Tighten Control of Distillation Processes with the New MXT® - 1HT Sim Dist Column

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High temperature crude oil simulated distillation using ASTM methods D6352 and ASTM D7500 presents many challenges to refineries and contract laboratories for the determination of petroleum distillates in the boiling range of 174°C to 720°C. Both the column construction and the polydimethylsiloxane polymer must be robust enough to withstand the high method temperatures without significant degradation. In addition, columns must yield a C50/C52 resolution value between 2 and 4, exhibit acceptable peak symmetry, and reliably produce accurate boiling point data. Accurate determination of the boiling range distribution of medium and heavy fractions using GC simulated distillation provides critical information regarding the composition of crude oil and other products related to refinery processes. While fused silica columns can be used, they are not the best option anymore for high temperature simulated distillation applications. Metal MXT®-1HT Sim Dist columns are a much better alternative. These new columns are more stable at 430°C than fused silica columns and easily meet all method criteria, even under accelerated conditions.

Exceptional Stability Extends Column Lifetime

The polyimide coating on a high temperature fused silica column can become damaged and delaminate during temperature cycling. In the example shown in Figure 1, the polyimide coating flaked off immediately upon removal of the column from the instrument, causing complete deterioration of the column. A similar process occurs for aluminium clad fused silica columns, also rendering them unsuitable for high temperature applications. In contrast, the metal MXT®-1HT Sim Dist column withstands the aggressive temperature conditions with no apparent damage. Another important component of high temperature analysis is the thermal stability of the polymer itself; it must be robust enough to exhibit low bleed at 430°C, thus eliminating retention time shifting, peak skewing, poor resolution, and final boiling point calculation errors. The MXT®-1HT Sim Dist polymer is stable to 430°C and programmable to 450°C with no degradation; typically, no deterioration is seen even after 400 injections. The stability of both the tubing and the polymer results in more reliable boiling point determinations and longer column lifetimes.

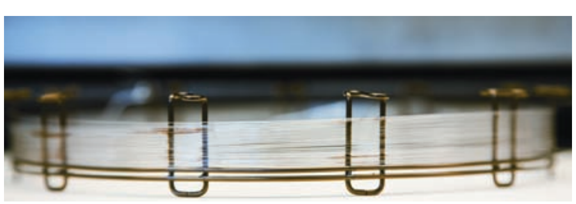

Figure 1: MXT®-1HT Sim Dist columns are unaffected by high method temperatures. In contrast, the coating on fused silica columns can be damaged and may delaminate completely, significantly reducing column lifetime. (Fused silica column, 72 hours at 430°C).

Meets all Method Criteria, Even under Accelerated Conditions

In addition to offering high temperature stability, Restek's MXT®-1HT Sim Dist columns undergo a unique deactivation treatment (Siltek®), which bonds an amorphous silica to the inner surface of the column tubing. This process results in a highly inert sample pathway that reduces active sites and allows accurate part-per-billion level sample analysis. As shown in Figure 2, MXT®-1HT Sim Dist columns meet all method requirements, including peak symmetry for C50 (skewness = 0.77) and the separation of C50 and C52 (resolution = 2.50). Figure 2 also illustrates column robustness; bleed is minimal even at 430°C, which is essential for precise slice times and accurate final boiling point determination.

The performance of the MXT®-1HT Sim Dist column is maintained even under accelerated thermal conditions. For example, data provided by Joaquin Lubkowitz (Separation Systems) demonstrate ideal performance at 2.6 times the oven ramp rate specified in the method (Figure 3). For this work, the MXT®-1HT Sim Dist column was used in conjunction with a Separation Systems near on-column PTV injector, a rapid cool-down injector that results in more efficient sample throughput. The high oven and PTV temperature ramps are made possible by two important factors, the focusing ability of the column/injector and the uniform polymer deposition (coating efficiency) of the column. Without the effectiveness of these characteristics, the nearly perfect peak symmetry and baseline resolution of the heavier hydrocarbons would not be possible at these accelerated temperature ramps and high flow rates. The resulting calibration curve is shown in Figure 4.

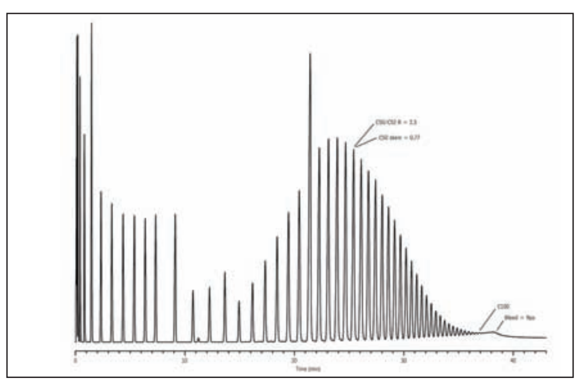

Figure 2: Outstanding peak symmetry and baseline resolution on MXT®-1HT Sim Dist columns result in more accurate final boiling point determinations.
Column: MXT®-1HT Sim Dist, 5m, 0.53mm ID, 0.20µm (cat.# 70103); Sample: C5-C100, 1% in carbon disulfide; Inj.: 1µL on-column (PTV); Inj. temp.: 53°C to 430°C @ 10°C/min. (hold 5 min.); Carrier gas: helium, constant flow; Flow rate: 18mL/min.; Oven temp.: 50°C to 430°C @ 10°C/min. (hold 5 min.); Det.: FID @ 430°C; Instrument: Shimadzu 2010.

Conclusion

The MXT®-1HT Sim Dist column is a robust analytical column and is the most reliable option for high temperature simulated distillation according to ASTM D6352. These columns are extremely stable and easily meet all method criteria exhibiting low bleed, unsurpassed C50/C52 resolution, excellent peak symmetry (C5-C100), and stable retention times. New MXT®-1HT Sim Dist columns are ideal for labs interested in more accurate, reliable final boiling point determinations and better control of refinery processes.

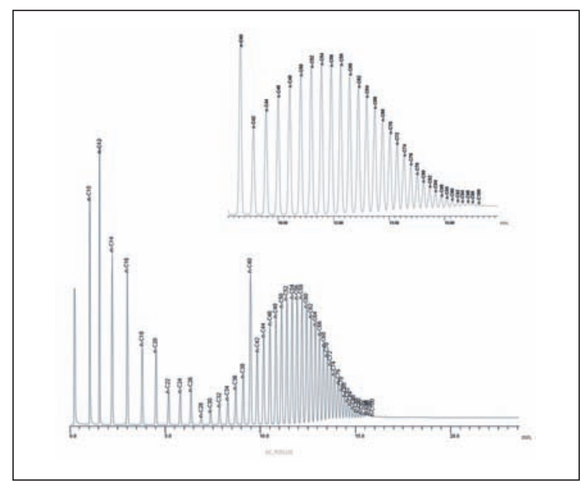

Figure 3: Robust MXT®-1HT Sim Dist columns meet all ASTM D6352 requirements, even under accelerated conditions. Column: MXT®-1HT Sim Dist, 5m, 0.53mm ID, 0.20µm (cat.# 70103); Sample: C10-C100, 1% in carbon disulfide; Inj.: 0.2µL near on-column (PTV); Inj. temp.: 40°C to 430°C @ 100°C/min.; Carrier gas: helium, constant flow; Flow rate: 20mL/min.; Oven temp.: 40°C to 430°C @ 25°C/min.; Det.: FID @ 430°C.

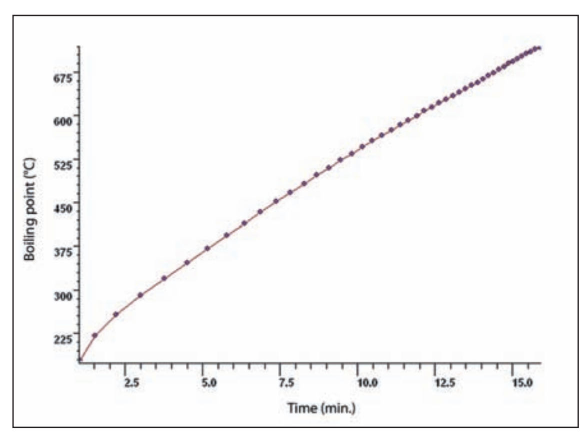

Figure 4: Boiling point calibration curve for C10-C100 hydrocarbons (instrument conditions shown in Figure 3).

Acknowledgements

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