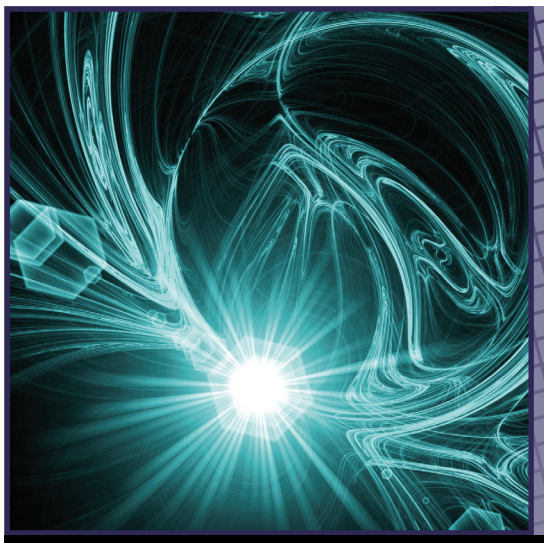

Improve High Temperature Simulated Distillation with MXT®-1HT SimDist Columns

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High temperature simulated distillation of crude oil for the determination of medium and heavy fraction petroleum distillates presents many challenges to refineries and contract laboratories. Due to the high boiling points of the hydrocarbons being analysed, both the column construction and the polymer used as the stationary phase must be robust enough to withstand the high temperatures required for analysis without significant degradation. Columns must yield sharp, symmetrical peaks that meet method requirements for resolution and peak shape. ASTM methods D6352, D7500, D7213, and D7169 all specify C50/C52 resolution values and list criteria for skewness (Table I). Low bleed, high efficiency, metal MXT®-1HT SimDist columns reliably meet all critical parameters and ensure accurate boiling point determination for simulated distillation methods.

Robust Columns are Required for Accurate Boiling Point Data

While fused silica columns can be used for simulated distillation, they deteriorate rapidly at extreme temperatures because of the limited stability of the outer polyimide coating. Metal columns, such as MXT®-1HT SimDist columns, are a much better choice. The polymer used for the stationary phase in the column must also be robust enough to exhibit low bleed levels, or retention time shifting, peak skewing, poor resolution, and final boiling point calculation errors can result. High stationary phase bleed eventually leads to shortened retention times, making it necessary to rerun boiling point calibrations more frequently. In contrast, low bleed columns produce stable retention times due to the stationary phase remaining in the column. This results in longer in-calibration periods, extended column lifetimes, and more accurate final boiling point determinations.

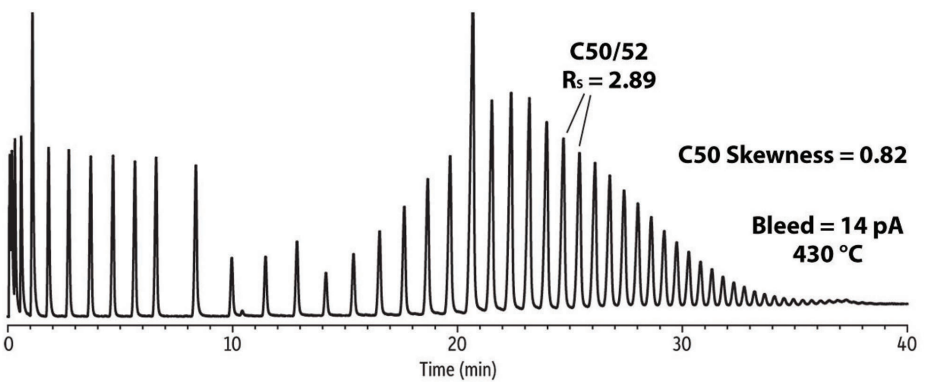
MXT®-1HT SimDist columns are exceptionally stable 100% dimethyl polysiloxane columns that can be used up to 450°C. These columns exhibit very low bleed and reliably meet or exceed requirements for multiple ASTM simulated distillation methods. The example chromatography shown here demonstrates typical results for ASTM Method D6352 (Figures 1 and 2), ASTM Method D7213 (D2887 extended, Figure 3), and ASTM Method D7169 (Figure 4). All method requirements are easily met, and the exceptionally low bleed levels result in precise slice times and accurate final boiling point determination.

Table I: Recommended high temperature columns for use in ASTM simulated distillation methods.

ASTM Method	Range	Recommended Column	Method Requirement
D2887	C5-C44	5/10 m x 0.53 mm ID, df = 0.88 - 2.65 µm	C16/C18 resolution (R_S) ≥ 3.00
D7213 (2887-ext.)	C5-C60	5 m x 0.53 mm ID, df = 0.15 - 1.2 µm	C50/C52 resolution: >1 and <10, C50 skewness: >0.5 and <2.0
D3710	Gasoline up to FBP 260°C (C14)	10 m x 0.53 mm ID, df = 2.65 µm	C12/C13 resolution: >2 and <4
D5307	Crude up to FBP 538°C (C42)	5 m x 0.53 mm ID, df = 0.20 µm PDMS	C16/C18 resolution: >3 and <10
D6352/D7500	C10-C90/C7-C110	5 m x 0.53 mm ID, df = 0.10 - 0.20 µm	C50/C52 resolution: >2 and <4, C50 skewness: >0.5 and <2.0
D7169	C5-C100	5 m x 0.53 mm ID, df = 0.10 - 0.20 µm	C50/C52 resolution: >1.8 and <4 Skewness (any C12-C24 paraffin): >0.8 and <1.2

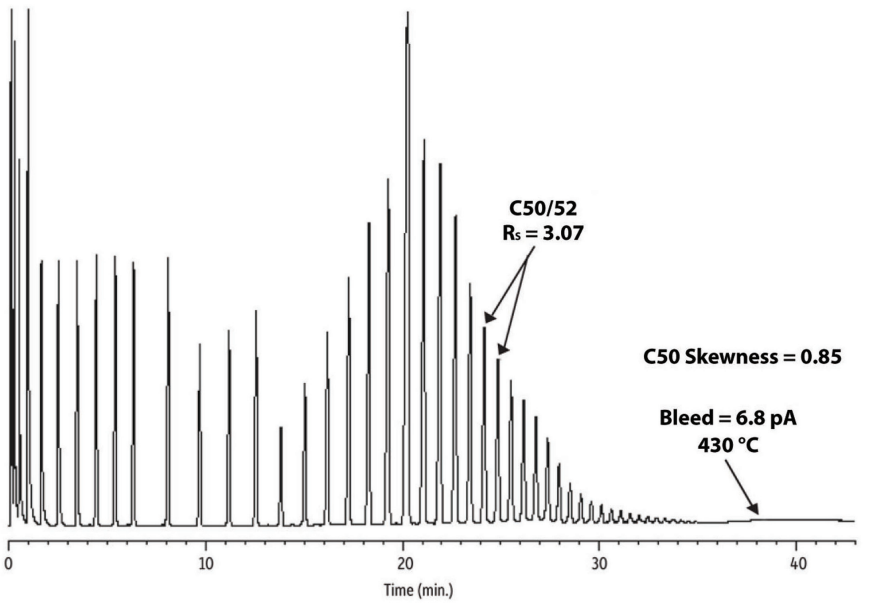
FBP = final boiling point

Figure 1: Low-bleed, high-efficiency 0.2 µm MXT®-1HT SimDist columns perform well under ASTM D6352 conditions.



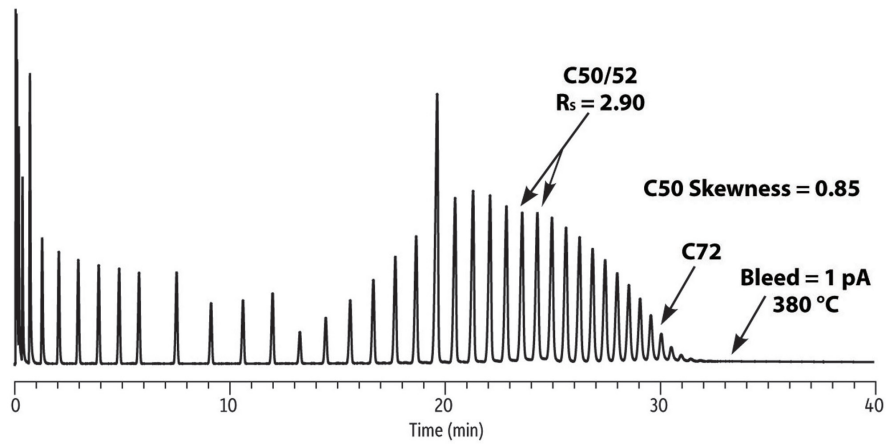
Column: MXT®-1HT SimDist, 5 m, 0.53 mm ID, 0.20 µm (cat.# 70115); Sample: C8-C100 hydrocarbons; Diluent: CS_2; Conc.: 1%; Injection: 0.5 µL cold on-column; Temp. Program: 53°C to 430°C at 10°C/min. (hold 5 min.); Oven Temp: 50°C to 430°C at 10°C/min. (hold 5 min.); Carrier Gas: He, constant flow, 18 mL/min.; Detector: FID @ 430°C; Make-up Gas Flow Rate: 24 mL/min.; Make-up Gas Type: N_2; Instrument: Shimadzu 2010 GC.

Figure 2: Excellent resolution and peak shapes are seen on 0.1 µm MXT®-1HT SimDist columns, resulting in more accurate final boiling point determinations (ASTM D6352 conditions).



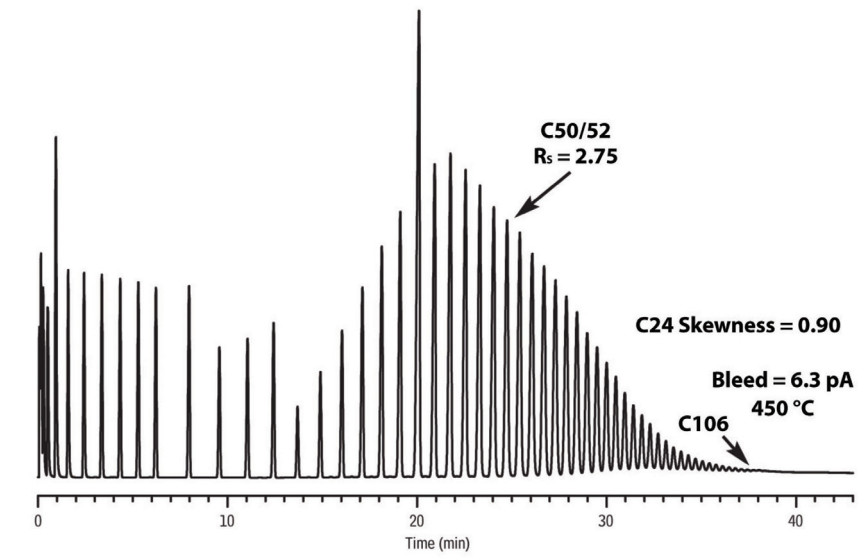
Column: MXT®-1HT SimDist, 5 m, 0.53 mm ID, 0.10 µm (cat.# 70112); Sample: C8-C100 hydrocarbons (Separation Systems SD-ss3e-05); Diluent: CS_2; Conc.: 1%; Injection: 0.5 µL cold on-column; Temp. Program: 53°C to 430°C at 10°C/min. (hold 5 min.); Oven Temp: 50°C to 430°C at 10°C/min. (hold 5 min.); Carrier Gas: He, constant flow, 18 mL/min.; Detector: FID @ 430 °C; Make-up Gas Flow Rate: 24 mL/min.; Make-up Gas Type: N_2; Instrument: Shimadzu 2010 GC.

Figure 3: ASTM D7213-05 (D2887 extended) resolution and skewness requirements are easily met using low bleed MXT®-1HT SimDist columns.



Column: MXT®-1HT SimDist, 5 m, 0.53 mm ID, 0.10 µm (cat.# 70112); Sample: C5-C72 hydrocarbons (Separation Systems SD-ss3e-03); Diluent: CS_2; Conc.: 1%; Injection: 0.5 µL cold on-column; Temp. Program: 53°C to 380°C at 10°C/min. (hold 12 min.); Oven Temp: 50°C to 380°C at 10°C/min. (hold 12 min.); Carrier Gas: He, constant flow, 12 mL/min.; Detector: FID @ 390°C; Make-up Gas Flow Rate: 18 mL/min.; Make-up Gas Type: N_2; Instrument: Shimadzu 2010 GC.

Figure 4: ASTM D7169 symmetry and resolution criteria are reliably met and exceptionally low bleed is observed at 450°C with MXT®-1HT SimDist columns.



Column: MXT®-1HT SimDist, 5 m, 0.53 mm ID, 0.10 µm (cat.# 70112); Sample: Custom C5-C106 hydrocarbon standard; Diluent: CS_2; Conc.: 1%; Injection: 0.5 µL cold on-column; Temp. Program: 53°C to 450°C at 10°C/min. (hold 5 min.); Oven Temp: 50°C to 450°C at 10°C/min. (hold 5 min.); Carrier Gas: He, constant flow, 18 mL/min.; Detector: FID @ 450°C; Make-up Gas Flow Rate: 24 mL/min.; Constant Column + Constant Make-up: 42 mL/min.; Make-up Gas Type: N_2; Data Rate: 20 Hz; Instrument: Shimadzu 2010 GC.

Increase Lab Productivity with High Efficiency Columns

In addition to low bleed, column efficiency is also a critical factor in simulated distillation. Columns that are higher in efficiency produce sharper peaks and higher resolution values, which result in more samples analysed before the column fails the minimum resolution specification. The chromatograms in Figures 1-4 show typical separations for simulated distillation following ASTM methods; in all cases, the high efficiency of the MXT®-1HT SimDist column provides the resolution needed for highly reproducible results.

In addition to high efficiency, column inertness can affect resolution as active sites in a poorly deactivated column can result in significant tailing. MXT®-1HT SimDist columns undergo a unique deactivation process, Siltek® treatment, which bonds amorphous silica to the inner surface of the column tubing. This process stabilises the dimethyl polysiloxane coating resulting in a highly inert, exceptionally low bleed column. Siltek® deactivation reduces active sites, minimises tailing, and allows more accurate low level sample analysis.

Conclusion

The MXT®-1HT Sim Dist column is a robust analytical column that performs well for ASTM high temperature simulated distillation methods. The exceptionally low-bleed and high-efficiency characteristics of this column translate directly into assured method performance, reliable final boiling point determinations, and better control of refinery processes.