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Application Note SI-01294

Simulated Distillation of Crude Oil According to ASTM D 5307

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Introduction

This note describes the determination of the boiling range distribution of water-free crude petroleum through 538 °C (1000 °F). Material boiling above 538 °C is reported as residue.

Instrumentation

Varian 450-GC Gas Chromatograph Simulated Distillation Analyzer

Injector: Temperature controlled on-column (Varian 1093 Injector) with full EFC control

Detector: High temperature FID with full EFC control

Autosampler: Varian CP-8400 AutoSampler

Software

GC control and data handling: Galaxie™ Software from Varian

Simulated distillation calculations: SimDist plug-in software fully integrated into Galaxie software

Materials and Reagents

Column: Varian CP-SimDist UltiMetal™, 10 m x 0.53 mm x 0.88 μm (CP7512)

Sample: Crude oil

Calibration: Mixture of n-paraffins (approx. 1%) dissolved in carbon disulfide

Internal Standard: ASTM D 5307 Crude Oil Int. St.

Sample Preparation

Weigh at least 10 g of a water free crude oil, to the nearest 0.1 mg, into a 25 mL vial. Add approximately 1 g of internal standard mixture into the same vial. Determine the weight to the nearest 0.1 mg. Dilute the mixture with an approximately equal volume of carbon disulfide. Cap the vial tightly and shake vigorously for 2 min or until the mixture is solubilized completely. In a second vial, dissolve approximately the same amount of dried sample with an approximately equal volume of carbon disulfide. Use this solution for the separate crude oil without internal standard analysis.

Conditions

Injector: On-column, 100 °C @ 15 °C/min to 350 °C

Detector: 350 °C

Carrier Gas: Helium, 20 mL/min

Oven: 35 °C @ 10 °C/min to 350 °C

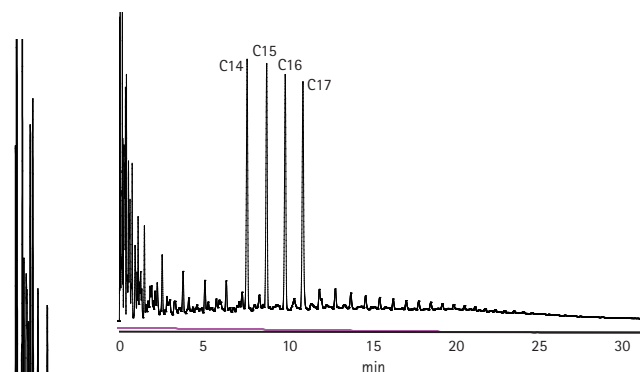


Figure 2. Crude oil plus internal standard.

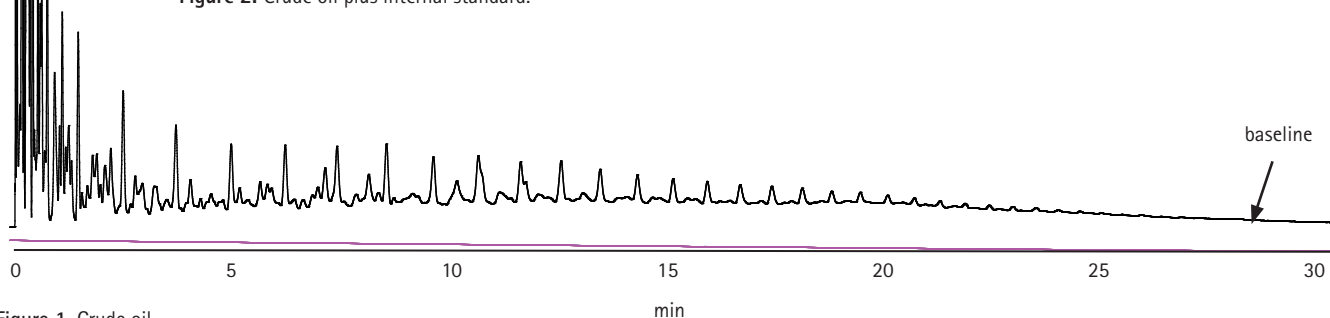


Figure 1. Crude oil.

Results and Discussion

The original column specified for use with ASTM D 5307 is a packed column. The method, however, permits the use of other columns. In this case a wide bore capillary column was chosen. In order to achieve the proper settings for boiling point distribution calculations a calibration mixture containing n-paraffins was analyzed (Figure 3). From this calibration mixture a calibration curve was derived (Figure 4).

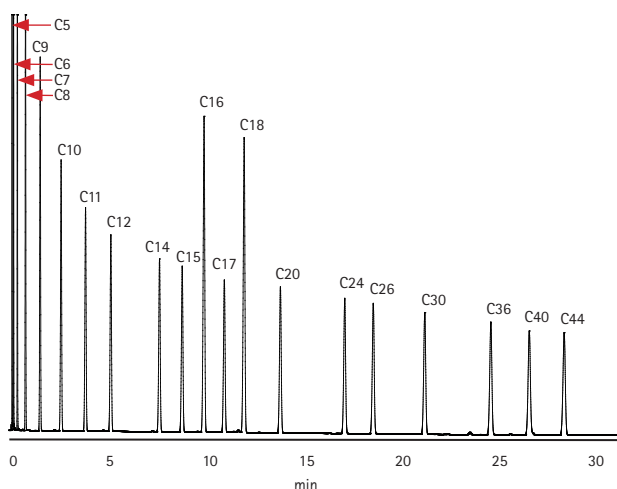


Figure 3. Calibration mixture.

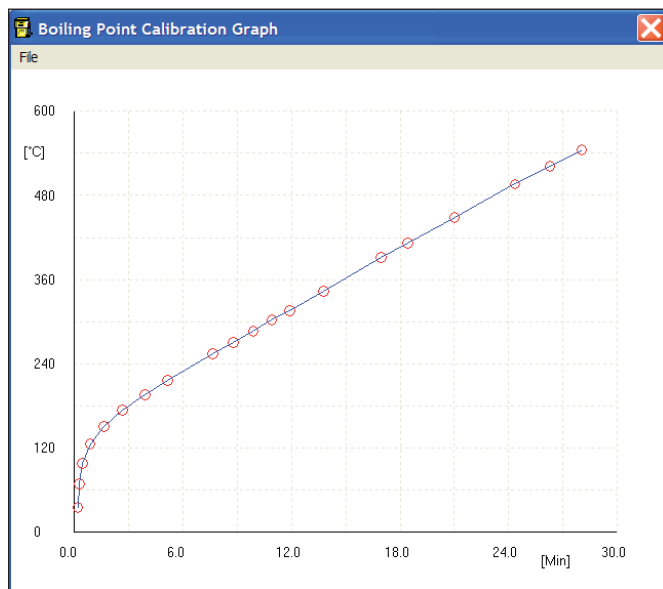


Figure 4. Calibration curve.

The boiling points of the n-paraffins were plotted against the elution time. A "smooth" line could be drawn through the various data points indicating that the system was properly calibrated. Since the recovery of crude oil will be less than 100% two sample injections were made (Figures 1 and 2). Figure 2 shows the chromatogram with an added internal standard to calculate the actual sample recovery, and corrected for the results shown in Figure 1. The SimDist software for Galaxie™ software produces a summary report (Table 1). The SimDist report shows the initial boiling point (IBP), the % off temperatures as well as the D1160 °C profile. The % recovery value is also listed.

Table 1. SimDist analysis report of crude oil

SimDist 450-GC		Crude oil 1% in CS2		Vial		Date Reported	
Description		Crude oil 1% in CS2		3		24 Sep 2007, 10:46:06	
Additional Type		Sample		3		24 Sep 2007, 10:46:06	
Raw Data File		104052_astmd5307_29_08_2007_3_19_18_pm_4.dat		3		24 Sep 2007, 10:46:06	
Instrument		450-GC		3		24 Sep 2007, 10:46:06	
Vial		450-GC		3		24 Sep 2007, 10:46:06	
Date Reported		24 Sep 2007, 10:46:06		3		24 Sep 2007, 10:46:06	
% Off Report							
% Off	°C	D1160 °C	% Off	°C	D1160 °C	% Off	°C
IBP	28.6	368.8	21%	236.2		42%	384.2
1%	33.4		22%	245.9		43%	391.5
2%	72.6		23%	252.6		44%	399.5
3%	85.3		24%	259.6		45%	406.8
4%	94.5		25%	266.6		46%	414.1
5%	100.5	90.9	26%	272.5		47%	421.4
6%	106.7		27%	280.3		48%	428.8
7%	115.7		28%	286.9		49%	436
8%	125.9		29%	294.5		50%	443.5
9%	132.9		30%	301.4	348.7	51%	451.1
10%	140.1	173.9	31%	307.6		52%	459.1
11%	150.5		32%	314.5		53%	467.5
12%	158.8		33%	320.2		54%	476.4
13%	166.8		34%	327.8		55%	485.7
14%	174.5		35%	334		56%	495.2
15%	185.6		36%	341.6		57%	504.9
16%	195.3		37%	348.2		58%	515.2
17%	203.8		38%	355.3		59%	526.1
18%	214.1		39%	362.4		60%	537.6
19%	221.1		40%	369.2	401.1	Recovery 60.6% at 545°C	
20%	229.4	268.3	41%	377		Residue 39.4%	

Conclusion

The Varian 450-GC based simulated distillation analyzer is well suited for use in determining the boiling point distribution of crude oil according to ASTM D 5307. All facets of this analyzer, including the Varian 450-GC gas chromatograph, the Varian CP-SimDist UltiMetal™ column and Galaxie software GC control and data handling software extended with a SimDist plug-in, are fully integrated. This approach ensures trouble free operation, easy data handling and reporting, all according to the method.

Reference

ASTM D 5307-97(2007), "Determination of Boiling Range Distribution of Crude Petroleum by Gas Chromatography" ASTM International, West Conshohocken, PA, www.astm.org.

These data represent typical results.
For further information, contact your local Varian Sales Office.

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