

# Application News

## Gas Chromatography

### Simulated Distillation Analysis of Heavy Canadian Crude Oil by ASTM D 5307

Simulated Distillation is a GC technique for determining the boiling point distribution of a crude oil stock. This information is useful to refinery engineers in determining the process that most efficiently utilizes a feedstock of raw crude oil. This technique is also used to monitor the refining process to insure the products produced are as expected. When a feedstock oil contains components that are larger than C44 and which boil above 538°C, ASTM D5307 becomes the analytical method of choice. This method requires that each sample be injected twice: the first time spiked with internal standard and the second time without. An internal standard calculation, outlined by the method, is used to compare the time-slice data of the two runs to determine an initial boiling point (IBP), a boiling distribution curve and the weight percent of residue that would boil between 538°C and 1000°C. This study was performed using bitumen samples obtained from the oil sands region of Canada.

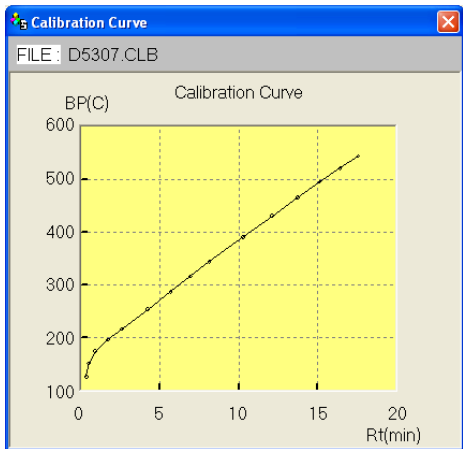
#### Analysis

Instrument: GC-2010 with a cool on-column injection port, flame ionization detector and GC oven cooling fan. The GC parameters are listed in the table below.

Column: Restek MTX-1 5M X 0.53mm ID X 0.1µf

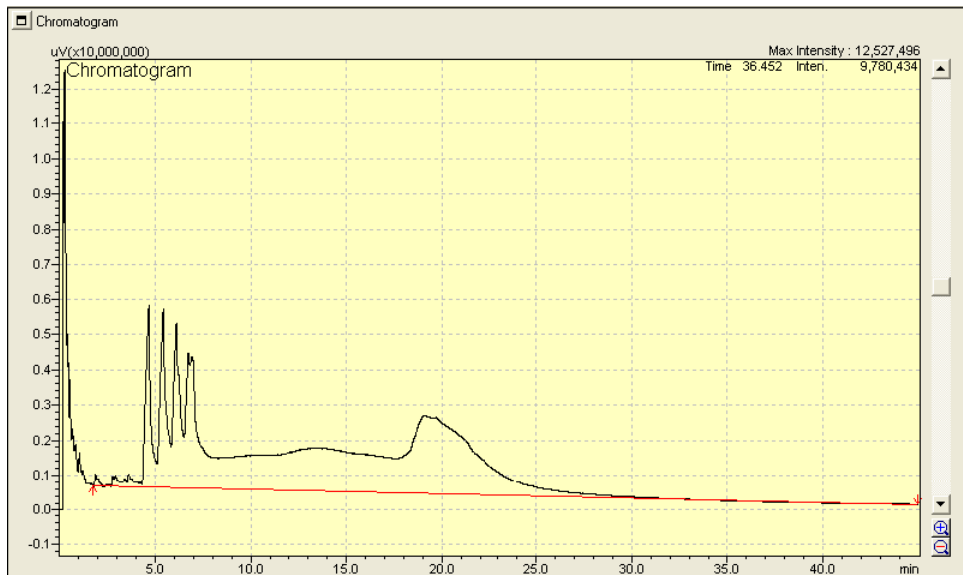
<b>Table 1: Recommended Instrument Parameters for Simulated Distillation</b>			
<b>Instrument Parameter</b>	<b>Petroleum Type</b>		
	<b>Kerosene</b>	<b>Diesel</b>	<b>Heavy Oil*</b>
Oven Temp Program	35°C - 250°C at 10°C/min	50°C - 300°C at 12°C/min	40°C - 310°C at 15°C/min hold 0 minutes 50 °C/min to 400 °C for 24 min.
OCI Temp Program	65°C hold 3 min, then 10°C/min to 250°C	65°C hold 1.5 min, then 12°C/min to 300°C	65°C hold 1 min, then 15°C/min to 330°C min hold 0 minutes 50 °C/min to 400 °C for 24 min.
Carrier Flow	3.5 mL/min	3.5 mL/min	3.5 mL/min
Injection Volume	0.2 - 0.5 µL	0.2 - 0.5 µL	0.5 - 1.0 µL
FID Temp	300°C	300°C	330°C
FID Makeup Flow	He at 30 mL/min	He at 30 mL/min	He at 30 mL/min
FID Hydrogen Flow	40 mL/min	40 mL/min	40 mL/min
FID Air Flow	400 mL/min	400 mL/min	400 mL/min
FID Sampling Rate	200 msec	200 msec	200 msec
Injection mode	Direct	Direct	Direct

A standard mixture of alkane compounds, usually up to C44, is analyzed on a thin film, non-polar GC column. The resultant retention times for each alkane component are plotted against their known boiling point temperatures (Figure 1).

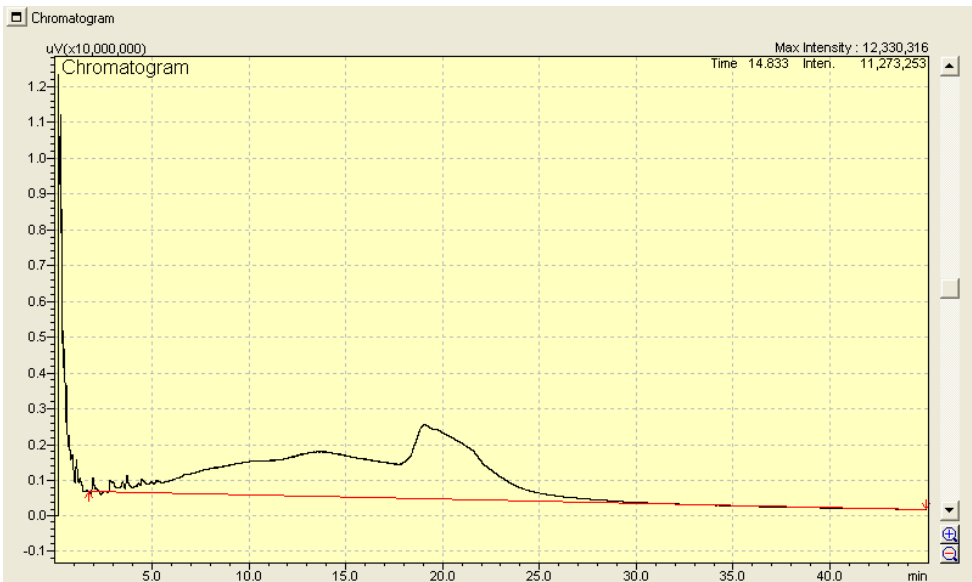


Then the crude oil sample is weighed and spiked with a mixture of four compounds: C14, C15, C16 and C17 (Figure 2). This sample is analyzed by the GC using the exact parameters employed in the standard run. A second sample is prepared like the first except without the internal standard spike. It is also analyzed on the gas chromatograph (Figure 3).

**Figure 1:** Retention Time vs Boiling Point Calibration Curve



**Figure 2:** Internal standard spiked crude bitumen



**Figure 3:** Crude bitumen non-spiked

## Data Processing and Report Generation

The chromatographic data produced in this analysis is integrated as a single peak. The peak area is divided up into time slices of 1°C increments and recorded, a standard feature in GCsolution chromatography software. Various calculations are performed using this data in post run by the Distillation GC software. The table below contains the integration parameters used in this study to create the time-slice data.

Time	Command
	HORIZ Baseline
1.7	On
1.7	Peak Detect
19.6	Peak Top
45	Peak End
45	Integration Off

The final calculation and report generation was accomplished using the Distillation GC software. This post-run software automatically executes as a user program from within the batch processing module of GCsolution. For method ASTM D5307 the following parameters were set in the Distillation GC parameter file to include the fraction from 538°C to 999°C. This temperature also defined the upper limit of analysis for ASTM D5307.

The screenshot shows the 'Distillation GC Initial Setting(GC 1)-[C:\GCsolution\ldgc1\parm\]' window. The 'Parameter File Setting' section is active, displaying the following configuration:

- FILE: D5307\_1000.PAR
- Method: Internal Standard (Differential)
- ASTM Correction:  D-88,  D-1180,  EFV/TBP Correction
- Terminus Automatic Detection:  Variety: 0.010, Proportion(%): 0.010
- Internal Standard Range (C):
 

IBP	FBP	Upper Limit Temperature
240.0	310.0	860.0
- Number of Specified Temperature Range: 2 (C)

The 'Number of Specified Temperature Range' section contains the following table:

Range	IBP	FBP	Range	IBP	FBP
1	0.0	538.0	6		
2	538.0	999.9	7		
3			8		
4			9		
5			10		

## Results

The final report consists of three tables: Table 1 details the two specified fractions with their % distillation. Table 2 is the resulting distillation curve plot and Table 3 is the detailed Simulated Distillation results table starting at the initial boiling point (IBP) and listing % distilled in 1°C increments up thru the final boiling point.

### \*\*\*\* BOILING RANGE DISTRIBUTION \*\*\*\*

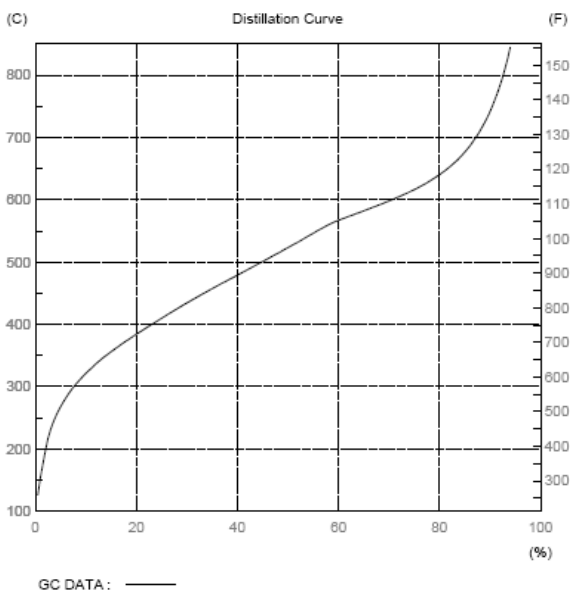
SAMPLE NAME : 1247-61-f  
 SAMPLE ID : 1247-61-f  
 SAMPLE TYPE : UNKNOWN  
 ANALYSIS TIME : 2008/05/09 17:56:47  
 TOTAL AREA : INTERNAL STANDARD (DIFFERENTIAL)  
 SAMPLE WEIGHT : 0.9938  
 IS WEIGHT : 0.1035  
 OUTPUT FILE : C:\GCsolution\dgc1\data\1247-61-f.DAT  
 CALIBRATION FILE : C:\GCsolution\dgc1\Calib\ID5307.CLB  
 PARAMETER DATA FILE : C:\GCsolution\dgc1\PARM\ID5307.PAR  
 REPORT DATA FILE : C:\GCsolution\dgc1\PARM\ID5307.REP

### Distillation Data in Specified Temperature Range

IBP (C)	FBP (C)	%
0.0	538.0	53.2
539.0	880.0	40.8

### \*\*\*\* BOILING RANGE DISTRIBUTION \*\*\*\*

SAMPLE NAME : 1247-61-f  
 SAMPLE ID : 1247-61-f  
 SAMPLE TYPE : UNKNOWN  
 ANALYSIS TIME : 2008/05/09 17:56:47  
 TOTAL AREA : INTERNAL STANDARD (DIFFERENTIAL)  
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 PARAMETER DATA FILE : C:\GCsolution\dgc1\PARM\ID5307.PAR  
 REPORT DATA FILE : C:\GCsolution\dgc1\PARM\ID5307.REP



SimDist GC Package 220-90863-20 includes:

- GC-2010 with FID and Temperature-programmable On-Column Injection Port
- Column: Restek MTX-1 5M X 0.53mm ID X 0.1µf
- GCsolution and Distillation GC software

GC Oven Fast Cooling Fan (221-47749-91), Cryogenic Cooling (LN2 221-49588-91), and Autosampler are sold separately.

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 CALIBRATION FILE : C:\GCsolution\dgc1\Calib\ID5307.CLB  
 PARAMETER DATA FILE : C:\GCsolution\dgc1\PARM\ID5307.PAR  
 REPORT DATA FILE : C:\GCsolution\dgc1\PARM\ID5307.REP

### GC DATA

%OFF (%)	BP (C)	BP (F)	Rt (min)
IBP	125.8	258.4	0.29
1	153.4	308.1	0.49
2	198.5	389.4	1.78
3	230.4	446.8	3.19
4	251.8	485.2	4.11
5	267.9	514.3	4.82
6	281.6	538.8	5.42
7	293.5	560.2	5.94
8	303.7	578.7	6.39
9	312.8	595.0	6.78
10	321.2	610.2	7.14
11	329.1	624.4	7.48
12	336.6	637.8	7.80
13	343.6	650.6	8.10
14	350.0	662.0	8.40
15	356.1	673.0	8.68
16	362.1	683.7	8.95
17	367.8	694.0	9.22
18	373.4	704.1	9.48
19	378.8	713.9	9.73
20	384.2	723.5	9.97
21	389.4	732.9	10.22
22	394.5	742.2	10.45
23	399.7	751.4	10.69
24	404.8	760.6	10.92
25	409.8	769.7	11.16
26	414.8	778.7	11.39
27	419.9	787.7	11.62
28	424.7	796.5	11.84
29	429.6	805.3	12.07
30	434.4	813.9	12.29
31	439.1	822.4	12.50
32	443.7	830.7	12.72
33	448.3	839.0	12.93
34	452.8	847.1	13.13
35	457.3	855.1	13.34
36	461.7	863.1	13.54
37	466.1	871.0	13.75
38	470.4	878.7	13.95
39	474.7	886.4	14.15
40	479.0	894.1	14.35
41	483.3	901.9	14.56
42	487.7	909.8	14.77
43	492.1	917.7	14.97
44	496.5	925.6	15.19
45	500.9	933.6	15.40
46	505.2	941.4	15.62
47	509.7	949.4	15.83
48	514.1	957.4	16.05
49	518.6	965.5	16.27
50	523.1	973.6	16.50
51	527.6	981.7	16.72
52	532.2	989.0	16.95
53	536.8	996.3	17.19
54	541.5	1006.8	17.43
55	546.3	1015.3	17.66
56	551.0	1023.7	17.90
57	555.5	1031.9	18.13
58	559.7	1039.5	18.34
59	563.8	1046.5	18.54
60	567.0	1052.6	18.71
61	570.1	1058.1	18.86
62	573.1	1063.5	19.02
63	576.1	1068.9	19.17
64	579.1	1074.5	19.32
65	582.3	1080.1	19.48
66	585.4	1085.6	19.64
67	588.4	1091.2	19.79
68	591.6	1096.8	19.95
69	594.8	1102.6	20.11
70	598.1	1108.5	20.28
71	601.5	1114.6	20.45
72	605.0	1120.9	20.62
73	608.6	1127.5	20.81
74	612.3	1134.2	21.00
75	616.3	1141.3	21.19
76	620.4	1148.7	21.40
77	624.9	1156.8	21.63
78	629.7	1165.4	21.87
79	634.8	1174.6	22.13
80	640.3	1184.5	22.40
81	646.2	1195.1	22.70
82	652.6	1206.8	23.03
83	659.8	1219.8	23.39
84	667.7	1233.8	23.79
85	676.5	1249.7	24.23
86	686.4	1267.5	24.73
87	697.6	1287.7	25.30
88	710.3	1310.5	25.94
89	724.9	1336.8	26.67
90	741.6	1366.9	27.52
91	761.2	1402.2	28.50
92	784.1	1443.4	29.66
93	811.4	1492.5	31.04
94	845.1	1553.1	32.73

VABP ——— 523.687 (C) ——— 974.637 (F)