



EN15199-1 Determination of boiling range distribution by gas chromatography method Part 1: Middle distillates and lubricating base oils

KEY WORDS: SIMULATED DISTILLATION, DISTILLATE FUELS, LUBRICATING BASE OILS

INTRODUCTION

SCION Instruments offers the solution for a simulated distillation of middle distillates and lubricating base oils.

EN15199-1 specifies a standardized method for the determination of boiling range distribution of middle distillates and lubricating base oils by gas chromatography, with initial boiling points (IBP) of at least 100°C and final boiling points (FBP) of below 750°C (C₁₂₀).

Simulated Gas chromatography distillations can be used as a reference method for conventional distillation methods which can provide a quantitatively percent mass yield as a function of the boiling point for hydrocarbon components in the sample. The boiling point distribution is a routinely used parameter to monitor and improve the production process and quality of refinery products. Simulated distillations are a safer and faster way than conventional distillations to perform a similar boiling range distribution of distillates.

This SIMDIST application is applicable on the SCION Instruments 8X00 GC-platform, shown in figure 1, with the 8400PRO autosampler.



Figure 1. SCION Instruments 8X00 GC platform equipped with the 8400PRO autosampler.

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RESULTS AND DISCUSSION

The SCION SIMDIST analyzer used for this application, is equipped with a SCION Instruments 8X00-GC who's configured with a temperature programmable cold on column (COC) injector with air cooling, a SCION-Simdist column and a high temperature Flame Ionization Detector (FID) with ceramic HT flame tip. The analyzer uses a 8400PRO autosampler for excellent repeatability results, used in Standard On-Column mode.

Note that method EN15199-1 is not applicable for the analysis of petroleum or petroleum products containing low molecular weight components, such as naphtha's, reformates, gasolines and diesels. For the determination of heavy distillates and residuals fuels method EN15199-part 2 is recommended. For the analysis of crude oils method EN15199-part 3 is recommended.

Table 1 is showing the analytical conditions of the SCION Instruments GC system.

Table 1. Analytical conditions

Part	Settings
Injector (COC)	100°C, 15°C/min to 430°C
Column	SCION-Simdist
Oven Program	35°C , 10°C/min to 430°C
Carrier	Helium
Column flow	19 ml/min
Detector	FID with ceramic jet, 450°C Air: 300 ml/min, Fuel gas (H ₂): 30 ml/min, Make up (N ₂): 11 ml/min
Injection Volume	0.5 µl
Autosampler	8400PRO
Software	Compass CDS (with Eclipse plug in)

Reference oil 5010 1%

A reference oil with known initial boiling point and final boiling point is injected to validate the method and system. See figure 2 and table 2 for the results and specifications of the reference oil. In figure 2, point 1 is the start of elution time, point 2 the initial boiling point (IBP) and point 3 is the final boiling point (FBP). Point 4 is the end of elution of the sample and where the septum purge is opened for cleaning the system. The results in table 2 show that all the targets of the critical points are easily achieved.

Table 2. Results Reference oil 5010 1%

% (m/m)	Target	Result	°C	
			Δ Spec	Δ
IBP	428	421	±9	-7
5	477	476	±3	-1
10	493	491	±3	-2
20	510	509	±3	-1
30	524	524	±4	0
40	537	536	±4	-1
50	548	547	±4	-1
60	560	559	±4	-1
70	572	571	±4	-1
80	585	584	±4	-1
90	602	601	±4	-1
95	616	615	±4	-1
FBP	655	658	±18	3

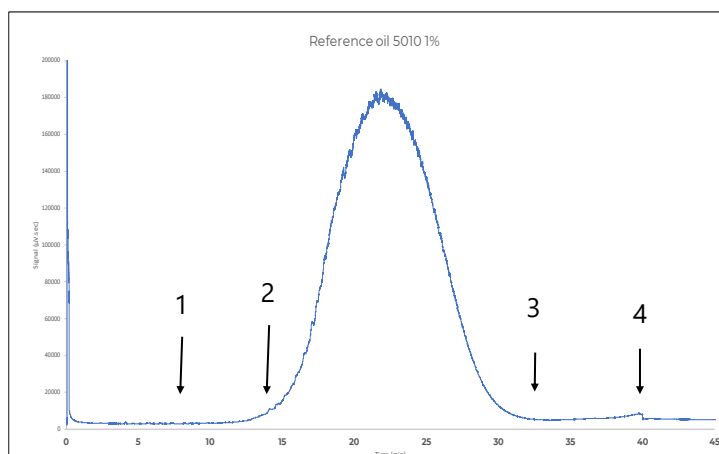


Figure 2. Chromatogram Reference Oil 5010 1%

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Repeatability (R) is determined by measuring the reference oil 5010 1% in duplicate. The difference (Δ) between the 2 generated boiling point distributions is calculated and compared with the set specification mentioned in EN15199-1. According to table 3 the system can easily achieve good repeatability results within specifications.

Table 3. Repeatability (R) results

°C				
%(m/m)	Measurement 1	Measurement 2	Δ Spec R	Δ R
IBP	421,5	426,5	± 6	-5
5	475,5	475,9	± 2	-0,4
10	491	491,3	± 1	-0,3
50	546,5	546,6	± 2	-0,1
60	558,4	558,5	± 2	-0,1
70	570,3	570,4	± 2	-0,1
80	583,7	583,8	± 2	-0,1
90	601	601,2	± 2	-0,2
95	615,4	615,9	± 4	-0,5
FBP	655,8	663,1	± 10	-7,3

A calibration mixture of normal paraffins covering the range from C_5 up to C_{120} dissolved in CS_2 was used to determine the relationship of boiling point (BP) versus retention times (RT).

See figure 3 for the chromatogram of the calibration standard and figure 4 for the retention time vs. boiling point of the calibration standard.

A gravimetric blend is analysed for checking the relative response of the two distillates and to check the baselines at the start, middle and end of the chromatogram. Figure 5 shows the chromatogram of analysis of the gravimetric blend, where a baseline separation is shown between the two fractions. Figure 6 is showing the boiling point curve vs. mass % of the gravimetric blend.

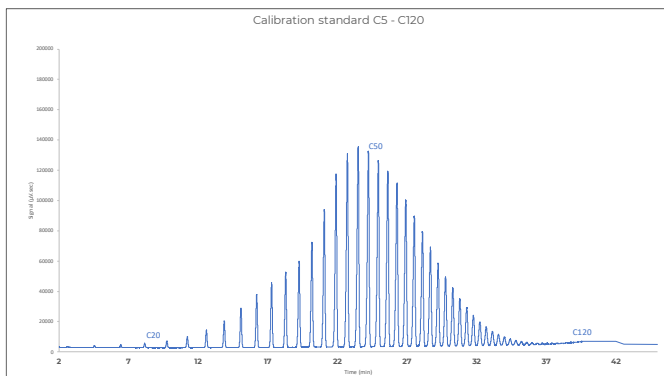


Figure 3. Chromatogram Calibration mix

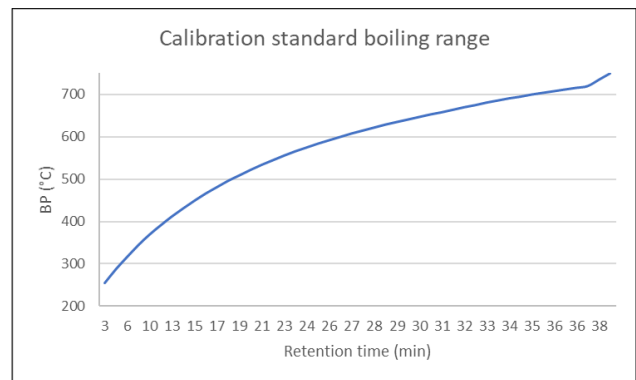


Figure 4. Retention time (RT) vs. boiling point

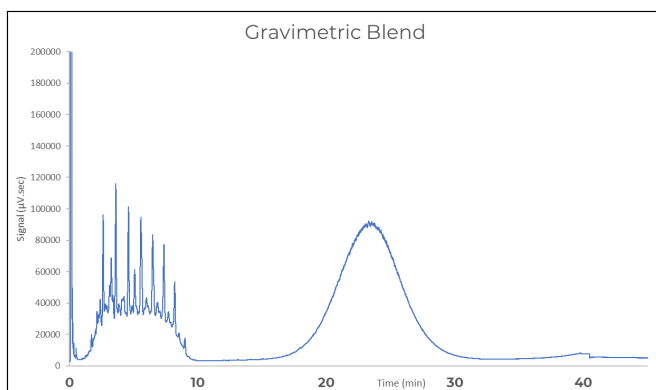


Figure 5. Chromatogram Gravimetric Blend

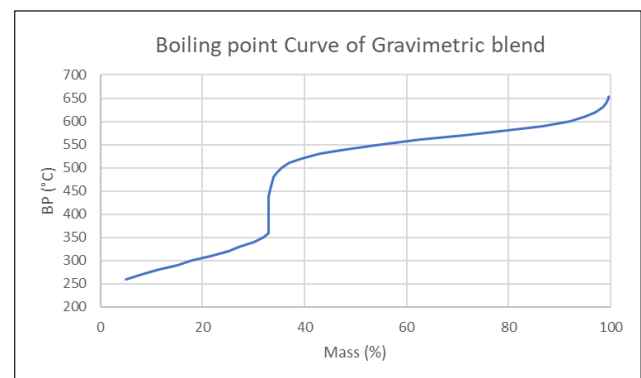


Figure 6. Boiling point curve of the Gravimetric Blend

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According to the results shown in table 4 the system and method are performing brilliantly within the specifications mentioned in EN15199-1.

Table 4. System performance results

Parameter	Specification EN15199-1	Found value
C ₂₀ Peak skewness at 5% peak height	1 ≥ 3	1,1
Column resolution	2 ≥ 4	2,75
Detector response (% m/m)	32,4 ± 0,6	33

Figure 7 is showing a chromatogram using the Compass Eclipse software plug in, with combined Retention time vs boiling point curve. The software makes it possible to easily obtain the boiling range distribution with different standardized methods, shown in the right handed column of the figure.

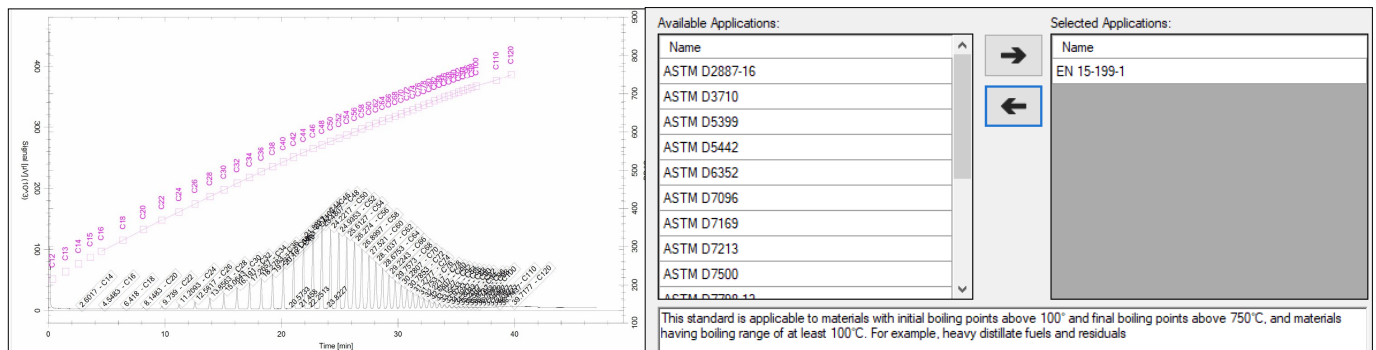


Figure 7. Chromatogram calibration mix using the Compass Eclipse software plug in, and other optional methods.

CONCLUSION

The SCION Instruments 8X00 GC-platform provides a robust and complete solution for the simulated distillation of distillates and lubricating base oils, following standardized method EN15199-1. The obtained results demonstrate that this method and the SCION Instruments GC system configuration performs excellent and meets all the specifications mentioned in EN15199-1.

The equipment of the 8X00-GC analyzer is pre determined, for ordering information or customisation please contact your local sales representative.

Although the 4X6-GC series is not shown in this application note, it is also possible to perform this method on the SCION instruments 4X6 GC series.

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