

A High-Temperature Polydimethylsiloxane-Phase Column for ASTM D-6352-98 Simulated Distillation Analyses: MXT® 1HT Sim Dist

- Stable to 430°C.
- Low bleed.
- Long lifetime at high temperatures.
- Symmetrical hydrocarbon peaks.
- Consistent resolution and retention times.
- Boiling point elution of hydrocarbons.
- Polarity equivalent to existing liquid phases.

Simulated distillation per ASTM D-6352-98 is used for determining the boiling point range distribution of petroleum distillate fractions with initial boiling points (BP) > 174°C and final boiling points < 700°C at atmospheric pressure. High temperature SimDist presents many challenges. The stationary phase must meet rigid resolution and retention time requirements, yet be stable at high temperatures. Further, the polyimide protective coating on the outer surface of most capillary columns has a maximum working temperature of about 380°C. Above this temperature the polyimide rapidly deteriorates. When repeatedly programmed to temperatures above 400°C, or allowed to cool below 50°C, the aluminum sheath on most aluminum-clad fused silica columns separates from the underlying fused silica surface. The tubing becomes extremely brittle, and column lifetime is significantly shortened.

To conform to the critical criteria set forth by ASTM, Restek chemists have developed the MXT® 1HT Sim Dist simulated distillation column. The MXT® 1HT polymer is a 100% polydimethylsiloxane (PDMS) material that is thermally stable to 430°C, requires minimal conditioning, and is 100% crosslinked. The MXT® 1HT phase is coated onto highly deactivated stainless steel tubing that has the inertness of fused silica without the temperature limitations. The MXT® 1HT Sim Dist column has a lifetime of at least 400 injections under typical SimDist conditions.

To demonstrate the robustness of MXT® 1HT Sim Dist columns, we made a series of 400 injections of Polywax® 1000 (cold on-column, CS₂ solvent, 1µL each) onto a randomly chosen column, and monitored critical performance characteristics over the course of these injections: resolution, retention times, stationary phase bleed. Figure 1 illustrates the Polywax® 1000 analysis after 400 injections. The hydrocarbon components still are well resolved and are easily quantified. Figure 2 plots the reproducibility of C50/C52 resolution and Figure 3 shows retention time reproducibility. After 400 injections, both of these critical characteristics still meet simulated distillation specifications. Figure 4 plots the consistently low bleed at 430°C over the series of 400 injections.

The stainless steel tubing used to make MXT® 1HT Sim Dist columns incorporates state-of-the-art Sulfinert™ deactivation. The deactivation layer is incorporated into the framework of atoms on the tubing surface, and therefore will not fracture or flake off, even if the column is flexed or bent. MXT® 1HT Sim Dist columns do not exhibit higher selectivity toward aromatics than toward normal hydrocarbons, thus they provide true boiling point values.

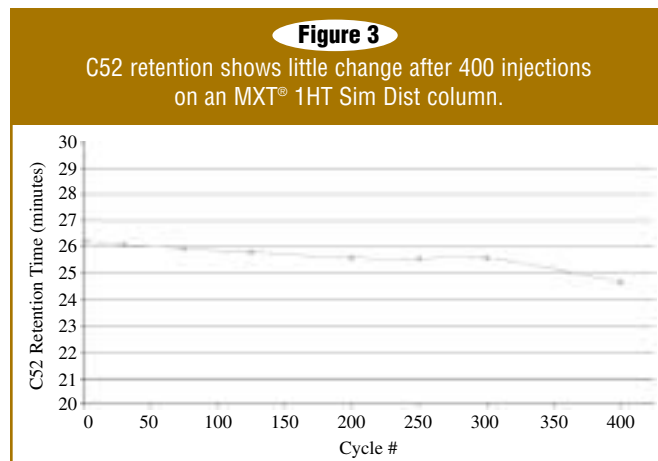
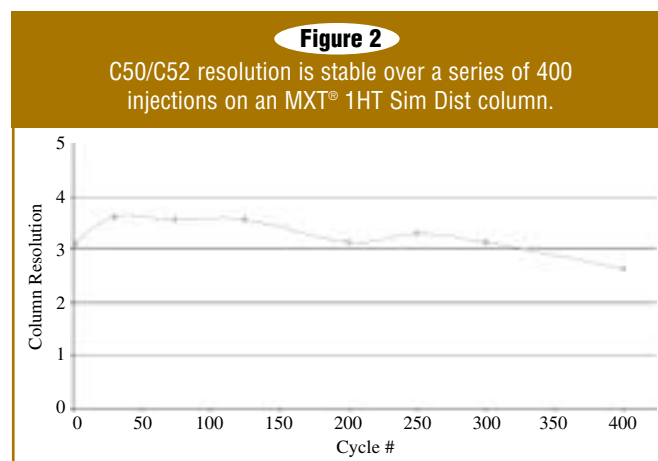
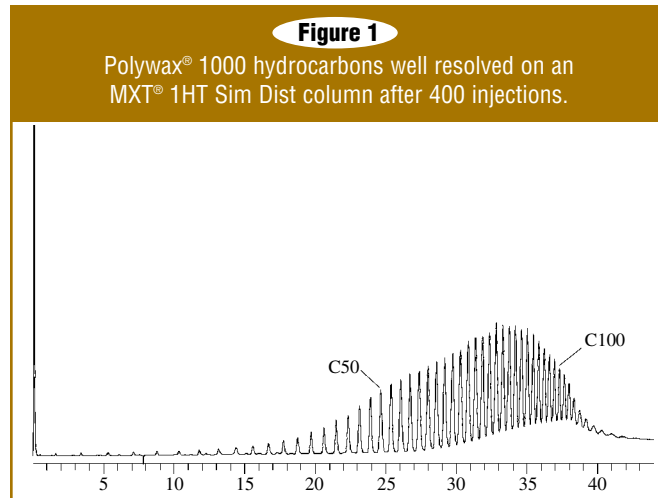
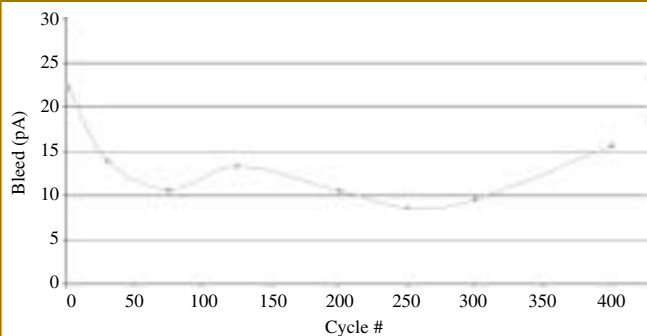


Figure 4

An MXT® 1HT column produces less than 20pA bleed over a series of 400 injections.



These excellent performance characteristics make MXT® 1HT Sim Dist columns the columns of choice for ASTM D-6352-98 SimDist analyses. Note that the demanding temperature conditions of simulated distillation analyses make GC system integrity a prime concern. It is imperative that the GC system be oxygen-free, to prevent phase degradation and maintain the highest level of chromatographic performance. We strongly recommend using oxygen-free carrier gas and routinely leak-testing your system with an electronic leak detection device, such as our Leak Detective™ II electronic leak detector (cat.# 20413), to ensure protection from oxygen.

visit us online at 
www.restekcorp.com

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

Description	qty.	cat.#
Polywax® 500	1 gram	36224
Polywax® 655	1 gram	36225
Polywax® 850	1 gram	36226
Polywax® 1000	1 gram	36227

Leak Detective™ II Leak Detector

The compact, affordable tool every analyst should have!

Description	qty.	cat.#
Leak Detective™ II Leak Detector	ea.	20413

Never use liquid leak detectors on a capillary system because liquids can be drawn into the system.

Caution: NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.

For permission to reproduce any portion of this application note, please contact Restek's publications/graphics department by phone (ext. 2128) or FAX.

Restek Trademarks: Leak Detective, MXT, Sulfinert, Restek logo.

Other Trademarks: Polywax - Petrolite Specialty Polymers Group.

