

BPX1-SimD A New Era in Simulated Distillation Technology

SGE would like to thank Dr. J. Lubkowitz and the staff at Separation Systems Inc for supplying the data for this product data sheet.

INTRODUCTION

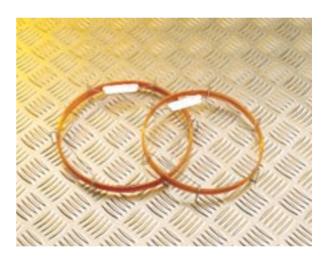
SGE's BPX capillary column range includes the most thermally stable, long life columns available.

BPX1 has been designed as a high temperature alternative to conventional 100% dimethylsilicone stationary phases. With a routine operating temperature of 430°C, extremely low bleed and excellent chemical inertness, the columns are superior to all "MS" grade columns on the market.

The BPX1 column offers two major advantages over competitors' conventional dimethylpolysiloxane columns;

- 1. The low bleed at the upper temperatures required for the extended high temperature analysis results in better integration and therefore better quantitation for the higher hydrocarbon numbers. This results in the ability to quantify C110 without background subtraction or column compensation from a blank analysis.
- 2. Lower column bleed means less loss of column phase and therefore a smaller decrease in capacity ratios (the phase thickness remains constant). This is important to the practising chromatographer as the calibration can be carried out less often because of the greater stability in retention times.

The phase is not the only important factor for an excellent column. All of SGE's polyimide clad columns utilize a polyimide that is very stable at temperatures above 400°C. This allows the ease of use of polyimide clad fused silica tubing with the stability of metal.



The BPX1-SimDist columns are specifically optimized for ASTM Method D2887 and the new High Temperature Simulated Distillation Method (HTSD). These columns can also be used for oil volatility and for gasoline and gasoline fractions (ASTM D3710).

Figure 1. Standard Mix for HTSD using BPX1-SimD

 Phase:
 BPX1, 0.1μm

 Column:
 5m x 0.53mm ID

 Initial Temp.:
 40°C

 Rate:
 15°C

 Final Temp.:
 420°C, 5 min.

 Detector Temp:
 440°C

 Carrier Gas:
 Helium, 10mL/min

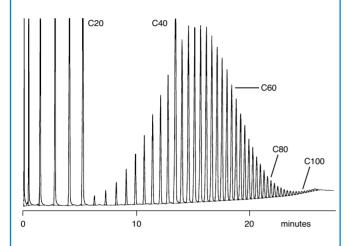
 Instrument:
 HP 6890

Part No:

054800

Separation Systems Injector
Initial Temp.: 40°C
Rate: 15°C
Final Temp.: 420°C, 5 min.

C, 5 min. 420 C, 5 m C Jum, 10mL/min 1890

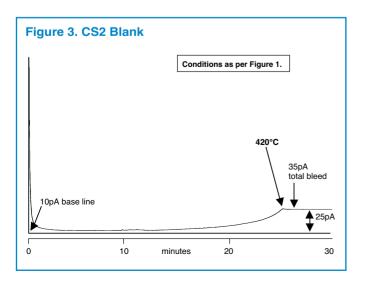


C40 C60 C80 C100 C90 C110

20

minutes

10



HTSD ON BPX1

The analysis of a standard mixture used for HTSD is shown in **Figure 1.** This mixture uses hydrocarbons ranging from C10 to C20, Polywax 655 and C40. C40 is spiked into the mix as a reference point in the mixture. All major peaks are even numbered carbons. This chromatogram shows excellent separation, minimal bleed and the ability to quantify C110 without using background subtraction or column compensation from a blank analysis - all in less than 25 minutes at a final temperature of 420°C.

A portion of the previous chromatogram from C40 to the end of the analysis (expanded vertically) shows excellent resolution and the ability to see beyond C110 (**Figure 2**).

Figure 3 is a blank analysis of neat carbon disulfide. This displays the bleed from the column at 420°C of 25 picoamps. This low bleed allows for easier integration of the peaks above C100.

The analysis of a reference gas oil (**Figure 4**) is used to verify the calibration of the system in regard to boiling points. It guarantees the effectiveness of the column to produce simulated distillation data that fits within specified guidelines of reproducibility.

The calculated data from this analysis are shown in **Table 2.** This data shows excellent correlation between the expected temperature at which a certain percentage of the reference gas oil is expected to elute and the calculated temperature from the calibration.

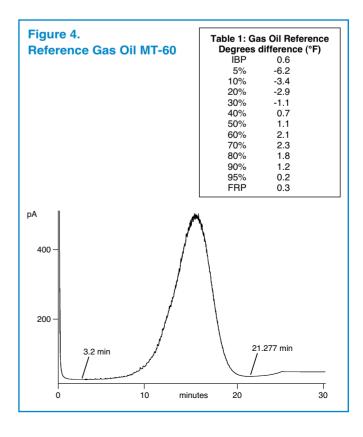


Table 2.	QC Boiling	Point Table	ASTM D2887 High-Temp.
% Off	BP(°F)	QC(°F)	Diff
IBP	698.6	698.0	0.6
5.00	872.8	879.0	-6.2
10.00	918.6	922.0	-3.4
15.00	943.8		
20.00	963.1	966.0	-2.9
25.00	979.6		
30.00	993.9	995.0	-1.1
35.00	1005.8		
40.00	1016.7	1016.0	0.7
45.00	1027.3		
50.00	1037.1	1036.0	1.1
55.00	1046.3		
60.00	1055.1	1053.0	2.1
65.00	1063.5		
70.00	1072.3	1070.0	2.3
75.00	1081.3		
80.00	1091.8	1090.0	1.8
85.00	1103.3		
90.00	1117.2	1116.0	1.2
95.00	1138.2	1138.0	0.2
FBP	1194.3	1194.0	0.3

EXTENDED SIMULATED DISTILLATION

The analysis of a standard retention time standard is shown in Figure 5. This mixture uses hydrocarbons ranging from C6 to C20, Polywax 655 and C40. C40 is spiked into the mix as a reference point in the mixture for easy carbon counting. All major peaks beyond C18 are even numbered carbons. This chromatogram shows excellent separation, minimal bleed and the ability to quantify to C78 with the operating conditions stated in the figure.

Figure 6 is a portion of the previous chromatogram from the beginning of the analysis to 10 minutes into the run. It shows excellent resolution and peak shape of the early eluting components (C6 to C20).

Figure 7 is a blank analysis of neat carbon disulfide. This displays the bleed from the column at 390°C of only 57 picoamps. This minimal bleed allows for easier integration of the peaks above C70.

The analysis of Reference Gas Oil #2 (Figure 8) is used to verify the calibration of the system with regard to boiling point distribution. It guarantees the effectiveness of the column to produce simulated distillation data that fits within specified guidelines of reproducibility.

The calculated data from this analysis are shown in **Table 3**. This data shows excellent correlation between the expected temperature at which a certain percentage of the reference gas oil is expected to elute and the calculated temperature from the calibration.

Distillation of crude oil and petroleum products has been a mainstay for decades in refinery and commercial laboratories in order to evaluate crude oils or products. Only in recent years have engineers accepted distillation data produced by gas chromataography. These "distillations" are called "simulated distillations" since they are not true distillations in the strict sense. ASTM method D2887 (Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography) is simulated distillation products and fractions which have a final boiling point of 538°C (1000°F) or lower at atmospheric pressure (C44). This method has been extended (but is still to be accepted by ASTM) to boiling points of up to 750°C (1380°F). Two methods which are used are called extended D2887 (to C70) and High Temperature Simulated Distillation (HTSD) (to C90 and beyond). Up to this point two capillary column phases have been used for these methods. The two phases used are SGE's HT5 and polymethylsiloxane.

Figure 5. Retention Time Standard

BPX1, 0.9μm Column: 10m x 0.53mm ID Initial Temp.: 40°C Rate 10°C Final Temp.: 390°C, 10 min. Detector Temp 400°C Helium, 20mL/min Carrier Gas: HP 6890 Instrument: Part No: 054801

Phase:

Rate: 10°C

Initial Temp.:

Separation Systems Injector

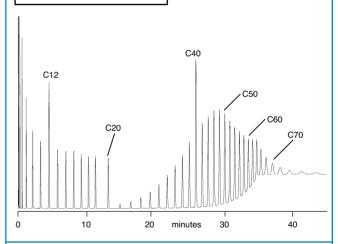
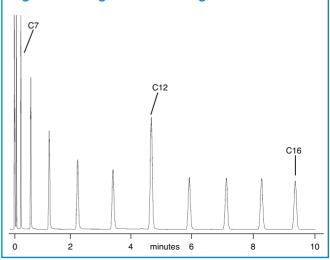
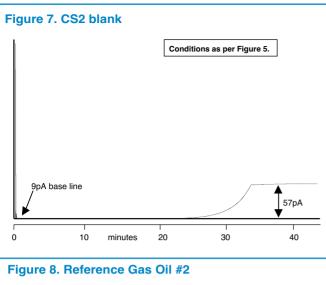
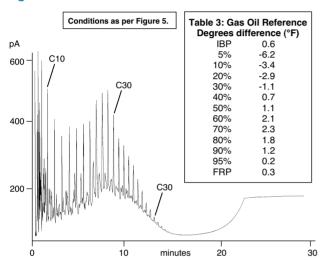


Figure 6. Enlarged section of Figure 5







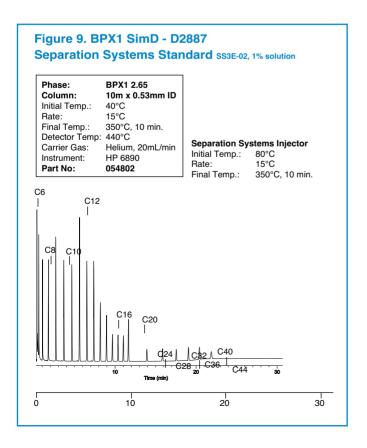


Table 3.	QC Boiling	Point Table	ASTM D2887 Extended
% Off	BP(°F)	QC(°F)	Diff
IBP	238.8	240.0	-1.2
5.00	305.0	304.0	1.0
10.00	348.0	348.0	0.0
15.00	394.6	393.0	1.6
20.00	436.8	435.0	1.8
25.00	471.3		
30.00	500.7	499.0	1.7
35.00	527.9		
40.00	553.6	552.0	1.6
45.00	577.6		
50.00	594.6	594.0	0.6
55.00	610.2		
60.00	629.2	629.0	0.2
65.00	648.7	649.0	-0.3
70.00	668.3	668.0	0.3
75.00	689.9	690.0	-0.1
80.00	712.1	712.0	0.1
85.00	736.6	736.0	0.6
90.00	764.1	764.0	0.1
95.00	803.2	803.0	0.2

D2887 SIMULATED DISTILLATION

The chromatographic parameters used for this analysis are shown in **Table 4**. The column gave excellent results for D2887 without forcing the column above 350°C.

The analysis of a standard retention time standard for D2887 is shown in **Figure 9.** This mixture uses hydrocarbons ranging from C5 to C44. The major peaks between C20 and C44 are separated by 4 carbons. This chromatogram show excellent separation, minimal bleed and the ability to quantify beyond C44 with the operating conditions stated in the figure.

Figure 10 is a blank analysis of neat carbon disulfide. This displays the bleed from the column at 350°C of only 59.5 picoamps. This bleed allows for easier integration of the peaks above C44 and also keeps the detector cleaner for extended periods. Low bleed means the column lasts longer since the phase does not deteriorate at a rapid pace.

The analysis of Reference Gas Oil (**Figure 11**) is used to verify the calibration of the system in regard to boiling point distribution. It guarantees the effectiveness of the column to produce simulated distillation data that fits within specified guidelines of reproducibility.

The calculated data from this analysis are shown in **Table 4.** This data shows excellent correlation between the expected temperature at which a certain percentage of the reference gas oil is expected to elute and the calculated temperature from the calibration.

BPX1-SimD

A New Era in Simulated Distillation Technology

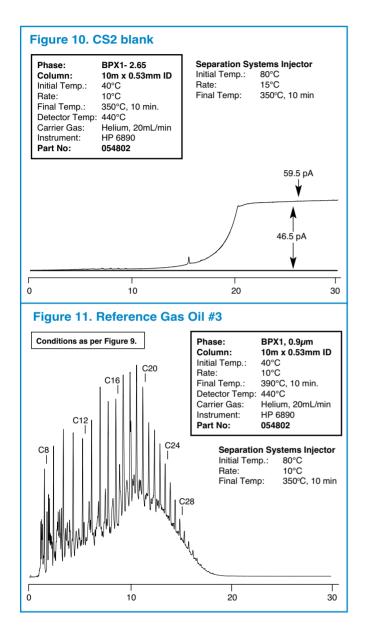


Table 4. 0	QC Boiling I	Point Table	
% Off	BP(°F)	QC(°F)	Diff
IBP	239.9	240.0	-0.1
5.00	304.4	304.0	0.4
10.00	348.3	348.0	0.3
15.00	395.6	393.0	2.6
20.00	438.3	435.0	3.3
30.00	502.3	499.0	3.3
40.00	555.3	552.0	3.3
50.00	596.1	594.0	2.1
60.00	629.3	629.0	0.3
65.00	651.0	649.0	2.0
70.00	670.5	668.0	2.5
75.00	692.1	690.0	2.1
80.00	714.5	712.0	2.5
85.00	737.7	736.0	1.7
90.00	766.8	764.0	2.8
95.00	806.4	803.0	3.4
FBP	888.6	888.0	0.6

ORDERING INFORMATION - BPX1 - SIMD

ID mm	Film <i>µ</i> m	5m (Aluminium)	10m (Polyimide)
0.53	0.1	054800	-
0.53	0.9	-	054801
0.53	2.65	-	054802



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