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Application News

No. A603

Spectrophotometric Analysis

Degradation Analysis of Lubricants Based on ASTM E2412 by Fourier Transform Infrared Spectrophotometer FTIR

Lubricants consist of a base oil and additives and are used for purposes such as internal lubrication of machinery, cooling, and rust prevention. For example, engine oil, which is a lubricant used in engines, is necessary and indispensable for normal operation of an engine. The parts in an engine operate at high speed, and in this process, metal wear and seizing (phenomenon causing damage to cylinders or pistons) occur. The internal parts of the engine are lubricated with a lubricant to alleviate these problems. Combustion and rotational motion also cause various types of sludge (soiling, burnt residue) which reduce engine performance and life. Lubricants also have the role of adsorbing and dispersing sludge.

Lubricants are degraded by oxidation, consumption of additives, and accumulation of sludge. Because degradation of the lubricant shortens engine life and causes operational problems, it is necessary to grasp the condition of degradation and carry out oil changes at the proper timing. Fig. 1 shows the representative causes of lubricant degradation in engines.

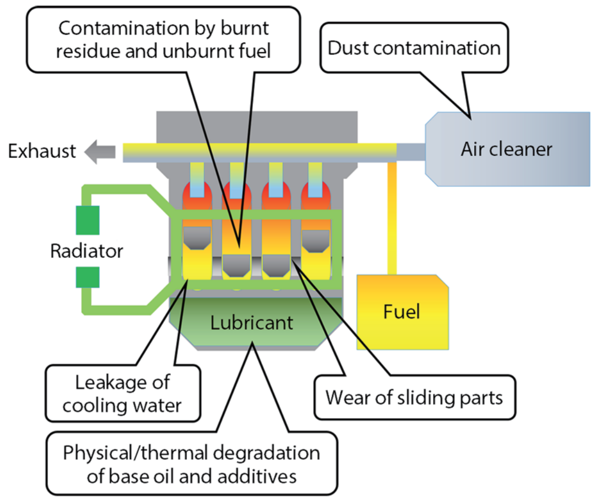

Fig. 1 Representative Causes of Lubricant Degradation in Engines

The relevant ASTM standard provides a method for evaluating lubricants from various parameters. It is necessary to select the proper analytical instrument for each evaluation item and measurement object. Table 1 shows a list of the lubricant evaluation items and measurement objects by the Fourier transform infrared spectrophotometer (FTIR), gas chromatograph (GC), and inductively coupled plasma-atomic emission spectrometer (ICP-AES) presented in the ASTM standard.

It is possible to evaluate chemical changes such as oxidation, nitration, and sulfonation of lubricants and contamination by moisture and dust by FTIR. These methods are provided in ASTM E2412.

Here, new automotive engine lubricant and degradation after use were evaluated by using the FTIR transmission method.

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Table 1 List of Lubricant Evaluation Items and Measurement Objects

Evaluation item/Measurement object		Instrument	ASTM
Degradation	Oxidation	FTIR	E2412
	Nitration		
	Sulfonation		
Contamination	Moisture	FTIR	E2412
	Soot		
	Gasoline	GC	D3525 D7593
		FTIR	E2412
	Diesel	GC	D3524 D7593
		FTIR	E2412
	Coolant (B, Na, K)	ICP-AES	D5185
		FTIR	E2412
	Antifreeze (Na)	ICP-AES	D5185
	Dust (Si)		
	Sealant (Si)		
Wear	Metal (Al, Fe, Cu, Cr, Ni, Zn, etc.)	ICP-AES	D5185
Additives	Antioxidant (Zn, Cu, B)	ICP-AES	D4951
		FTIR	E2412
	Anti-wear agent (B, Cu, K, S, Zn, etc.)	ICP-AES	D4951
		FTIR	E2412
	Surfactant (Ba, Mg, Ca, etc.)	ICP-AES	D4951
	Corrosion inhibitor (Ba, Zn)		
	Anti-rust agent (K, Ba)		
	Lubricant (Mo)		

Measurement Conditions and Samples

In the transmission measurements, the Pearl™ horizontal type liquid FTIR transmission accessory (Specac) shown in Fig. 2 was used. Pearl™ is a convenient accessory, as liquid samples can be held horizontally, intrusion of air bubbles is difficult, and cleaning is easier than with the liquid cells that are generally used. Setting of the samples is extremely simple, as shown in Fig. 3, requiring only dripping the liquid sample and then covering it with the window. Table 2 shows the measurement conditions, and Table 3 shows the details of the used lubricants A and B. For comparison, new lubricants A and B were also measured.

Fig. 2 Pearl™ Horizontal Type Liquid FTIR Transmission Accessory

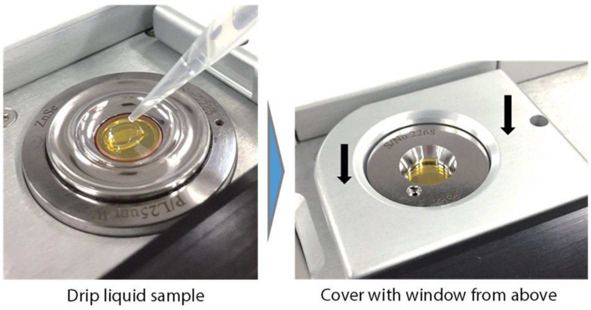

Fig. 3 Sample Setting Procedure

Table 2 Measurement Conditions

Instruments	:	IRSpirit™-T (KBr window plate) Fourier transform infrared spectrophotometer Pearl™ horizontal type liquid FTIR transmission accessory (optical path length: 100 μm)
Resolution	:	4 cm^{-1}
Accumulation	:	40 times
Apodization function	:	Happ-Genzel
Detector	:	DLATGS

Table 3 Details of Used Lubricants

Automotive engine lubricant	
A	B
Viscosity: 10 W-60[*1] Travel distance: 3,000 km Use duration: 3 months Use condition: Use in high rpm range	Viscosity: 0 W-20[*1] Travel distance: 5,000 km Use duration: 1 year Use condition: Use in general urban driving

*1 SAE classification notation (SAE: Society of Automotive Engineers) In the first part, 10 W and 0 W indicate that use is possible to -25 °C and -35 °C, respectively. In the second part, 60 and 20 are viscosity at high temperature (100 °C).

Results of Degradation Evaluation

Fig. 4 (a) and (b) show the infrared spectra of the new and used lubricants A and B, respectively.

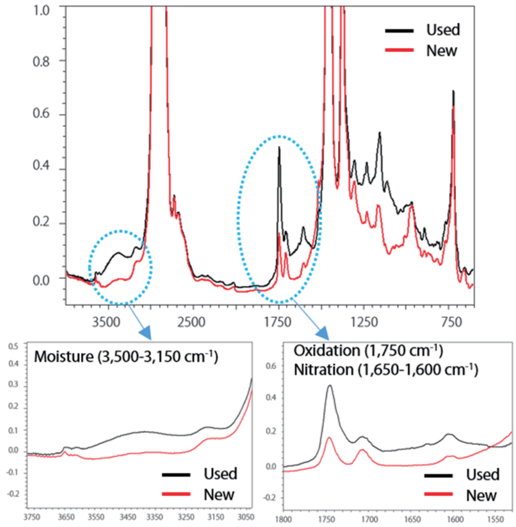

Fig.4(a) Infrared Spectra of New and Used Lubricant A

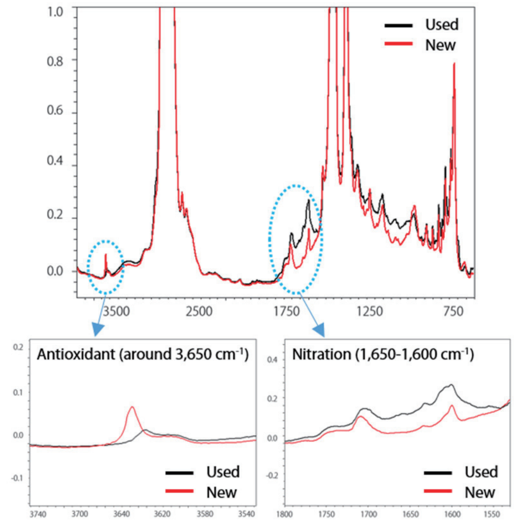

Fig.4(b) Infrared Spectra of New and Used Lubricant B

In lubricant A, in addition to contamination by moisture, degradation by oxidation and nitration could also be confirmed from Fig. 4 (a). On the other hand, in lubricant B, a decrease in the antioxidant, which is an additive, and degradation by nitration could be confirmed from Fig. 4 (b). No change in the spectrum in the range of 1,800 to 1,670 cm^{-1} by oxidation could be observed, and it can be presumed that this was the effect of the antioxidant.

Conclusion

Evaluations of lubricants by FTIR are simple and do not require Sample pretreatment. Efficient work is possible by using the Pearl™ horizontal type liquid FTIR transmission accessory, and cleaning is easy in comparison with conventional liquid cells. Moreover, because Pearl™ maintains the optical path length with high accuracy, data with good reproducibility can be obtained in measurements based on ASTM E2412.

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