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Application News

No.G312A

Gas Chromatography

Testing the Gasoline Dilution Rate of Gasoline in Engine Oil in Accordance with ASTM D3525 and Japan Petroleum Institute Standard JPI-5S-24

If gasoline or diesel mixes into the engine oil, it decreases the oil viscosity and prevents achieving the proper performance as a lubricating oil. Measuring the fuel dilution rate serves as a key indicator during oil replacement, because it can determine the degradation status of engine oil.
The test methods used to measure the fuel dilution rate are specified in standards such as U.S. ASTM D3524, D3525, and D7593. The standard for gasoline is ASTM D3525. It corresponds to the Japan Petroleum Institute standard JPI-5S-24 in Japan. This article describes an example of measuring the dilution rate of gasoline in engine oil in accordance with ASTM and JPI standards.

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Preparing Samples

20 µL of the internal standard substance n-C_{14}*1 was added to a 1 g sample of engine oil that contains gasoline. Mixture samples for measuring the column resolution were prepared by adding 1 % each, by volume, of n-C_{14} and n-C_{16}*3 to n-C_8*2.
Each sample was analyzed without dilution with a solvent.

Analytical Conditions

Analytical conditions specified based on respective standards are indicated in Table 1.

Table 1 Analytical Conditions

Model	: Nexis™ GC-2030 AF/AOC-20i
Column	: SH-1 (5 m × 0.53 mm I.D., df = 1.00 µm), 3 pcs sets *4
Column Temp.	: 50 °C (0 min) - 45 °C/min - 290 °C (10 min) Total: 5.33 min
Injection Temp.	: 255 °C
Carrier Gas	: N_2, 15 mL/min
Purge Flow	: 3 mL/min
Injection Method	: Split 1:5
Carrier Gas Controller	: Constant linear velocity mode
Detector	: FID
Detector Temp.	: 300 °C
Injection Volume	: 0.1 µL

*1 Tokyo Chemical Industry Co., Ltd., 99.0 % or higher
*2 FUJIFILM Wako Pure Chemical Corporation, 98.0 % or higher
*3 Tokyo Chemical Industry Co., Ltd., 98.0 % or higher
*4 P/N: 227-36350-01

Chromatogram of Engine Oil Containing Gasoline

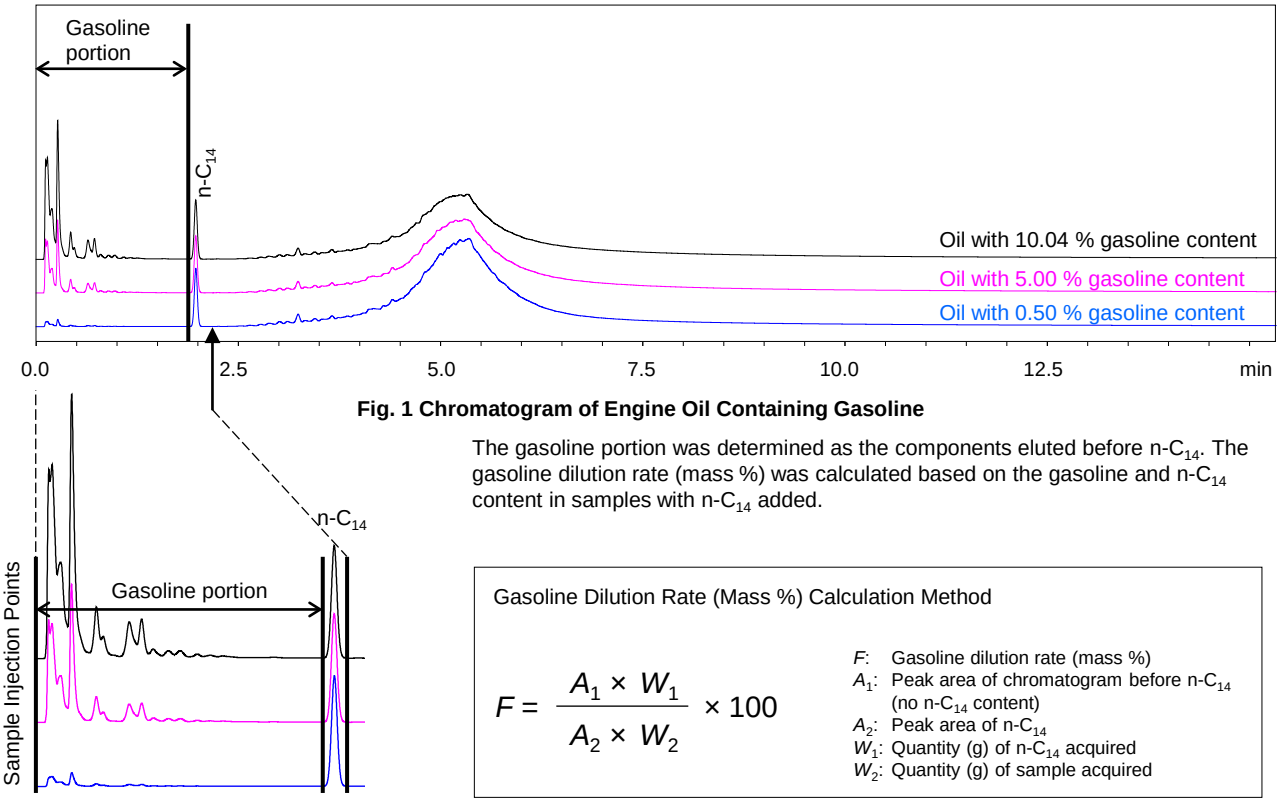

Fig. 1 Chromatogram of Engine Oil Containing Gasoline

The gasoline portion was determined as the components eluted before n-C_{14}. The gasoline dilution rate (mass %) was calculated based on the gasoline and n-C_{14} content in samples with n-C_{14} added.

Gasoline Dilution Rate (Mass %) Calculation Method

$$F = \frac{A_1 \times W_1}{A_2 \times W_2} \times 100$$

F: Gasoline dilution rate (mass %)
A_1: Peak area of chromatogram before n-C_{14} (no n-C_{14} content)
A_2: Peak area of n-C_{14}
W_1: Quantity (g) of n-C_{14} acquired
W_2: Quantity (g) of sample acquired

Precautions for Injecting Samples

The given analytical conditions do not require any pretreatment steps, such as dilution with n-C_{16}, carbon disulfide (CS_2), or other solvent. A small quantity of highly viscous samples is injected, but the results confirm that adequate accuracy can be achieved by modifying the consumables or injection actions involved. Sample injection conditions are summarized in Table 2.

Table 2 Sample Injection Conditions

Syringe:	0.5 µL volume syringe (P/N 000445 in Fig. 2) *1
Rinse Solvent:	CS_2
Plunger Aspiration Speed:	Low
Sample Rinsing:	None
Number of Pump Strokes:	Zero times
Insert:	P/N 227-35007-01 (Fig. 3) Wool is positioned 18 mm from top

*1 If new, repeatedly aspirate and discharge solution to ensure the plunger slides smoothly before use.

Fig. 2 0.5 µL Syringe (P/N 000445)

Fig. 3 Deactivated Insert for Split Injection (P/N 227-35007-01)

Confirming the Column Resolution

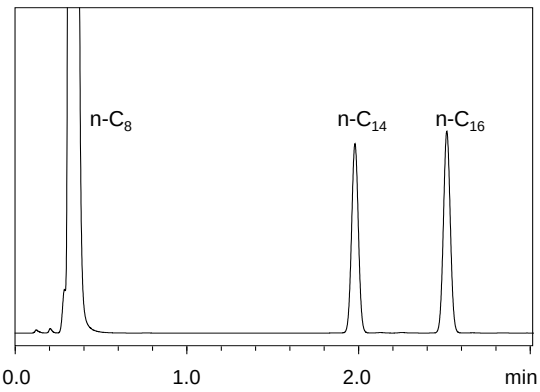

Fig. 4 Chromatogram of Column Resolution Measurement Sample

Results from analyzing column resolution measurement samples are shown in Fig. 4. The column resolution between n-C_{14} and n-C_{16} was 7.3. Results confirmed that the resolution between n-C_{14} and n-C_{16} in samples for measuring column resolution is at least 3 and not more than 8 (USP), as required by ASTM standards.

Repeatability of Gasoline Dilution Rates

The repeatability of gasoline dilution rates is shown in Table 3. Excellent repeatability %RSD (n = 10) results were obtained. The results also confirmed that all within-laboratory accuracy values satisfy tolerances required by standards.

Table 3 Repeatability %RSD (n = 10) of Gasoline Dilution Rates (%)

	Sample 1	Sample 2	Sample 3
1	0.66	4.91	9.91
2	0.66	4.87	9.89
3	0.66	4.86	9.97
4	0.65	4.89	9.90
5	0.65	4.87	9.80
6	0.65	4.84	9.76
7	0.65	4.82	9.76
8	0.65	4.88	9.73
9	0.65	4.86	9.71
10	0.65	4.85	9.64
Average	0.65	4.87	9.81
%RSD	0.74	0.52	1.08

Summary

This analysis achieved accuracy levels required by ASTM D3525 and JPI-5S-24 using the indicated analytical conditions with a nitrogen carrier gas, without involving dilution with a solvent or other pretreatment steps. These standards specify requirements for using the backflush method to eliminate residual components. For more information about high-throughput analysis for gasoline dilution rate testing using backflushing, refer to Application News No. G313 regarding ASTM D7593. Other Application News bulletins related to fuel dilution rates are indicated in the list of references below.

List of References

Standard	Item Analyzed	Application News No.
D3524	Diesel	G310
JPI-5S-23	Diesel	G311
D3525	Gasoline	G312
JPI-5S-24	Gasoline	G312
D7593	Gasoline	G313
	Diesel Biodiesel	G314

Reference Documents
ASTM D3525-04
JPI-5S-24-2017

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