Simulated Distillation of a Heavy Gasoil and FCC Feed according to IP 480

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Introduction

The IP480 standard specifies a method for the determination of the boiling range distribution of petroleum products by capillary gas chromatography using flame ionization detection. The standard is applicable to materials having a vapor pressure low enough to permit sampling at ambient temperature and a boiling range of at least 100 °C. The standard is applicable to distillates with initial boiling points (IBP) above 100 °C and final boiling points (FBP) below 750 °C, for example, middle distillates and lubricating base stocks.

Instrumentation

Technique: Varian 450-GC Gas Chromatograph Simulated Distillation Analyzer
Injector: Temperature controlled on-column (1093) with full EFC control
Detection: High temperature FID with full EFC control
Autosampler: Varian CP-8410 AutoSampler (or Varian CP-8400 AutoInjector)

Software
GC Control and Data Handling: Galaxie™ Software
Simulated Distillation Calculations: SimDist plug-in software fully integrated into Galaxie Workstations

Materials and Reagents

Sample: Crude oil
Column: Varian CP-SimDist UltiMetal™, 5 m x 0.53 mm x 0.09 µm (pn: CP7569)
Calibration: Mixture of n-paraffins (approx 1 %) dissolved in carbon disulfide
Internal Standard: ASTM D 5307 Crude Oil Int. St.

Sample Preparation

The calibration mix was prepared by dissolving 0.1 g polywax 1000 in 7 mL CS_2 and adding 10 µL of an equal volume mixture of n-alkanes, all according to the method. The samples were obtained by making a 2 to 3 % (m/v) solution in CS_2.

Conditions

Sample Size: 1 µL
Carrier Gas: Helium, 19 mL/min
Oven Program: 35 °C @ 10 °C/min to 430 °C
Injector Program: 100 °C @ 15 °C/min to 430 °C
Detection: 450 °C

Results and Discussion

The software calculates the IBP and FBP of the samples using the retention times of n-alkanes in the calibration mix.

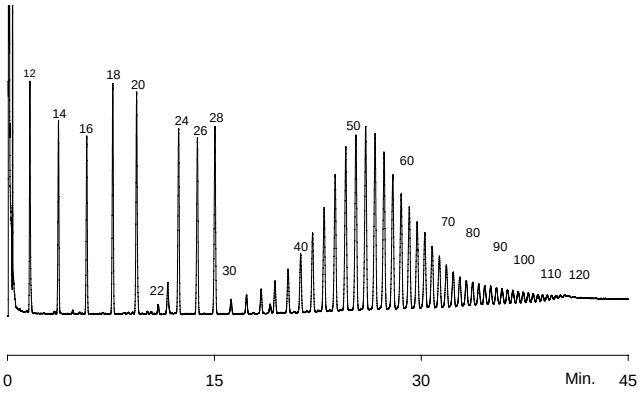

Figure 1. Polywax 1000 spiked with n-alkanes, calibration mix.

The same conditions were used to analyze the samples. A CS_2 blank was used for baseline subtraction. It is important to note that in spite of the fact that a temperature stable, ultra low bleed, UltiMetal column was used, some low level column bleed will always be present and will influence analysis results if no baseline subtraction is employed.

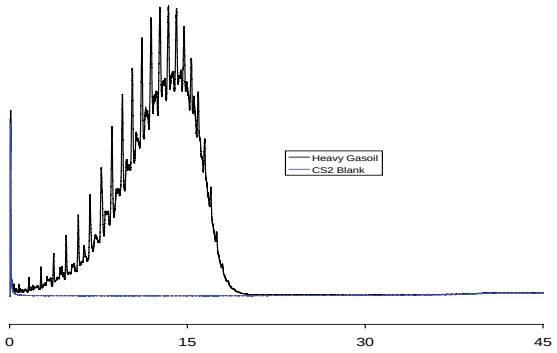

Figure 2. Heavy gasoil and CS_2 blank.

Table 1. Reproducibility values of heavy gasoil analysis.

% Off Report

% Off	File 1 °C	File 2 °C	File 3 °C	File 4 °C	File 5 °C	Average °C	St.dev. °C
IBP	239.8	239.0	239.6	239.2	239.7	239.46	0.344
1%	254.3	254.0	254.1	254.1	254.1	254.12	0.110
5%	297.0	296.7	297.0	296.8	297.1	296.92	0.164
10%	320.2	320.0	320.2	320.1	320.3	320.16	0.114
15%	336.7	336.6	336.7	336.6	336.7	336.66	0.055
20%	348.7	348.7	348.8	348.7	348.8	348.74	0.055
25%	359.6	359.7	359.6	359.6	359.7	359.64	0.055
30%	368.7	368.8	368.8	368.7	368.9	368.78	0.084
35%	376.0	376.1	376.0	376.0	376.1	376.04	0.055
40%	383.2	383.3	383.2	383.2	383.3	383.24	0.055
45%	389.4	389.5	389.4	389.4	389.5	389.44	0.055
50%	395.6	395.8	395.7	395.6	395.8	395.70	0.100
55%	402.0	402.2	402.0	402.0	402.1	402.06	0.089
60%	407.9	408.1	407.9	407.9	408.0	407.96	0.089
65%	414.2	414.4	414.2	414.2	414.3	414.26	0.089
70%	419.8	420.0	419.8	419.7	419.9	419.84	0.114
75%	425.8	426.0	425.7	425.7	425.9	425.82	0.130
80%	431.9	432.1	431.8	431.8	432.0	431.92	0.130
85%	438.7	438.9	438.6	438.6	438.8	438.72	0.130
90%	446.5	446.6	446.3	446.4	446.5	446.46	0.114
95%	457.0	456.9	456.6	456.6	456.7	456.76	0.182
99%	473.4	472.7	472.3	472.3	472.4	472.62	0.466
FBP	478.8	477.7	477.3	477.3	477.4	477.70	0.636

A FCC feed can be analyzed in the same way. In this case, a CS_2 blank is used for baseline subtraction.

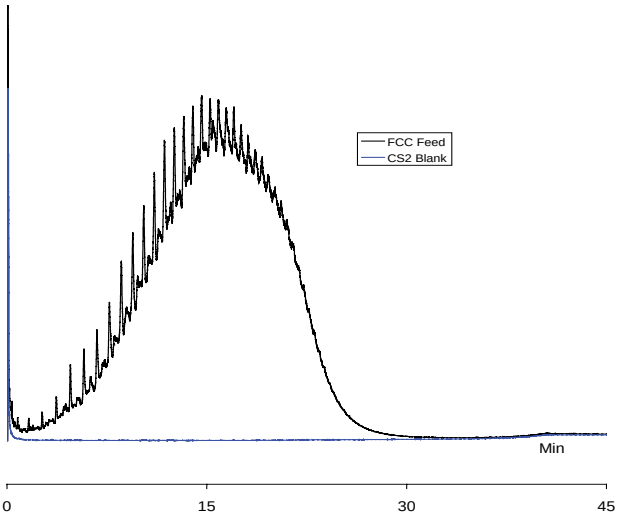

Figure 3. FCC feed and CS_2 blank.

Table 2. Reproducibility values of FCC feed analysis.

% Off Report % Off	File 1 °C	File 2 °C	File 3 °C	File 4 °C	File 5 °C	Average °C	St.dev. °C
IBP	255.0	255.0	255.9	255.6	255.6	255.4	0.402
1%	269.5	269.5	270.2	270.1	270.2	269.9	0.367
5%	315.9	315.9	316.3	316.3	316.3	316.1	0.219
10%	343.6	343.6	344.0	344.0	344.1	343.9	0.241
15%	362.7	362.7	363.1	363.1	363.2	363.0	0.241
20%	377.8	377.8	378.2	378.1	378.2	378.0	0.205
25%	390.4	390.4	390.7	390.6	390.7	390.6	0.152
26%	392.8	392.8	393.0	393.0	393.1	392.9	0.134
27%	394.9	394.9	395.2	395.1	395.2	395.1	0.152
28%	397.4	397.4	397.7	397.6	397.7	397.6	0.152
29%	399.9	399.8	400.1	400.0	400.2	400.0	0.158
30%	402.3	402.3	402.6	402.5	402.7	402.5	0.179
35%	413.2	413.2	413.4	413.4	413.5	413.3	0.134
40%	423.2	423.1	423.3	423.3	423.4	423.3	0.114
45%	432.8	432.7	432.9	432.8	433.0	432.8	0.114
50%	442.3	442.3	442.4	442.3	442.5	442.4	0.089
55%	451.8	451.8	451.9	451.8	452.0	451.9	0.089
60%	461.3	461.2	461.4	461.3	461.5	461.3	0.114
65%	470.8	470.8	470.9	470.8	471.0	470.9	0.089
70%	480.5	480.4	480.5	480.4	480.6	480.5	0.084
75%	491.0	491.0	491.0	491.0	491.2	491.0	0.089
80%	501.5	501.5	501.4	501.4	501.6	501.5	0.084
85%	512.6	512.6	512.5	512.5	512.7	512.6	0.084
90%	525.7	525.7	525.6	525.6	525.8	525.7	0.084
95%	542.6	542.6	542.4	542.4	542.5	542.5	0.100
99%	573.0	572.9	572.4	572.4	572.5	572.6	0.288
FBP	584.1	584.0	583.2	583.2	583.2	583.5	0.467

Conclusion

The chromatograms from the calibration mix for both heavy gasoil and FCC feed provide firm evidence of the solid performance of the Varian Simulated Distillation Analyzer The reproducibility values shown are consistent with those prescribed in the IP 480 method.

Reference

IP 480, 2007 "Determination of boiling range distribution by gas chromatography method - Part 1: Middle distillates and lubricating base oils," Energy Institute, London, UK.

These data represent typical results.
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