



## IP601 Determination of light hydrocarbons in stabilised crude oils — Gas Chromatography method

**KEY WORDS: DHA, IP601, Crude Oils, Engine Fuels, Boiling Point Distribution**

### INTRODUCTION

SCION Instruments offers the solution for detailed hydrocarbon analysis (DHA) of stabilized crudes.

Detailed hydrocarbon analysis provides the boiling point distribution of light hydrocarbons who are present in stabilized crude oils. (A stabilized crude oil is defined as having a Reid Vapor pressure equivalent to or less than 82.7 kPa) A boiling point distribution of these compounds is a frequently used parameter to monitor refining operations.

IP601 describes a standardized method for the determination of light hydrocarbons in stabilised crude oils up to and including n-nonane(C<sub>9</sub>). IP601 is issued under the fixed designation of ASTM D7900. For a full range of boiling point distribution of crude oils (>n-C<sub>9</sub>) this method can be combined with standardized method IP545 or ASTM D7169.

This application is applicable on the SCION Instruments 4X6 and the new 8X00 GC-platform, shown in Figure 1, equipped with the 8400PRO autosampler.

Table 1 is showing the analytical conditions of the GC method.



**Figure 1. SCION Instruments 8300 and 8500 GC platform equipped with the 8400PRO autosampler.**

**Table 1. Analytical conditions**

<b>Injector (SSL)</b>	Split 100:1, 275 °C
<b>Column 1</b>	SCION-1
<b>Column 2</b>	Scion-DHA
<b>Oven Program</b>	35°C (30 min), 2°C/min to 185°C
<b>Carrier</b>	Helium
<b>Pressure</b>	365 kPa (53 psi)
<b>Detector</b>	FID with ceramic jet, 300°C
	Air: 300 ml/min, Fuel gas (H <sub>2</sub> ): 30 ml/min, Make up (N <sub>2</sub> ): 28 ml/min
<b>Inj. Volume</b>	0.1 µl
<b>Autosampler</b>	8400PRO
<b>Software</b>	Compass CDS (with Eclipse plug in)

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### RESULTS AND DISCUSSION

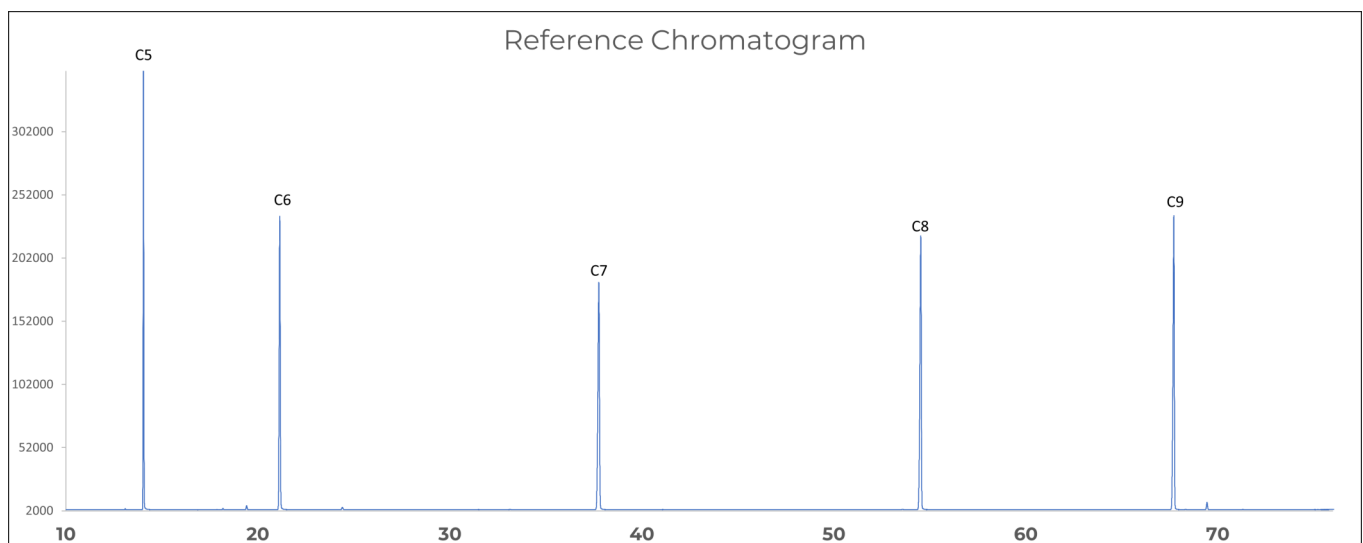
Standardized method IP601 determines the boiling point range distribution of hydrocarbons in stabilized crude oil up to n-nonane (n-C<sub>9</sub>). As mentioned earlier, a full range boiling point distribution of a crude oil is possible to measure in association with standardized method IP545 or ASTM D7169. These methods are perfectly capable to determine a hydrocarbons in crude oils, however the lighter hydrocarbons in these methods are showing more significant errors. IP601 is specially developed to determine the boiling point distribution of the front end more accurately without the interference imposed by the diluent solvent CS<sub>2</sub>.

The Eclipse software plug in makes it easily to merge these 2 methods for a reliable full boiling range distribution for the analysed crude.

A reference calibration mixture C<sub>5</sub>-C<sub>9</sub> is injected on the system for identification, see figure 2.

The identification of compounds is based on the Kovats index, generated by the Eclipse software plug in. The usage of this index has the advantage that it only depends on 2 psychical properties, in comparison with retention time who depends on 6 physical properties.

The calculated indices are compared with known data in the database and the rest of the peaks are assigned accordingly. Based on this standard, the composition of the crude oil front-end can be identified and the boiling point distribution can be calculated.



**Figure 2: Example chromatogram of the calibration mixture (reference C<sub>5</sub>-C<sub>9</sub>)**

Repeatability of the system and method is checked by analysing consecutively 6 times the reference calibration mixture of n-paraffins. Table 2 shows the repeatability results of the DHA IP601 analyzer. The analyzer is capable of achieving excellent results with an RSD below 1% for all components.

A critical point of attention during this analysis is the peak skewness ratio of the hexane peak at 5% peak height. A good peak skewness ratio shall be between 1 and 4, according to the specification mentioned in IP601. The skewness of the hexane peak shown in figure 2 has a good ratio of 2.33

**Table 2. Repeatability of 5 consecutive injections**

	Peak Area (µV·Sec)				
	C <sub>5</sub>	C <sub>6</sub>	C <sub>7</sub>	C <sub>8</sub>	C <sub>9</sub>
1	131323	127444	130738	133423	130878
2	131066	127878	131649	133557	130948
3	131713	128784	132889	134843	132215
4	129025	125388	128944	131787	129795
5	130322	126814	130613	132797	130818
6	130410	126511	129912	132728	130819
n	6	6	6	6	6
Average	130643,4	127136,9	130790,8	133189,0	130912,2
Stdev	872,1	1073,4	1249,0	936,1	702,9
RSD%	0,67	0,84	0,95	0,70	0,54

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A crude oil is injected on the system as an unknown sample. Figure 3 shows the chromatogram of the crude oil. In the chromatogram a select group of compounds are named. With the Eclipse software plug in all components are successfully identified.

Table 3 shows the group report of the crude oil in mass (%). Based on the mass report and internal standard, a boiling point distribution was made, shown in table 4.

A critical point of attention during this analysis is the resolution between the hexane peak and 1-hexene peak (internal standard) a good resolution shall be at least 2 listed in the IP601. The resolution in figure 3 between the peaks of interest is 2,33.

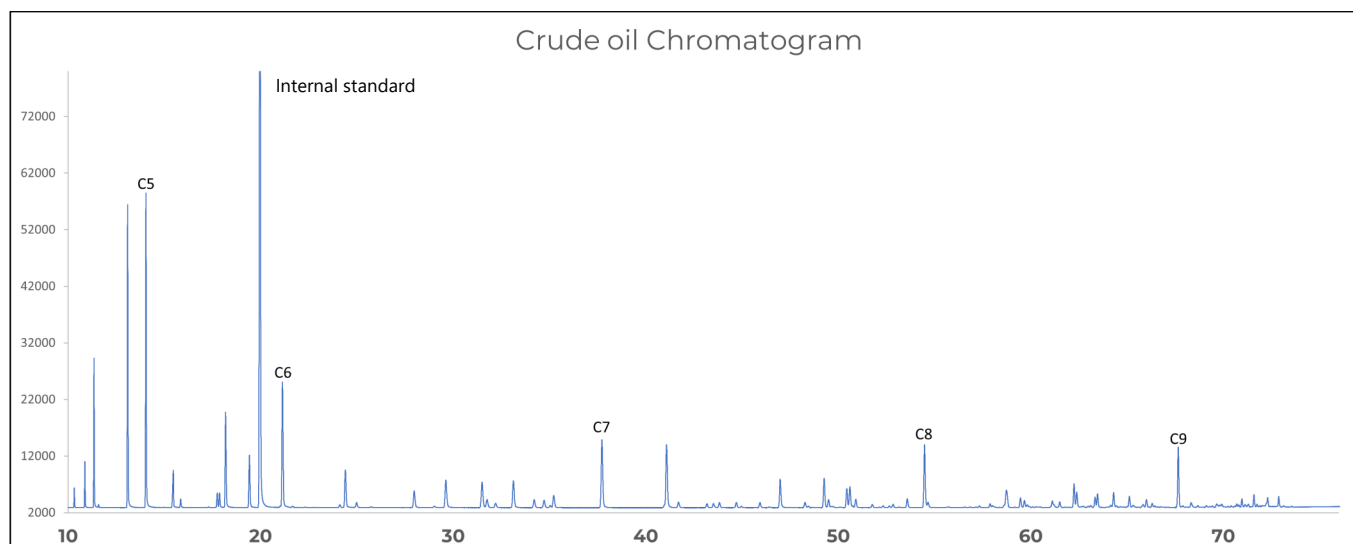
If desired the data from table 4 can be merged with method ASTM D7169 (or IP545) for the total boiling point distribution of the crude oil. SCION application note AN088 will give further information of the merge of methods IP545(or ASTM D7169) and IP601 (or D7900) to give this full boiling point distribution.

**Table 4. Boiling point distribution report of the crude oil**

Percentage (mass)	TPB (°C)	Percentage (mass)	TPB (°C)
IBP	-10.9	12.0	89.1
1.0	-5.4	13.0	91.8
2.0	6.3	14.0	94.0
3.0	17.9	15.0	98.8
4.0	28.2	16.0	104.6
5.0	31.3	17.0	114.6
6.0	34.4	18.0	118.9
7.0	58.1	19.0	124.0
8.0	60.2	20.0	136.1
9.0	64.5	21.0	138.9
10.0	67.6	22.0	146.7
11.0	73.7	23.0	165.0

**Table 3. Group report in mass% of the crude oil.**

	N-Paraffins	i-Paraffins	Naphthene's	Aromatics	Unknown	Total
<b>C3</b>	0.13	0.00	0.00	0.00	0.00	0.13
<b>C4</b>	1.03	0.30	0.00	0.00	0.00	1.32
<b>C5</b>	2.67	2.41	0.15	0.00	0.02	5.25
<b>C6</b>	1.65	2.00	1.11	0.28	0.00	5.03
<b>C7</b>	1.24	1.26	1.63	0.44	0.00	4.56
<b>C8</b>	0.99	1.41	0.93	0.76	0.17	4.27
<b>C9</b>	0.87	0.68	0.39	0.00	0.09	2.03
<b>Heavy</b>	0.00	0.00	0.00	0.00	0.66	0.66
<b>Total</b>	8.56	8.05	4.23	1.47	0.94	23.3



**Figure 3: Example chromatogram of crude oil with 1-hexene as internal standard.**

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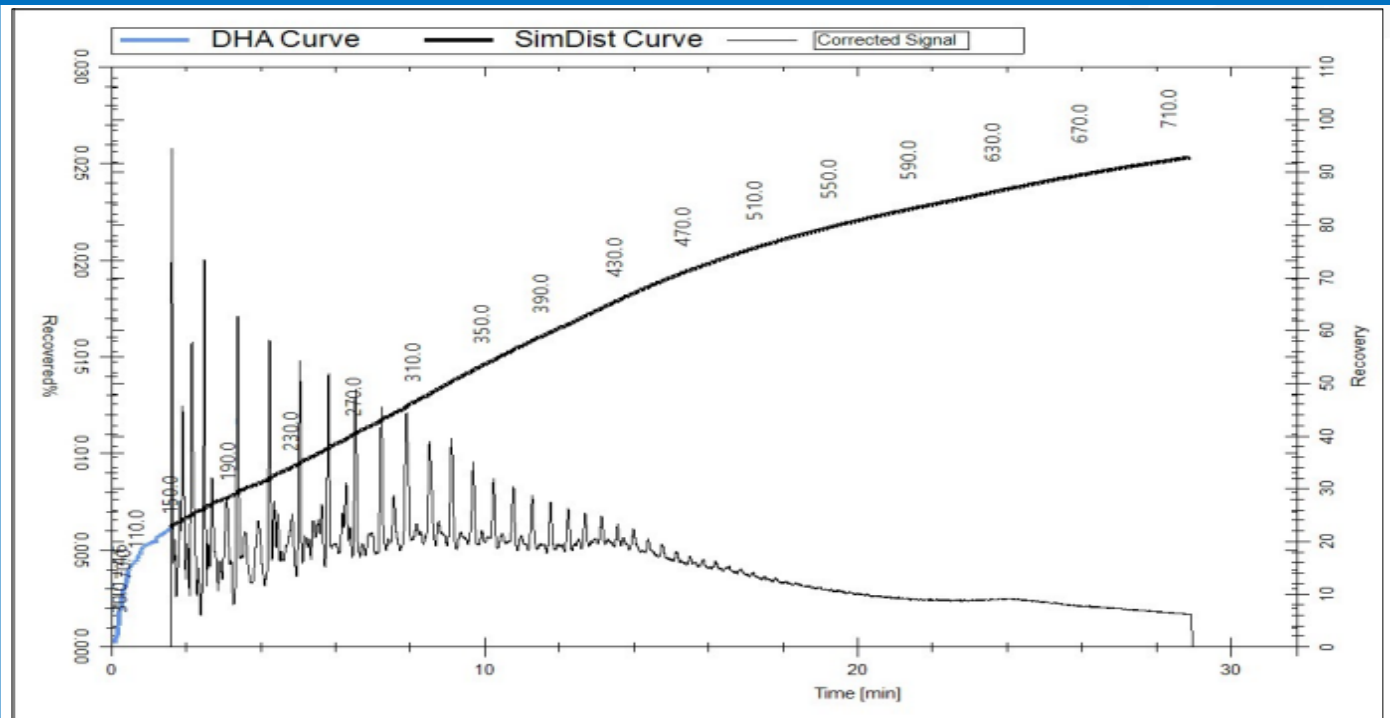


Figure 4: Example of a merged crude oil distribution report in Eclipse.

The Compass CDS Eclipse plug in is used for analysing the data. IP601 is related to method ASTM D7900, the obtained results are calculated in Eclipse according to ASTM D7900. The plug in is an addition to the basic Compass CDS and this will provide all the tools to calculate the necessary data.

Figure 4 shows an example of the merged crude oils distribution report in Eclipse. The front end boiling point distribution (<151°C) is based on the DHA IP601 data and is shown in blue. The back end of the crude boiling point distribution (>151°C) is based on standard method IP545 and is shown in black. Figure 5 shows an example of how the chromatogram looks like in the Eclipse plug in software, and the available standardized methods to select.

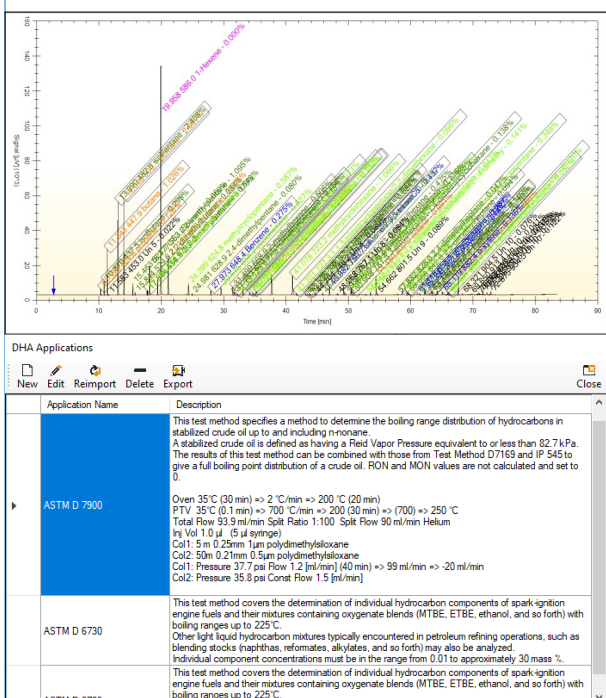


Figure 5: Chromatogram of a crude oil sample using the Compass Eclipse plug in.

## CONCLUSION

The SCION 8X00-GC analyzer is perfectly capable of analysing the boiling point distribution of the crude oil front end in compliance with IP601. The analyzer easily achieves the specifications mentioned in the standardized method.

If a full boiling point distribution is desired it is possible to merge this method with method IP545.

The equipment of the 8X00-GC analyzer is pre determined. Ordering information can be found on the next page. For 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.

## ORDER INFORMATION

Part number	8300 GC configuration
839941815	DHA Analyzer including 8410 for D7900, IP 344 or IP601 front-end, 8300 GC based, 120V Includes PC and CompassCDS and DHA Plug-in software.
839941833	DHA Analyzer including 8400 for D7900, IP 344 or IP601 front-end, 8300 GC based, 120V Includes PC and CompassCDS and DHA Plug-in software.
839941816	DHA Analyzer including 8410 for D7900, IP 344 or IP601 front-end, 8300 GC based, 230V Includes PC and CompassCDS and DHA Plug-in software.
839941834	DHA Analyzer including 8400 for D7900, IP 344 or IP601 front-end, 8300 GC based, 230V Includes PC and CompassCDS and DHA Plug-in software.
839941715	DHA Analyzer Includes 8410 for D7900, IP 344 or IP601 front-end, 8300 GC based, 120V Includes DHA plug in software CD. Excludes PC and CompassCDS.
839941733	DHA Analyzer Includes 8400 for D7900, IP 344 or IP601 front-end, 8300 GC based, 120V Includes DHA plug in software CD. Excludes PC and CompassCDS.
839941716	DHA Analyzer Includes 8410 for D7900, IP 344 or IP601 front-end, 8300 GC based, 230V Includes DHA plug in software CD. Excludes PC and CompassCDS.
839941734	DHA Analyzer Includes 8400 for D7900, IP 344 or IP601 front-end, 8300 GC based, 230V Includes DHA plug in software CD. Excludes PC and CompassCDS.
Part number	8500 GC configuration
859941815	DHA Analyzer Includes 8410 for D7900, IP 344 or IP601 front-end, 8500 GC based. 120V Includes PC and CompassCDS and DHA Plug-in software.
859941833	DHA Analyzer Includes 8400 for D7900, IP 344 or IP601 front-end, 8500 GC based. 120V Includes PC and CompassCDS and DHA Plug-in software.
859941816	DHA Analyzer Includes 8410 for D7900, IP 344 or IP601 front-end, 8500 GC based. 230V Includes PC and CompassCDS and DHA Plug-in software.
859941834	DHA Analyzer Includes 8400 for D7900, IP 344 or IP601 front-end, 8500 GC based. 230V Includes PC and CompassCDS and DHA Plug-in software.
859941715	DHA Analyzer Includes 8410 for D7900, IP 344 or IP601 front-end, 8500 GC based, 120V Includes DHA plug in software CD. Excludes PC and CompassCDS.
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859941734	DHA Analyzer Includes 8400 for D7900, IP 344 or IP601 front-end, 8500 GC based, 230V Includes DHA plug in software CD. Excludes PC and CompassCDS.
Part number	ASTM D7900/IP601 standards
<b>ST50000002</b>	ASTM D7900/IP 601 VALVE TIMING/LINEARITY CHECK STANDARD - 10X2 ML
<b>ST50000003</b>	ASTM D7900/IP601 C5-C14 STANDARD IN HEXADECANE - 10X2 ML

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