

## HIGH TEMPERATURE STATIONARY PHASE FOR CAPILLARY GAS CHROMATOGRAPHY

### INTRODUCTION

HT5 is the first carborane modified siloxane phase to be commercially available on fused silica capillary columns. With an equivalent polarity to 5% phenyl, HT5 is a bonded phase capable of operating at temperatures up to 480°C (with exterior coated aluminum columns); a temperature well above the maximum limits of many modern gas chromatographs.

For many classes of high

molecular weight materials, gas chromatography using high temperature columns, is a more desirable technique than alternatives such as HPLC and SFC. HT5 equips the analyst with a robust column, providing higher resolution, shorter analysis time and ease of operation.

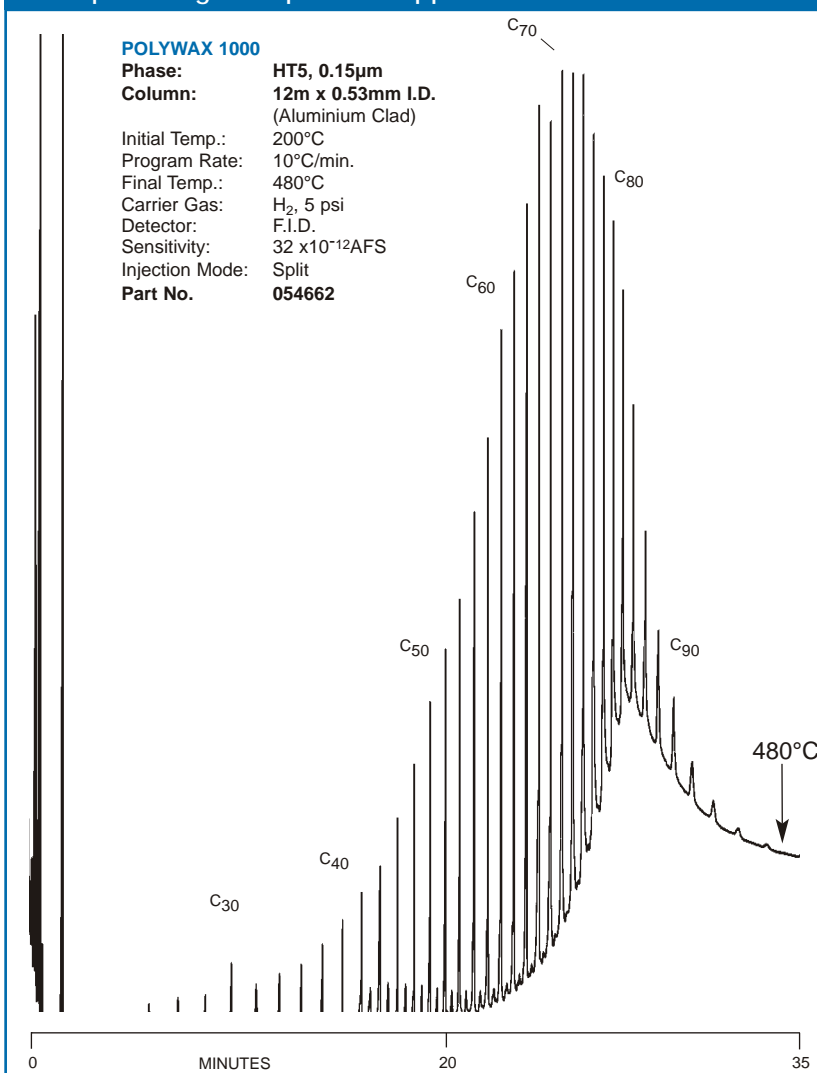
Clearly, the performance limitations of high temperature gas chromatography will be the thermal stability of the compounds to be analysed, and

their possible interaction with the capillary column. Well suited to this technique are many high molecular weight polar and functionalised compounds.

Although HT5 was designed with high temperature analysis in mind, the column is also well suited for operation at more conventional temperatures (280°C/300°C). HT5 effectively exhibits zero bleed at these temperatures which makes it perfectly suited for use with mass spectrometers and other specific detector systems where column bleed is critical. The siloxane-carborane backbone of the HT5 stationary phase, which gives HT5 its outstanding thermal stability, also provides some unusual, and often very useful, selectivity characteristics.

A quick, simple and reliable chromatographic technique is now available for the analysis of high molecular weight waxes and polyethylenes. Due to the excellent resolving power of the HT5 column and a temperature operating range of 10°C to 480°C, the analysis of hydrocarbons from C4 to C100 and above can be achieved in a relatively short analysis time.

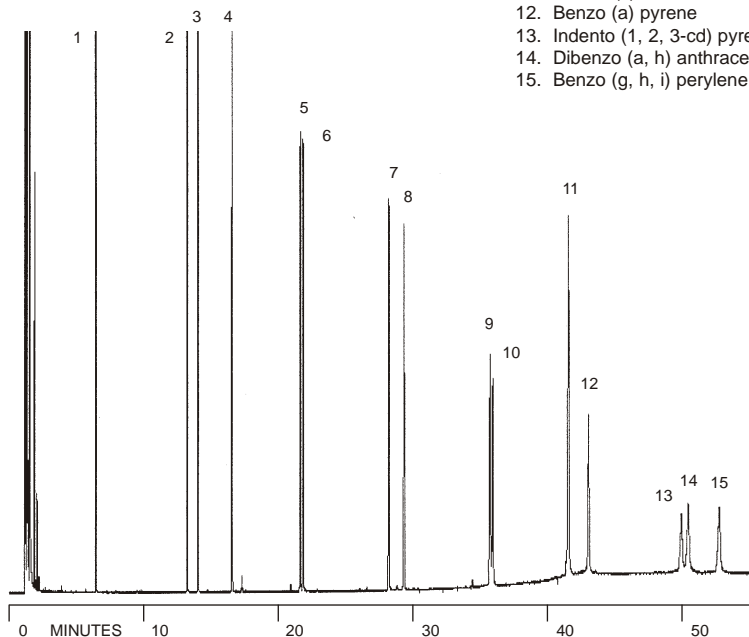
### Example of High Temperature Application



## Figure 1 - Polynuclear Aromatic Hydrocarbons

**Phase:** HT5, 0.1 $\mu$ m  
**Column:** 25m x 0.22mm I.D.  
 (Polyimide Clad)  
 Initial Temp.: 80°C  
 Program Rate: 5°C/min.  
 Final Temp.: 290°C, 10 min  
 Carrier Gas: H<sub>2</sub>, 10 psi  
 Detector: F.I.D.  
 Sensitivity: 16 x 10<sup>-12</sup>AFS  
 Injection Mode: Split  
**Part No.** 054632

1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Flouranthene
8. Pyrene
9. Benzo (a) anthracene
10. Chrysene
11. Benzo (b) flouranthene & Benzo (k) flouranthene
12. Benzo (a) pyrene
13. Indento (1, 2, 3-cd) pyrene
14. Dibenzo (a, h) anthracene
15. Benzo (g, h, i) perylene



## SPECIFICATIONS

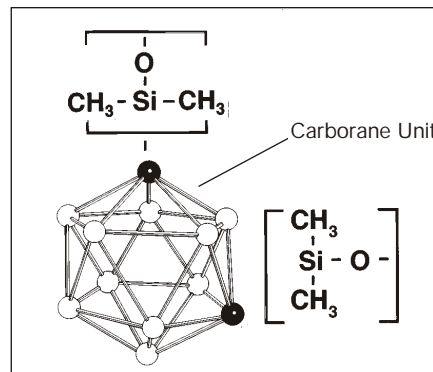
HT5 is a carborane modified polysiloxane with an equivalent polarity of 5% phenyl.

Min. Operating Temp.= 10°C

Max. Continuous Temp.= 460°C\*

Max. Cycling Temp.= 480°C\*

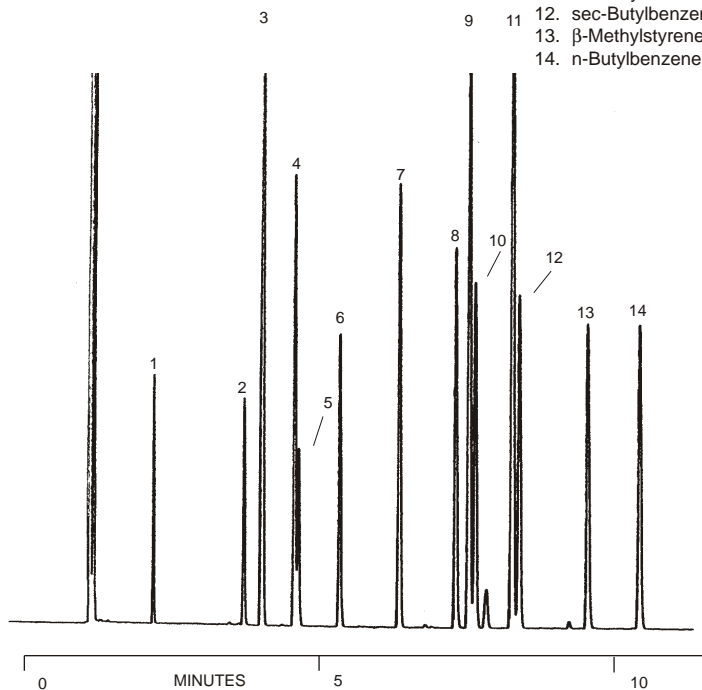
\* aluminum clad column



## Figure 2 - Substituted Benzenes

**Phase:** HT5, 0.1 $\mu$ m  
**Column:** 25m x 0.32mm I.D.  
 (Polyimide Clad)  
 Initial Temp.: 40°C, 2 min  
 Program Rate: 4°C/min.  
 Final Temp.: 70°C, 4 min  
 Carrier Gas: H<sub>2</sub>, 6 psi  
 Detector: F.I.D.  
 Sensitivity: 32 x 10<sup>-12</sup>AFS  
 Injection Mode: Split  
**Part No.** 054652

1. Toluene
2. Ethylbenzene
3. m & p-Xylene
4. Styrene
5. o-Xylene
6. iso-Propylbenzene
7. n-Propylbenzene
8. 1, 3, 5-Trimethylbenzene
9.  $\alpha$ -Methylstyrene
10. tert-Butylbenzene
11. 1, 2, 4-trimethylbenzene & iso-butylbenzene
12. sec-Butylbenzene
13.  $\beta$ -Methylstyrene
14. n-Butylbenzene

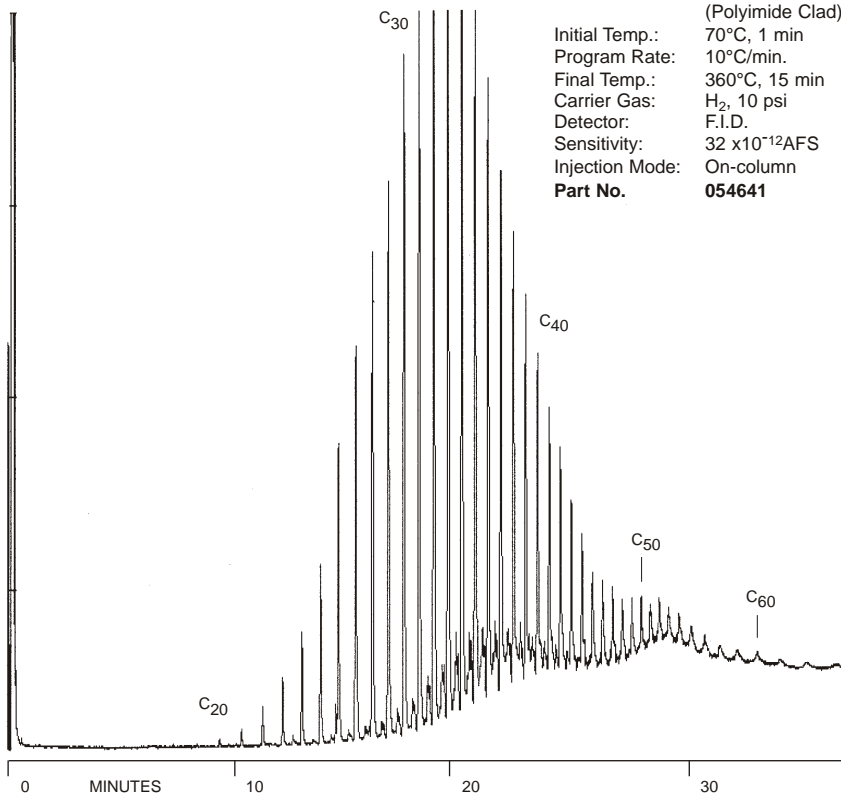


The analysis of 16 PAH (Polynuclear Aromatic Hydrocarbons) illustrates the compatibility of the HT5 column for this application (figure 1). Excellent separation of the isomer pairs phenanthrene/anthracene and benzo(a)anthracene/chrysene is achieved while maintaining a constant stable baseline.

HT5 can also be used to separate complex mixtures of volatile alkyl substituted aromatics (figure 2).

Figure 3 - Wax Sample

Phase: HT5, 0.1µm  
Column: 12m x 0.32mm I.D. (Polyimide Clad)  
Initial Temp.: 70°C, 1 min  
Program Rate: 10°C/min.  
Final Temp.: 360°C, 15 min  
Carrier Gas: H<sub>2</sub>, 10 psi  
Detector: F.I.D.  
Sensitivity: 32 x10<sup>-12</sup>AFS  
Injection Mode: On-column  
Part No. 054641



These two chromatograms illustrate the range of high and low molecular weight waxes that can be analysed on polyimide (figure 3) and aluminium coated (figure 4) HT5 columns.

Though restricted to a maximum temperature of 360°C-370°C, the polyimide coated capillaries still allow a considerable range of hydrocarbons to be analysed. In the above example straight chain hydrocarbons up to C60 are analysed without difficulty.

Figure 4 - Crude Oil and Wax Mixture

Phase: HT5, 0.1µm  
Column: 12m x 0.22mm I.D. (Aluminum Clad)  
Initial Temp.: 35°C  
Program Rate: 10°C/min.  
Final Temp.: 480°C  
Carrier Gas: H<sub>2</sub>, 15 psi  
Detector: F.I.D.  
Sensitivity: 32 x10<sup>-12</sup>AFS  
Injection Mode: Split  
Part No. 054631

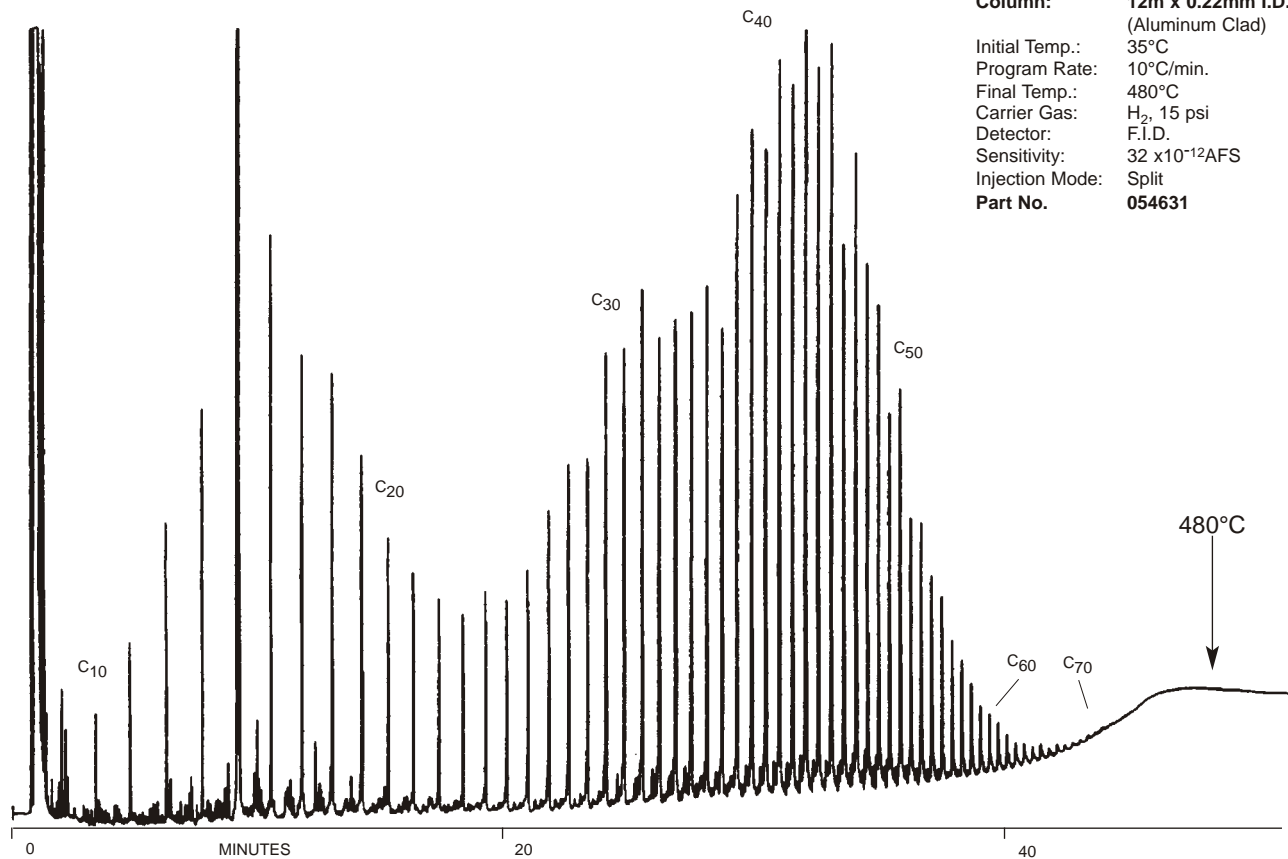


Figure 5 - Porphyrins

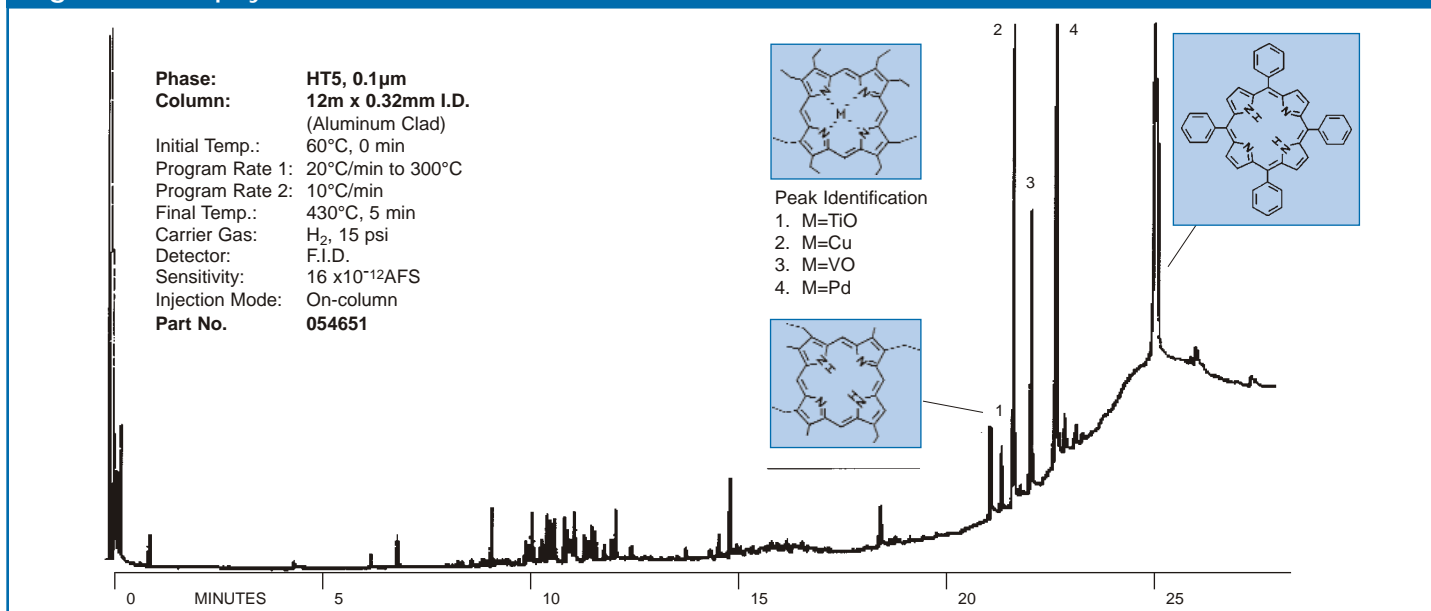
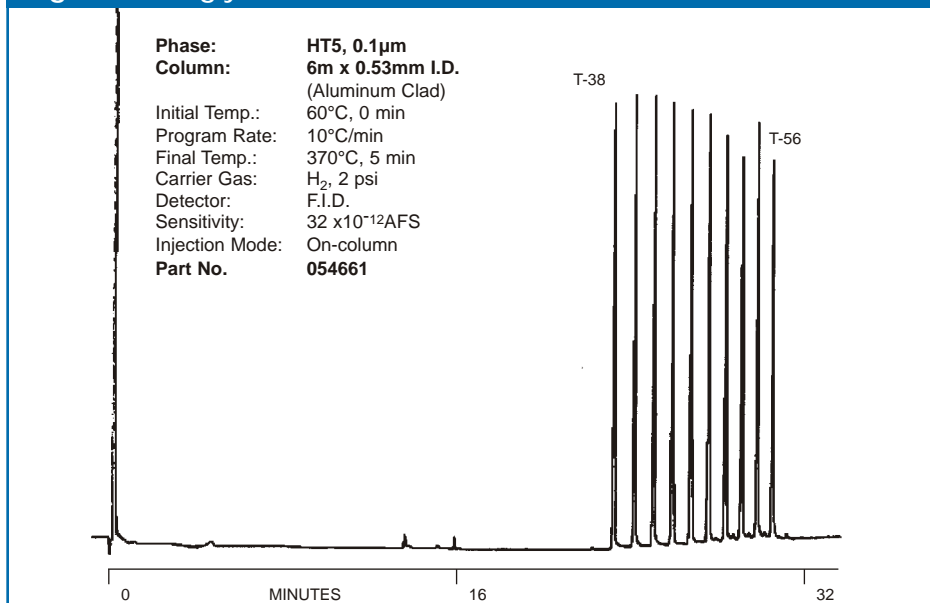
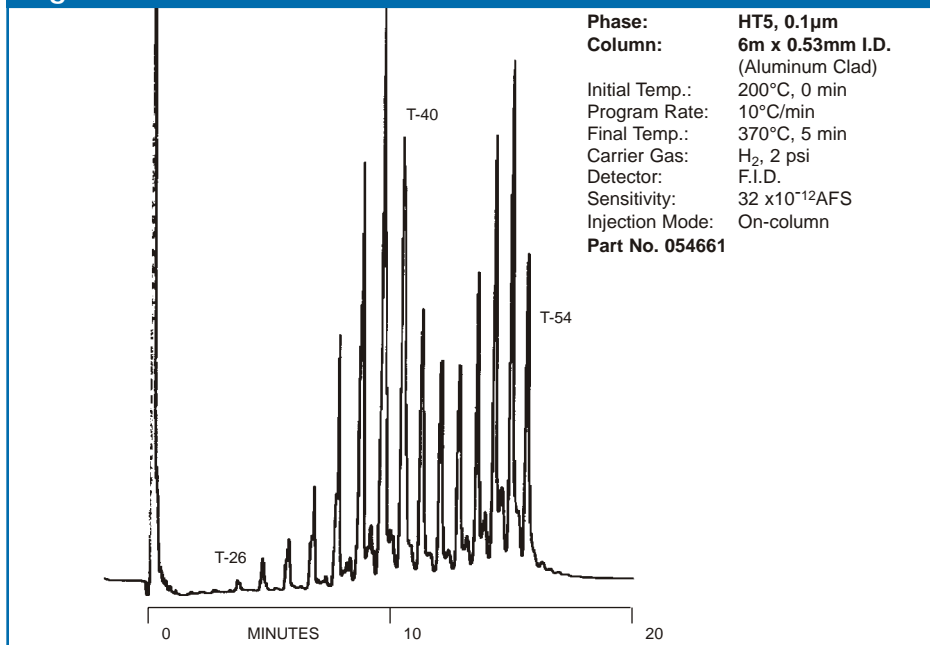


Figure 6 - Triglycerides Standard



Another example of the unique quality of HT5 capillary columns is in the analysis of metal complexed and free based alkylporphyrins (figure 5). Traditionally their analysis has required the preparation of low boiling point derivatives as no stationary phase could operate at temperatures higher than 400°C. Because alkylporphyrins are very thermally stable, their analysis, either as metal complexes or free bases, becomes extremely easy. The ease in which this analysis can be performed is well illustrated, where baseline resolution is obtained for a mixture of metal complexed and based alkylporphyrins.

Figure 7 - Butter Milk Fat

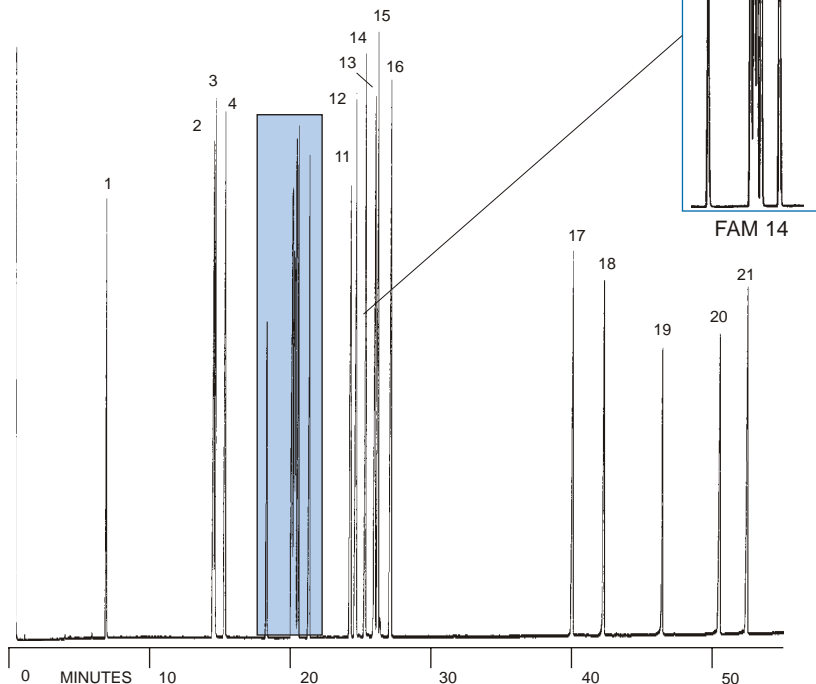


HT5 allows the routine analysis of triglycerides by capillary gas chromatography to be performed without difficulty. The separation of triglycerides by their respective carbon number is of considerable importance particularly in the various food, cosmetic and confectionery industries. A range of triglyceride standards analysed (figure 6) using a relatively short column illustrates the high level of performance the HT5 column can provide. Butter milk fat was also analysed using a HT5 column of only 6 meters in length and 0.53 mm I.D (figure 7). Excellent resolution between each respective carbon number group was achieved with an analysis time of only 17 minutes.

**Figure 8 - Fame Mixture**

**Phase:** HT5, 0.1µm  
**Column:** 25m x 0.22mm I.D. (Polyimide Clad)  
 Initial Temp.: 120°C  
 Program Rate: 3°C/min  
 Final Temp.: 300°C  
 Carrier Gas: H<sub>2</sub>, 10 psi  
 Detector: F.I.D.  
 Sensitivity: 32 x10<sup>-12</sup>AFS  
 Injection Mode: Split  
**Part No. 054636**

1. 13:0	12. 20:4
2. 16:1 cis	13. 20:3
3. 16:1 trans	14. 20:2
4. 16:0	15. 20:1
5. 17:0	16. 20:0
6. 18:2 cis	17. 25:0
7. 18:2 trans	18. 26:0
8. 18:1 cis	19. 28:0
9. 18:1 trans	20. 30:0
10. 18:0	21. 31:0
11. 19:0	



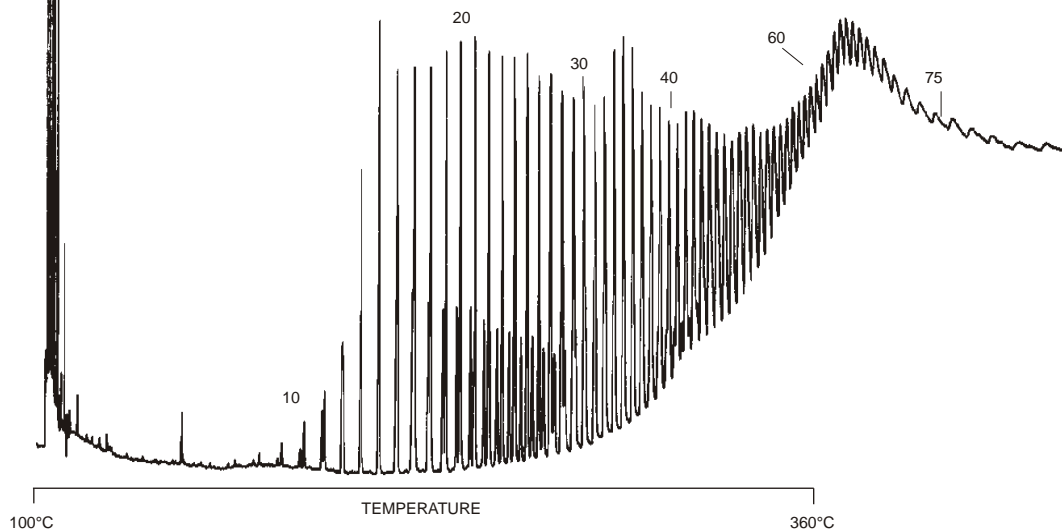
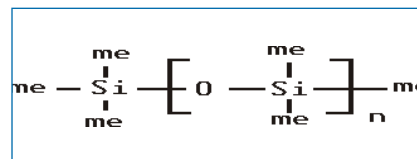
The HT5 stationary phase has unique selectivity because of its siloxane backbone. An example of this unique characteristic is the analysis of fatty acid methyl esters where excellent resolution of the C18:1 and C18:2 cis/trans isomers is achieved (figure 8).

With a maximum operating temperature some 150°C higher than conventional siloxane phases, the analysis of much higher molecular weight methyl esters or other derivatised compounds would become routine.

An indication of the superior stability of HT5 as a stationary phase for capillary gas chromatography is its ability to actually chromatograph many materials that themselves have been used as stationary phases. The analysis of DC200 (500cs) containing long chain siloxanes is easily performed with a HT5 column (figure 9).

**Figure 9 - DC200 Silicon Oil**

**Phase:** HT5, 0.1µm  
**Column:** 12m x 0.32mm I.D. (Aluminum Clad)  
 Initial Temp.: 100°C  
 Program Rate: 10°C/min, 10 min hold  
 Final Temp.: 360°C  
 Carrier Gas: H<sub>2</sub>, 10 psi  
 Detector: F.I.D.  
 Sensitivity: 32 x10<sup>-12</sup>AFS  
 Injection Mode: On-column  
**Part No. 054651**



# HT5

## HIGH TEMPERATURE STATIONARY PHASE FOR CAPILLARY GAS CHROMATOGRAPHY

### WHICH COLUMN SHOULD I SELECT ?

Though originally developed for high temperature applications, HT5 has also found a unique place in the analysis of complex mixtures analysed at more conventional temperatures (280°C-300°C). To accommodate these different areas of application, HT5 is available with two protective fused silica outer coatings, aluminum (AQ) and polyimide .

### APPLICATIONS BELOW 360°C-370°C

For analyses performed at temperatures below 370°C SGE recommends the use of the conventional polyimide outer coated fused silica (QC).

The polyimide used is a high temperature material, and can withstand continuous operation at these elevated temperatures for prolonged periods of time.

### ORDERING INFORMATION - Phase Type HT5

#### Polyimide Clad Columns

ID mm	Film µm	10m	15m	30m
0.25	0.1	–	<b>054633</b>	<b>054634</b>
0.32	0.5	–	<b>054667</b>	<b>054668</b>
0.53	0.5	<b>054670*</b>	<b>054671*</b>	<b>054672</b>

ID mm	Film µm	6m	12m	25m
0.22	0.1	–	<b>054631</b>	<b>054632</b>
0.32	0.1	–	<b>054641</b>	<b>054642</b>
0.53	0.1	<b>054655</b>	–	–
0.53	0.15	<b>054656</b>	<b>054657</b>	<b>054658</b>

#### Aluminum Clad Columns

ID mm	Film µm	6m	12m	25m	50m
0.22	0.1	–	<b>054635</b>	<b>054636</b>	–
0.32	0.1	–	<b>054651</b>	<b>054652</b>	<b>054653</b>
0.53	0.075	<b>054673**</b>	–	–	–
0.53	0.1	<b>054661*</b>	–	–	–
0.53	0.15	–	<b>054662*</b>	<b>054665</b>	–

\*Simulated Distillation \*\*5 meter column for Simulated Distillation



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