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Application News

GC Nexis™GC-2030

Simulated Distillation by High Temperature Gas Chromatography Based on ASTM D7500

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User Benefits

- The boiling range distribution of base oils and lubricating base stocks can be determined based on ASTM D7500.
- LabSolutions™ SIMDIS GC Software supports data acquisition, SIMDIS data processing, and report preparation.
- The i-PeakFinder function makes it simpler to determine integration parameters to calculate peak skewness.

■ Introduction

Petroleum products such as gasoline, diesel oil, lubricating oil, and asphalt are crucial to support our everyday lives. These materials are the complex of several hydrocarbons, and their properties vary depending on their composition. By investigating the distillation characteristics, it is possible to make more effective use of them, control quality, and determine pricing.

ASTM D7500 is a standard test method for determining the boiling range distribution of petroleum products by simulated distillation using capillary gas chromatography and a flame ionization detector (FID). This applies to distillates with an initial boiling point (IBP) of 100 °C or higher and a final boiling point (FBP) of 735 °C (the boiling point of carbon 110) or lower, such as base oil and lubricating base stocks. The injected sample must elute completely from the gas chromatographic column, and the distillation curve is obtained using the total area of the sample chromatogram.

In this article, we introduced an example of the analysis of lubricating oil based on ASTM D7500 with the new SHIMADZU OCI-2030 NX injection unit.

■ Sample Preparation

Samples were prepared as follows. Carbon disulfide was used for the washing solvent.

(1) Calibration Mix

Polywax 655, including high boiling point compounds, was diluted to 0.5% w/w with cyclohexane. Then a mixture of equal amounts of C5-10, C12, C14, C16, C18, C20, C24, C26, C28 was added (referred to below as the Polywax 655 Standard). For system performance check of low boiling point compounds, n-Alkanes Standard was prepared by diluting C8-C40 Alkanes Calibration Standard (P/N: 40147-U) to 100ppm with cyclohexane.

(2) Reference Oil Sample

Reference Material 5010 (P/N: SD-020-02) was diluted to 1% w/v with carbon disulfide.

(3) Lubricating Oil Sample

Commercially available lubricating oil was diluted to 1% w/w with carbon disulfide.

■ Instruments Used

Table 1 shows the instruments used in this analysis.

Table 1 List of Instruments

Model	:	Nexis GC-2030
Auto Injector	:	AOC™-20i Plus
Injection Port	:	OCI-2030 NX
Detector	:	FID-2030 0.5φ Nozzle (P/N: 221-75597-05)
Column	:	Ultra ALLOY-SIMDIS(HT) (5 m × 0.53 mm I.D., df = 0.1 µm) (P/N: UASIM (HT)-5W-0.1F)
Syringe	:	OCI Syringe (P/N: 221-37282-02)

■ Conditions

The operating conditions for this analysis are shown in Table 2.

Table 2 Typical Operating Conditions Based on ASTM D7500

Injector Temperature Program	: 100 °C →(15 °C/min)→ 430 °C
Equilibration Time	: 10 min
Column Oven Temperature Program	: 35 °C →(10 °C/min)→430 °C (10 min)
FID Temperature	: 450 °C
Makeup Gas	: He (24 mL/min)
Air Flow	: 200 mL/min
H_2 Flow	: 32 mL/min
Carrier Gas	: He, Constant Flow (19 mL/min)
Injection Volume	: 0.6 µL

■ System Performance Check

Two types of calibration mix were prepared and analyzed, and the system performance was checked based on the results. Fig. 1 shows each of the chromatograms.

The column resolution of C50 and C52 peaks was 2.5, which met the standard value of 2 to 4. The skewness of all peaks from C12 to C110 was between the standard value of 0.8 to 1.8. Skewness is converted from the tailing factor. The standard value converted into tailing factor is from 0.778 to 1.125. An example of the tailing factors obtained and the skewness is shown in Table 3. The chromatogram of n-Alkenes Standard was used to check the skewness of C12-C30 and the Polywax 655 Standard for C32-C110.

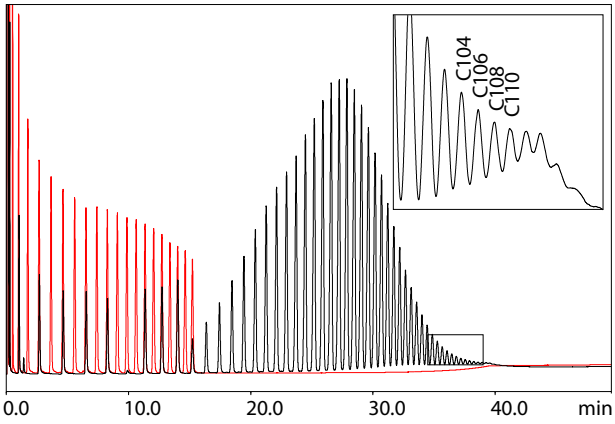

Fig. 1 Typical Chromatograms of Calibration Mixes and Enlarged View of the Peaks around C110
(Red: n-Alkanes Standard, Black: Polywax 655 Standard)

Table 3 Examples of Boiling Points (b.p.), Tailing Factor, and Skewness Obtained by Results of Calibration Mix Analyses

Carbon No.	b.p. (°C)	Tailing Factor	Skewness	Carbon No.	b.p. (°C)	Tailing Factor	Skewness
16	287	1.01	0.99	60	615	0.80	1.68
18	316	1.00	1.00	62	622	0.81	1.62
20	344	0.98	1.05	64	629	0.81	1.61
22	369	0.98	1.04	66	635	0.83	1.53
24	391	0.98	1.04	68	641	0.84	1.48
26	412	0.99	1.02	70	647	0.85	1.42
28	431	0.98	1.03	72	653	0.86	1.38
30	449	0.99	1.03	74	658	0.87	1.35
32	466	1.07	0.88	76	664	0.89	1.29
34	481	1.04	0.92	78	670	0.92	1.20
36	496	0.97	1.05	80	675	0.91	1.22
38	509	0.94	1.13	82	681	0.93	1.16
40	522	0.91	1.21	84	686	0.95	1.11
42	534	0.90	1.26	86	691	0.99	1.03
44	545	0.87	1.35	88	695	0.99	1.02
46	556	0.84	1.46	90	700	0.97	1.07
48	566	0.85	1.43	92	704	1.02	0.95
50	575	0.83	1.51	94	708	0.98	1.05
52	584	0.82	1.54	96	712	1.00	1.00
54	592	0.81	1.61	98	716	1.03	0.94
56	600	0.80	1.69	100	720	1.03	0.94
58	608	0.79	1.75	110	735	0.92	1.18

■ Use of i-PeakFinder

i-PeakFinder, a peak integration algorithm included in SHIMADZU LabSolutions, helps to process the even complex chromatogram easily. To calculate the peak skewness, even slight peaks of a Polywax 655 Standard chromatogram need be detected, although its baseline drifts a lot.

By employing i-PeakFinder, detecting the slight peaks and processing the baseline is much easier, as shown in Fig. 2. i-PeakFinder saves the inconvenience of determining integration parameters to calculate skewness.

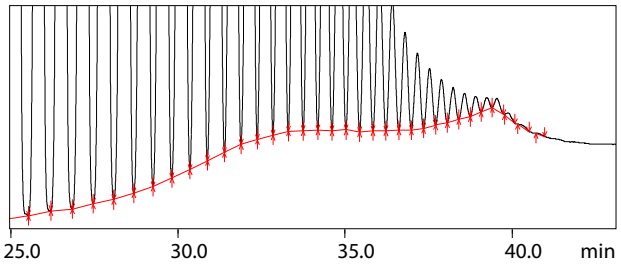

Fig. 2 Typical Polywax 655 Chromatogram Processed Using i-PeakFinder

Note: The results obtained using i-PeakFinder, as shown in Fig. 2, are required not to determine the boiling range distribution but to calculate the calibration curve, column resolutions, and skewness. To calculate the boiling range distribution, the sliced area is employed.

■ Calibration Curve of Retention Time versus Boiling Point

Fig. 3 shows the calibration curve of retention time versus boiling point obtained from the results of the Calibration Mix analyses shown in Table 3.

This calibration curve can be easily generated using LabSolutions SIMDIS GC software.

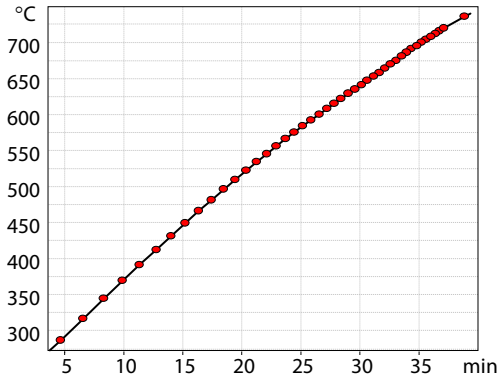

Fig. 3 Typical Calibration Curve of Retention Time versus Boiling Point

■ Reference Oil Sample Analysis

Fig. 4 and Table 4 show the chromatogram and the results of reference oil check results, respectively. It was confirmed that to determine the boiling range distribution using this analysis system met enough repeatability tolerances.

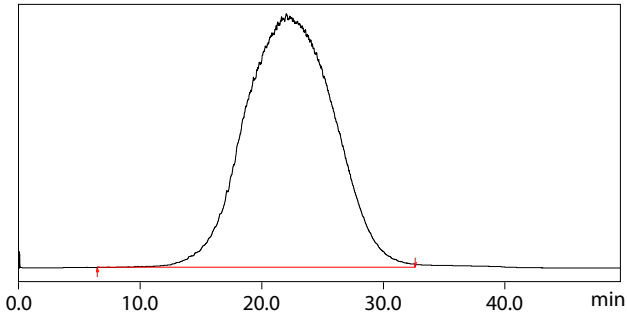

Fig. 4 Typical Chromatogram of Reference Oil Sample

Table 4 Example of the Reference Oil Check Results

Recovered Mass %	Analysis b.p. (°C)	Accepted b.p. (Reference Material 5010) (°C)	Deviation (°C)	Allowed Deviation (°C)
IBP	429.3	428	1.3	±9
5%	476.5	477	-0.5	±3
10%	492.2	493	-0.8	±3
15%	502.1	502	0.1	±3
20%	510.2	510	0.2	±3
25%	517.7	518	-0.3	±4
30%	524.6	524	0.6	±4
35%	531	531	0	±4
40%	536.9	537	-0.1	±4
45%	542.6	543	-0.4	±4
50%	548.4	548	0.4	±5
55%	554.3	554	0.3	±4
60%	560.1	560	0.1	±4
65%	566	566	0	±4
70%	571.7	572	-0.3	±4
75%	577.8	578	-0.2	±5
80%	584.5	585	-0.5	±4
85%	591.7	593	-1.3	±4
90%	600.7	602	-1.3	±4
95%	613.7	616	-2.3	±4
FBP	648.7	655	-6.3	±18

■ Sample Analysis

Lubricating Oil Sample was analyzed using this test method. The chromatogram is shown in Fig. 5, and the distillation curve is shown in Fig. 6. This curve was also generated with LabSolutions SIMDIS GC Software.

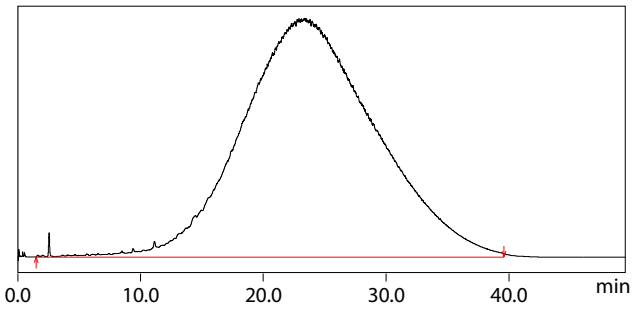

Fig. 5 Typical Chromatogram of Lubricating Oil Sample

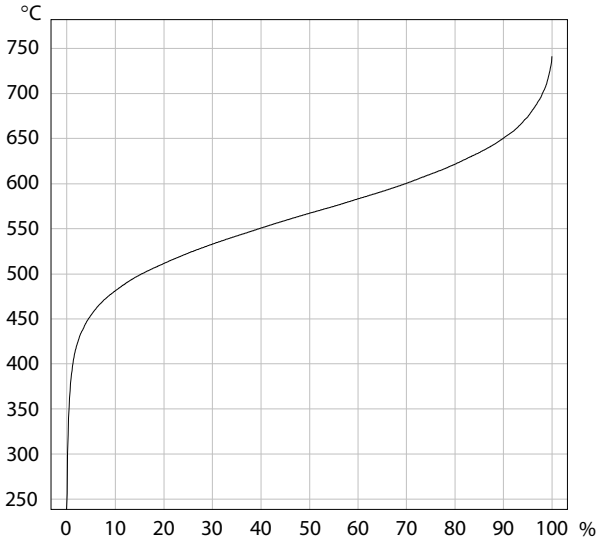

Fig. 6 Typical Distillation Curve of Lubricating Oil Sample

■ LabSolutions SIMDIS GC Software

LabSolutions SIMDIS GC Software was used for these analyses and calculations. It has many practical functions, such as creating calibration curves and distillation curves, as described above. In addition, this software provides a variety of report formats. For example, reports like the one shown in Fig. 7 can be easily output. The reference oil check results also can be converted to a report as shown in Fig. 8 below.

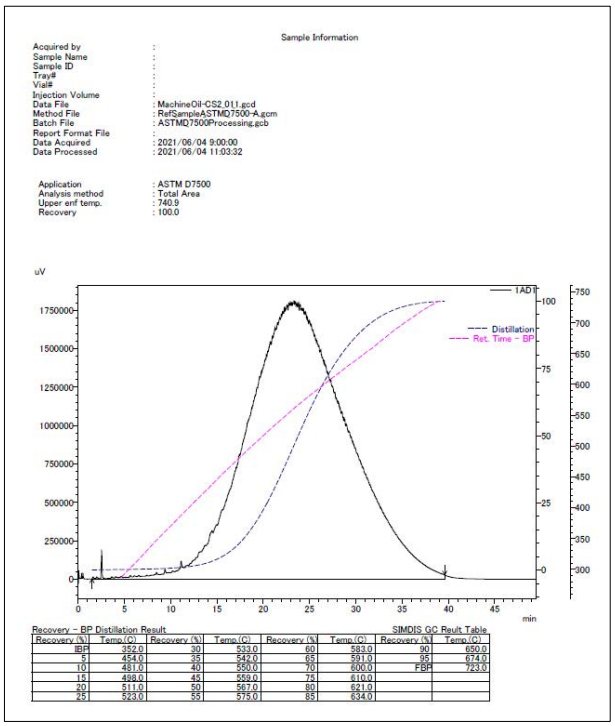

Fig. 7 Example of Report Format Output by LabSolutions GC Software

■ Precautions

Some Calibration Mixes and Lubricating Oil Samples used in this SIMDIS analysis were heated as necessary to correctly introduce them into the gas chromatograph. Please ensure operator safety and take care to prevent the solvent from igniting.

Note that samples that have been heated to soften them re-solidify easily and may fail to enter the gas chromatograph, which affects reproducibly. Please pay attention to the changes of the boiling range distribution due to the re-solidification.

■ Conclusion

Lubricating oil was analyzed according to the ASTM D7500 standard using Nexis GC-2030 with the new SHIMADZU OCI-2030 NX injection unit. System performance and the reference oil repeatability was checked, and those results met the standard values.

The analyses were performed using LabSolutions SIMDIS GC Software. This software comes with many practical functions to support operators and facilitate the creation of reports from the results.

Recovery – BP Distillation Result / SIMDIS GC Result Table

Recovery (%)	Temp.(C)	BP Criteria	Threshold	Check	Recovery (%)	Temp.(C)	BP Criteria	Threshold	Check
IBP	429.0	428	9	Pass	55	554.0	554	4	Pass
5	477.0	477	3	Pass	60	560.0	560	4	Pass
10	492.0	493	3	Pass	65	566.0	566	4	Pass
15	502.0	502	3	Pass	70	572.0	572	4	Pass
20	510.0	510	3	Pass	75	578.0	578	5	Pass
25	518.0	518	4	Pass	80	585.0	585	4	Pass
30	525.0	524	4	Pass	85	592.0	593	4	Pass
35	531.0	531	4	Pass	90	601.0	602	4	Pass
40	537.0	537	4	Pass	95	614.0	616	4	Pass
45	543.0	543	4	Pass	FBP	649.0	655	18	Pass
50	548.0	548	5	Pass					

Fig. 8 Example of Reference Oil Check Output by LabSolutions SIMDIS GC Software

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