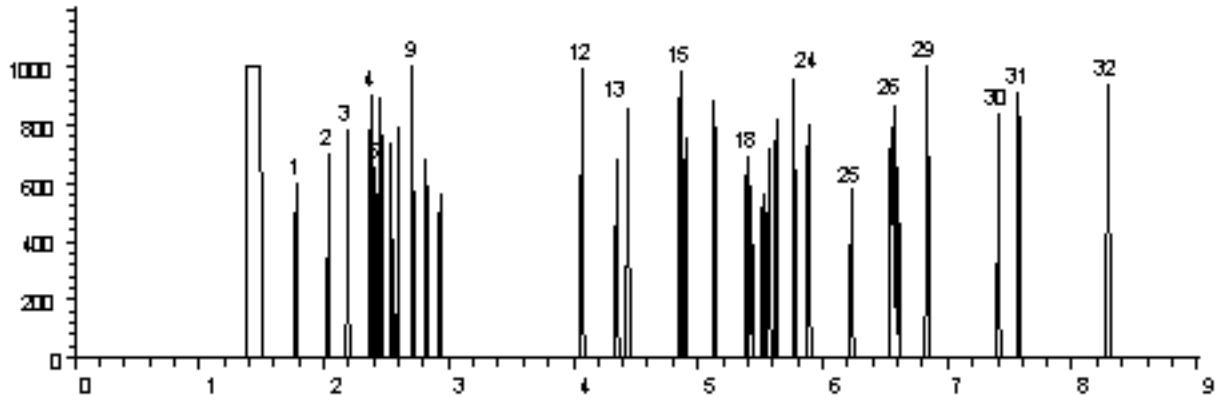
Often the first step in optimizing a GC analysis is to obtain adequate resolution of the target analytes. Once that is achieved, then further optimization of the analysis may proceed.

This Food and Flavors (F&F) example uses 32 compounds that were analyzed on two separate bonded phase columns from Restek Corporation, Stabilwax® (Crossbond® Carbowax®) and Rtx®-1 (crossbonded polydimethylsiloxane). The following two chromatograms show the fastest run times that resolved the most components on each column.

F&F - Stabilwax

Stabilwax, 30 m x 0.25 mm x 0.25 µm

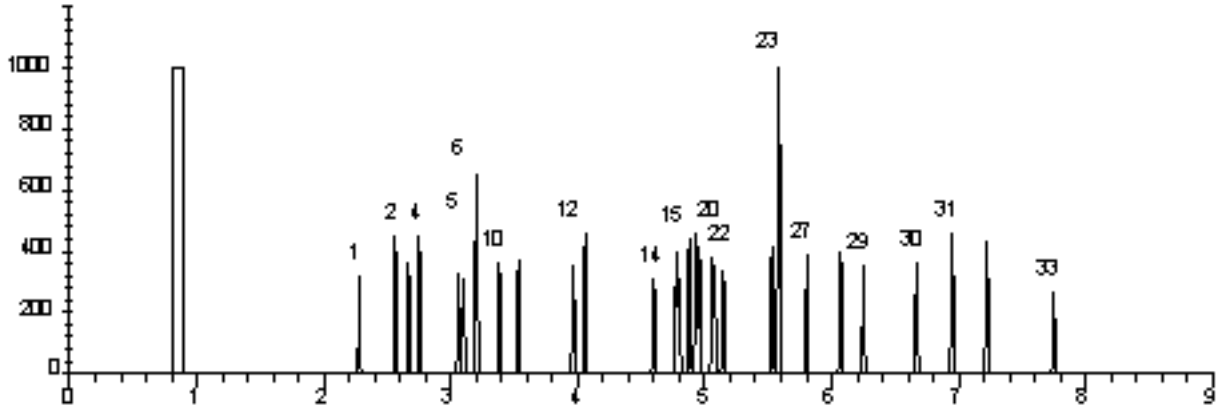
80°C @ 12°C/min to 100°C @ 14°C/min to 200°C, Inlet Pressure : 128.8 kPa



Run Time : 8.28 min	Min Rs Pair : 1.01
Separations : 28 of 32 resolved	
Carrier : Helium	Regulation : Constant Pressure
Flow : 1.51 ml/min	Temperature : 80°C
Dead Time : 1.375 min	Linear Velocity : 36.35 cm/sec

F&F - Rtx-1

Rtx-1 30 m x 0.25 mm x 0.25 µm
70°C @ 9°C/min to 100°C @ 14°C/min to 200°C, Inlet Pressure : 261.8 kPa

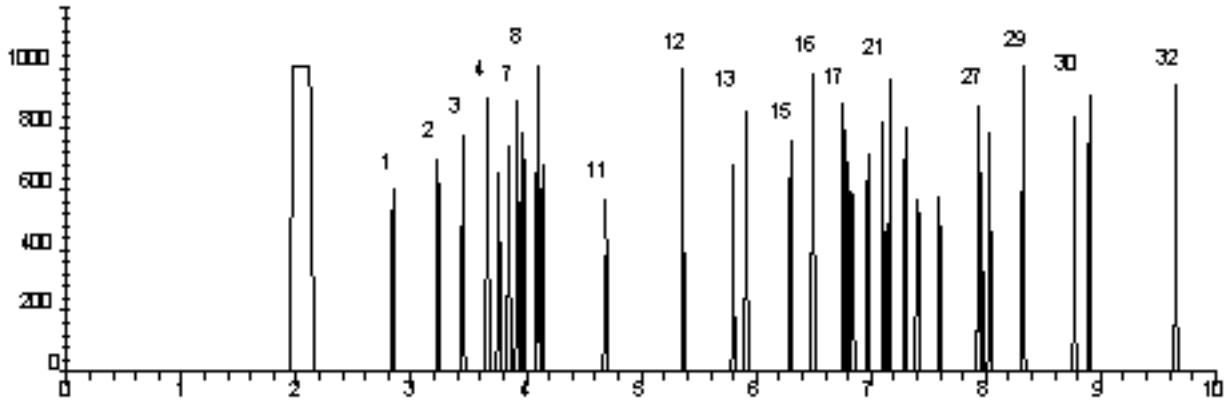


Run Time : 7.75 min	Min Rs Pair : 1.23
Separations : 21 of 33 resolved	
Carrier : Helium	Regulation : Constant Pressure
Flow : 2.85 ml/min	Temperature : 70°C
Dead Time : 0.838 min	Linear Velocity : 59.66 cm/sec

Neither the Rtx-1 nor the Stabilwax column was able to resolve all the components. The following chromatogram shows that a serially coupled column composed of the Rtx-1 and the Stabilwax resolved all the components.

F&F - Serially Coupled Column

Stabilwax, 27m x 0.25 mm x 0.25 µm and Rtx-1, 12 m x 0.25 mm x 0.25 µm
55°C @ 14°C/min to 200°C (1), Inlet Pressure : 170.5 kPa

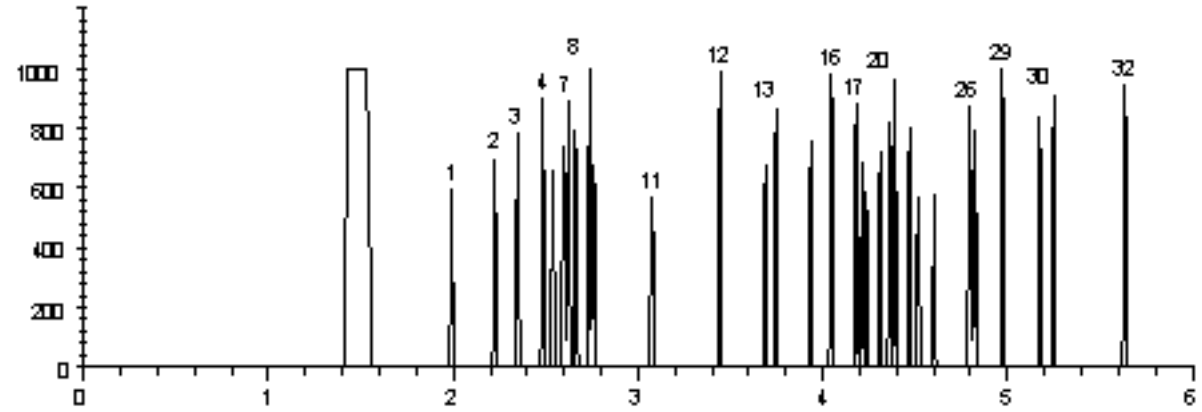


Run Time : 9.65 min	Min Rs Pair : 1.01
Separations : 32 of 32 resolved	
Precolumn : 5 m x 0.53 mm	
Carrier : Helium	Regulation : Constant Pressure
Flow : 1.86 ml/min	Temperature : 65°C
Dead Time : 1.736 min	Linear Velocity : 37.44 cm/sec

The previous chromatogram shows the fastest run time that resolved all of the components. The run time was reduced by 40% on the serially coupled column when a non-critical pair of components was left unresolved.

F&F - Serially Coupled Column

StabilWax, 19 m x 0.25 mm x 0.25 µm and Rtx-1, 8.50 m x 0.25 mm x 0.25 µm
60°C @ 18°C/min to 85°C @ 30°C/min to 200°C, Inlet Pressure : 160.3 kPa



Run Time : 5.63 min	Min Rs Pair : 1.00

Separations : 30 of 32 resolved

Precolumn : 5 m x 0.53 mm

Carrier : Helium | Regulation : Constant Pressure

Flow : 2.45 ml/min | Temperature : 60°C

Dead Time : 1.393 min | Linear Velocity : 38.90 cm/sec

Third Example

In this example, the unique thermodynamic properties of compounds on a specific phase are used to model widely different column lengths, column diameter and column film thickness.

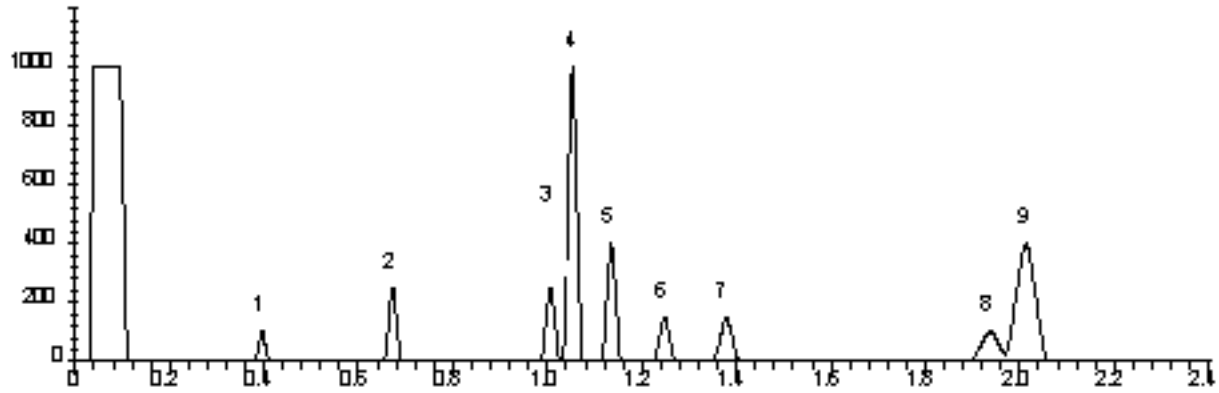
The FAMEs data for this example was originally collected using a 60 meter, 0.25 mm, 0.25 µ Stabilwax column. The thermodynamic properties calculated from this data were used to model a multicapillary column consisting of more than 900 separate 1 m x 0.04 mm x 0.02µm columns.

The following chromatogram shows the predicted elution profile for the multicapillary column that was calculated from the original data. This predicted chromatogram is in very close agreement with Alltech Chromatogram #2140 on MC-WAX.

FAMEs

Carbowax, 1 m x 0.040 mm x 0.200 µm

180°C (0.20) @ 40°C/min to 210°C, Pressure Program : 260kPa(0.4) @ 75kPa/min to 360kPa



Run Time : 2.01 min | Min Rs Pair : 0.87

Carrier : Helium | Regulation : Pressure Program

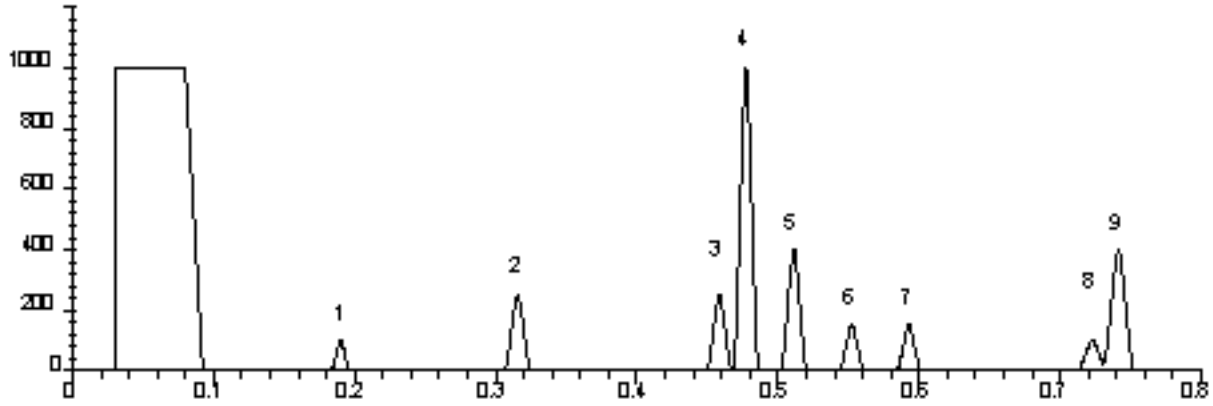
#	Component Name	Retention Time (min)	Peak Width (min)	Resolution
1.	me myristate	0.395	0.0289	9.37
2.	me palmitate	0.672	0.0352	9.37
3.	me stearate	1.005	0.0382	1.23
4.	me oleate	1.052	0.0384	1.23
5.	me linoleate	1.135	0.0389	2.14
6.	me linolenate	1.247	0.0433	2.76
7.	me arachidate	1.378	0.0515	2.84
8.	me behenate	1.936	0.0816	0.87
9.	me erucate	2.012	0.0868	0.87

A further reduction in run time could be achieved by using faster temperature and pressure programs.

FAMEs

Carbowax, 1 m x 0.040 mm x 0.200 µm

200°C (0.20) @130°C/min to 250°C, Pressure Program : 320kPa @ 140kPa/min to 380kPa



Run Time : 0.74 min

Min Rs Pair : 0.90

Carrier : Helium

Regulation : Pressure Program

#	Component Name	Retention Time (sec)	Peak Width (sec)	Resolution
1.	me myristate	11.40	0.741	9.92
2.	me palmitate	18.91	1.053	8.17
3.	me stearate	27.49	1.001	1.13
4.	me oleate	28.62	0.994	1.13

5. me linoleate	30.66	0.989	2.06
6. me linolenate	33.13	0.980	2.49
7. me arachidate	35.56	0.959	2.49
8. me behenate	43.37	1.174	0.90
9. me erucate	44.46	1.246	0.90

Conclusion

Modeling and optimizing your present GC and capillary columns can provide improved run times without any additional expenditure of time and money.

In addition, modeling provides a practical way to explore the following features of Fast GC .

- Pressure programming
- Extremely fast temperature ramps (300 °C/sec)
- Microbore detector systems
- New capillary columns
- Short columns and narrow bore columns

You can use modeling to aid in justifying the purchase of new GC hardware or columns.