RESTEK
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Applications note

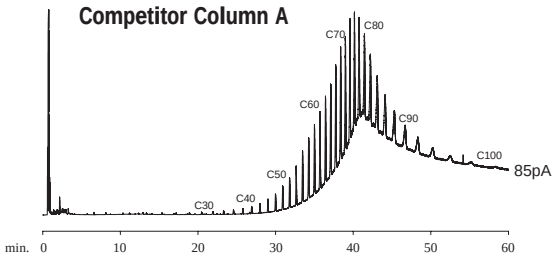
Fast, High-Temperature Sim Dist Analyses Using MXT®-1 HT Sim Dist Capillary GC Column and the GC Racer

ASTM Method D-6352 is a gas chromatography (GC) method developed for the determination of petroleum distillates with a boiling point range of 174˚C to 700˚C. Often referred to as "high-temperature simulated distillation," this method requires a capillary column capable of withstanding GC oven temperatures up to 430˚C. This presents many challenges for analysts because most capillary columns are manufactured using polyimide-coated fused silica tubing. At temperatures above 380˚C, even the best polyimide coating becomes brittle, which leads to very short column lifetimes. In addition, the methyl silicone stationary phase recommended in the method also must survive these high temperatures.

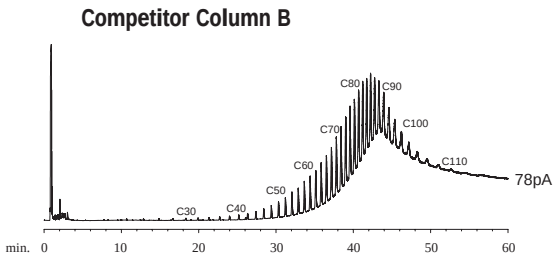
The MXT®-1 HT Sim Dist column is a major improvement in column technology for high-temperature simulated distillation. By combining a new, proprietary polymer synthesis technology, Siltek™ deactivation, and rugged Silcosteel® tubing, we developed a capillary column that meets all the criteria of ASTM Method D-6352. This MXT®-1HT Sim Dist column is available as in a 5m, 0.53mm ID, 0.1µm film dimension to conform to the requirements of this method. It exceeds the resolution, peak shape, and bleed criteria for hydrocarbons ranging up to C-110. Because the MXT®-1HT Sim Dist column is coated with a 100% dimethyl polysiloxane polymer, it will give the correct retention time/boiling point curve. The MXT®-1HT Sim Dist column exhibits low bleed and excellent inertness, and the rugged Silcosteel® tubing will hold up to temperatures in excess of 430°C.

To demonstrate the lower bleed and improved peak shape of this innovative column, a Polywax® 1000 reference material was analyzed using a MXT®-1HT Sim Dist column and two other columns that are commonly used for this application **(Figure 1)**. The MXT®-1 HT Sim Dist column exhibits lower bleed and improved peak symmetry compared to competitor columns.

Figure 1
The MXT-1HT Sim Dist column offers lower bleed and improved peak symmetry compared to other high temperature columns.



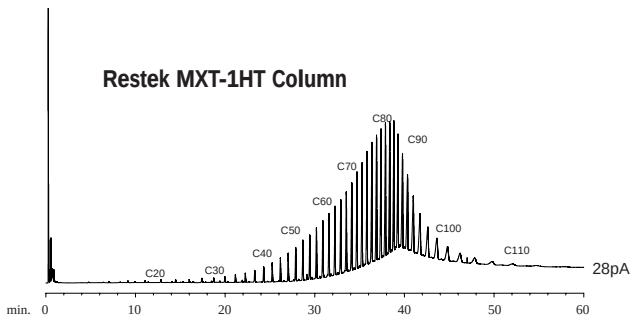
GC_PC00541



GC_PC00542

Run Conditions: C44-C100 Hydrocarbons

Column:	MXT-1HT Sim Dist
Serial #:	339749B
Description:	5m, 0.53mmID, 0.10µm
Sample:	Polywax® 1000
Part #:	36227
Solvent:	Carbon Disulfide
Sample Size:	0.2uL
Instrument:	HP6890 GC
Injector:	on-column (track oven)
Carrier Gas:	Helium(constant pressure)
Head Pressure:	1.0psi
Linear Velocity:	60cm/sec.
Flow Rate:	7.8mL/min.
Detector:	FID/430°C
Make up Gas Flow:	40cc/min.
Temp. Program:	40°C to 430°C@10°C/min. (hold 30 min.)



GC_PC00530

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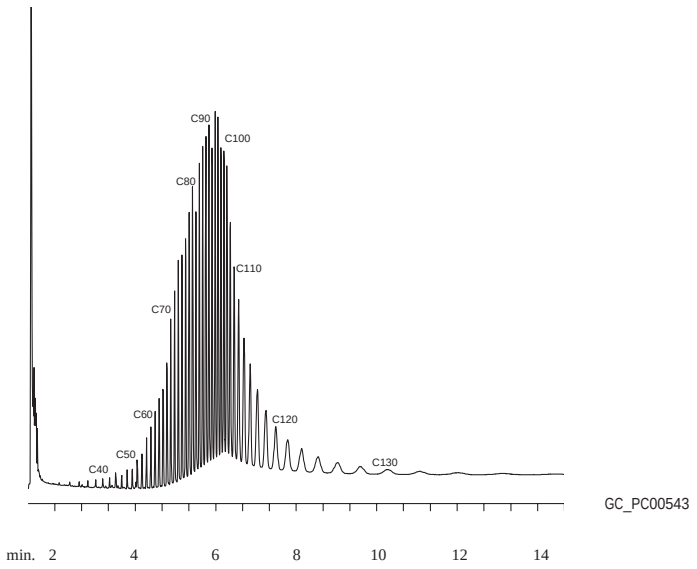
To maintain the low bleed and high performance of the MXT®-1HT Sim Dist column, it is critical to prevent oxygen from entering the column. This can be achieved by checking your entire system for leaks before and after each thermal cycle. We also recommend the use of graphite ferrules; Vespel® or Vespel®/graphite ferrules will not withstand the high temperatures required for this analysis as they may crack.

As part of cost reduction efforts, many laboratories try to reduce individual analysis time in the interest of increasing overall throughput. High-temperature simulated distillation analyses can take as long as an hour, especially when samples contain hydrocarbons up to C110. An effective technique to reduce analysis time is to use rapid temperature programming. Unfortunately, most GC systems have temperature-programming limitations of 20˚C to 25˚C/min. Some GCs can heat quickly at low temperatures, but cannot maintain the fast rate at higher temperatures, like those needed for Sim Dist. To overcome these limitations, Restek offers the GC Racer, an attachment to your Agilent 5890A GC and 5890 Series II, that increases the rate of temperature programming. Using the GC Racer, the analysis of the Polywax® 1000 reference material can be reduced from over 50 minutes to less than 15 minutes by temperature programming at 60˚C/min. **(Figure 2)**!

The Restek MXT®-1HT Sim Dist column is the ideal choice for high-temperature simulated distillation. It meets all the criteria of ASTM Method D-6352 while providing low bleed, excellent peak shape and resolution. Combining the MXT®-1HT Sim Dist column with the GC Racer attachment significantly reduces overall analysis time and greatly increases sample throughput.

Figure 2
Significantly reduce simulated distillation analysis time using the MXT®-1HT Sim Dist column and the GC Racer attachment.



Column:	MXT-1HT Sim Dist
Description:	5m, 0.53mmID, 0.10um
Part #:	70100
Sample:	Polywax 1000
Solvent:	Carbon Disulfide
Sample Size:	0.2ul
Sample:	Polywax 1000
Solvent:	Carbon Disulfide
Sample Size:	0.2ul
Temp. Program:	40°C 60°C/min. to 430°C (30min.)

Product Listing

MXT®-1HT Sim Dist (metal column)

ID	df (µm)	temp. limits	5-Meter
0.53mm	0.10	-60 to 430°C	70100

Polywax Standards

Decription	qty.	cat#
Polywax 1000	1 gram	36227

GC Racer™ GC Temperature Programmer

Decription	qty.	cat#
For Agilent 5890 Series II (only) GC	ea.	23024
For Agilent 5890A (only) GC	ea.	23025

For more information on the GC Racer attachment contact Restek's Technical Service Department or your local representative.