



IP507 Determination of boiling range distribution of heavy distillate and residual fuel oils — Gas chromatography method

KEY WORDS: SIMULATED DISTILLATION, HEAVY (VACUUM) DISTILLATE FUELS, RESIDUALS

INTRODUCTION

SCION Instruments offers the solution for a simulated distillation of heavy distillate and residual fuel oils.

Standardized method IP507 describes the determination of the boiling range distribution of heavy distillate and residual fuel oils, with initial boiling points (IBP) of at least 100°C and final boiling points of (FBP) above 750°C. This method is not applicable for analyzing bituminous samples or petroleum products containing low molecular weight components, such as naphthas, reformates, gasolines and diesels. Hetero atoms, such as alcohols, ethers, acids or esters or residue are not the be analyzed by this method.

Simulated Gas chromatography distillations can be used to replace conventional distillation methods for control of refining operations. Simulated distillations are a safer and faster way to perform an similar boiling range distribution of heavy distillates and residual fuel oils.

This application is applicable on the SCION Instruments 8X00 GC-platform, shown in figure 1.



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

IP507 Determination of boiling range distribution of heavy distillate and residual fuel oils — Gas chromatography method

RESULTS AND DISCUSSION

The SCION SIMDIST analyzer used for this application, comprises of a small footprint 8X00-GC, configured with a temperature programmable air cold on column (COC) injector with air cooling, a SCION SIMDIST column and a Flame Ionization Detector (FID) with ceramic HT flame tip.

A 8400PRO Autosampler was used in Standard On-Column mode. Standards were prepared in CS₂ and used to determine system performance and response factors. A qualitative mixture of normal paraffins covering the range from C₅ up to C₁₂₀ was used to determine the relationship of boiling point (BP) versus retention times (RT). CS₂ blanks were run and evaluated for cleanliness of the blank, for blank subtraction.

After running a calibration standard sample, the septum purge time is switched open after elution of the C₁₂₀ peak, for cleaning the system for the next injection. This action is shown in figure 2, 4 and 6 at 40,5 minutes.

Table 1 is showing the analytical conditions of the system.

Reference oil 5010 1%

A reference oil with known initial boiling point and final boiling point is injected to check if the method and system are working correctly. 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), point 3 final boiling point (FBP) and point 4 is the end of elution of the sample. The results in table 2 show that all the critical points are within allowance.

Repeatability of the reference oil is determined by analyzing 5 times the same sample consecutively. RSD (%) of the 5 measurements is 1.36%, which is excellent for simulated distillations.

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 FID with ceramic jet, 450°C
Detector	Air: 300 ml/min, Fuel gas (H ₂): 30 ml/min, Make up (N ₂): 11 ml/min
Inj. Volume	0.5 µl
Autosampler	8400PRO
Software	Compass CDS (with Eclipse plug in)

Table 2. Results Reference oil 5010 1%

% Off	Target (°C)	Result (°C)	Allowable Difference (°C)
IBP	428	430	9
5	477	477	3
10	493	492	3
20	510	509	3
30	524	523	4
40	537	536	4
50	548	547	4
60	560	559	4
70	572	570	4
80	585	584	4
90	602	601	4
95	616	615	4
FBP	655	673	18

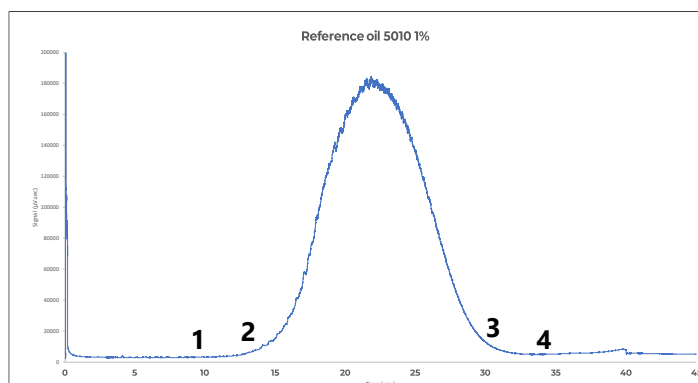


Figure 2. Chromatogram Reference Oil 5010

IP507 Determination of boiling range distribution of heavy distillate and residual fuel oils — Gas chromatography method

Calibration standard C₅-C₁₂₀

A calibration sample is injected for identification of the n-alkanes during analysis of an unknown sample.

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

System performance

The resolution of the column can be determined by using the C₅₀ and C₅₂ peaks and following equation:

$$R = \frac{2 * (t_2 - t_1)}{1.699 * (W_1 + W_2)}$$

Where:

t₁ = retention time in seconds for C₅₀ peak

t₂ = retention time in seconds for C₅₂ peak

W₁ = width in seconds at half-height of C₅₀ peak

W₂ = width in seconds at half-height of C₅₂ peak

The calculated resolution is 2.57, which is excellent since a good column resolution is between 2 and 4.

Another critical point is the peak skewness of peak C₂₀ at 5% peak height. A good peak skewness ratio shall not be less than 1 or greater than 3. The skewness of the C₂₀ peak is with 1.04 within specification.

Figure 5 is showing a chromatogram using the Compass Eclipse software plug in, with combined Retention time vs boiling point curve.

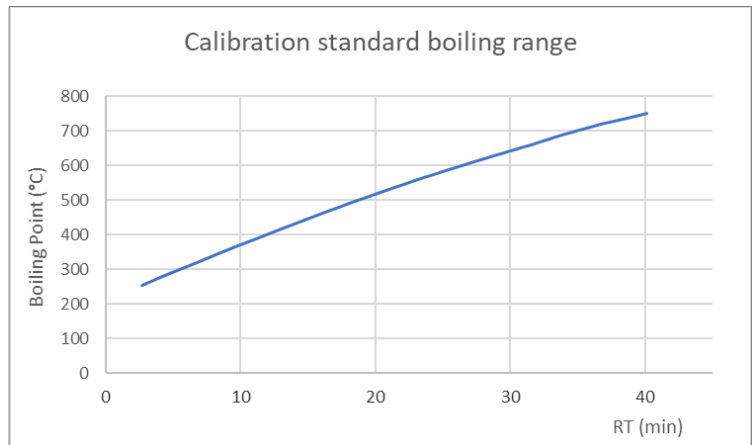


Figure 3. Retention time (RT) vs. boiling point

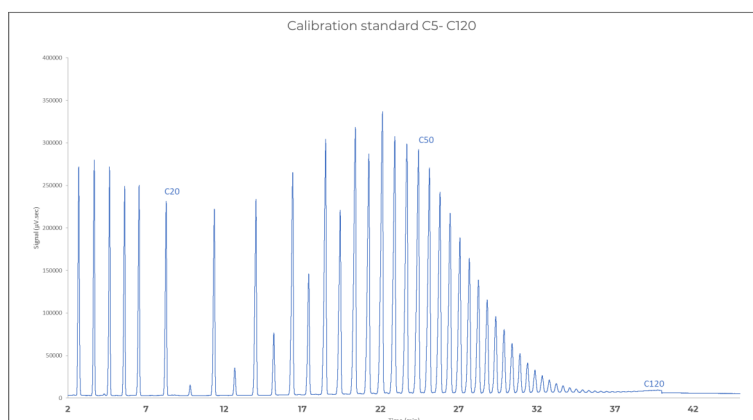


Figure 4. Chromatogram Calibration mix

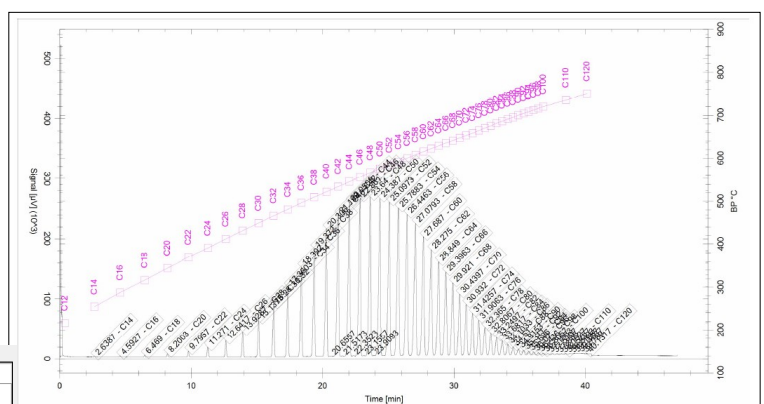


Figure 5. Chromatogram calibration mix using the compass Eclipse software plug in and optional methods

Available Applications:		Selected Applications:	
Name		Name	
ASTM D2887-16	→	IP 507	
ASTM D3710			
ASTM D5399			
ASTM D5442			
ASTM D6352			
ASTM D7096			
ASTM D7169			
ASTM D7213			
ASTM D7500			
ASTM D7700-13			

This test method covers the determination of the boiling range distribution of gasoline and gasoline components. This test method is applicable to petroleum products and fractions with a final boiling point of 500°F (260°C) or lower as measured by this test method.

IP507 Determination of boiling range distribution of heavy distillate and residual fuel oils — Gas chromatography method

Gravimetric blend

A gravimetric blend is used for checking the relative response of the two distillates and to check the baselines at start, middle and end of the chromatogram. Figure 6 shows the chromatogram of analysis of the gravimetric blend, where a baseline separation is shown between the two peaks.

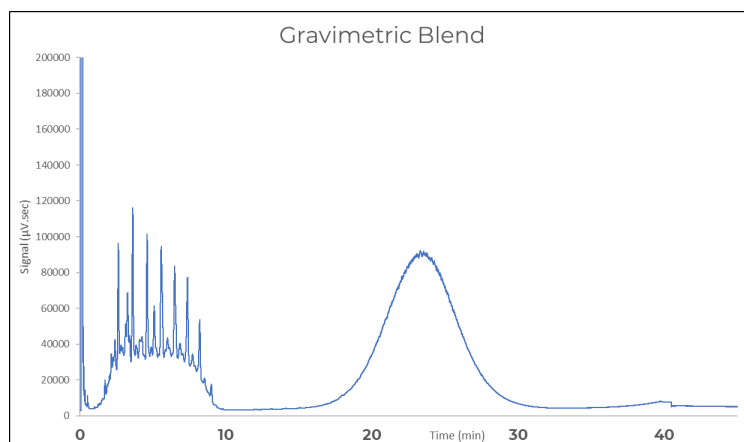


Figure 6. Chromatogram Gravimetric Blend

CONCLUSION

The SCION Instruments 8X00 GC-platform provides a robust and complete solution for the simulated distillation of heavy distillates and residual fuel oils, following standardized method IP507. The obtained results demonstrate that this method and SCION Instruments system configuration is excellent and meets all the required limits for IP507.

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 possible to perform this analysis on the SCION instruments 4X6 GC series.

SCION Instruments

UK

4 Michaelson Square
Livingston
EH54 7DP, Scotland, UK
Phone +44 1506 300 200
sales-eu@scioninstruments.com

The Netherlands

Amundsenweg 22-24
4462 GP Goes,
The Netherlands
Phone +31 (0) 113 287 600
sales-eu@scioninstruments.com