C10G-E085

Excellence in Science

Solutions for Lubricant Monitoring

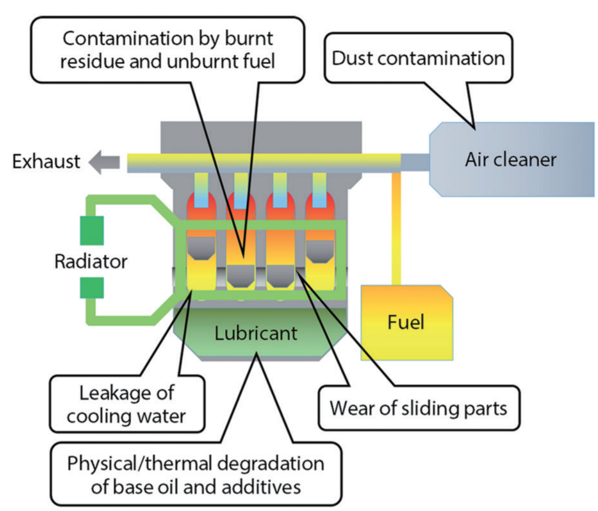
Lubricants are the lifeblood of mechanical engines and provide critical friction-reducing, cooling, and cleaning properties that are essential to their proper functioning and operation. Over time and throughout their use, lubricants degrade, and that functionality and those properties become inhibited. This degradation can cause mechanical problems and shorten the lifespan of an engine.

Just as clinical analyses of human blood can diagnose health conditions, chemical analysis of lubricants ensures their performance and can indicate mechanical issues that demand attention, which is critical to the functioning of engines and motors. Particularly on large engines or turbines in which the volume of oil in the sump can be thousands of times greater than in a passenger car, routine monitoring of lubricants ensures optimal performance, minimizes costs, and can protect the engine. Shimadzu has all of the tools necessary for common lubricant analysis, including additives and degradation as well as fuel dilution, wear metals, and other contaminants.

Why do lubricants degrade?

Lubricants degrade during use through a variety of physical and chemical phenomena. These include the molecular breakdown of lubricant and its additives due to thermal, shear, and oxidative stresses, water emulsification into the oil, contamination by airborne dust, wear of moving parts, leakage of coolant, and incorporation of unburned fuel, soot, and exhaust gases into the oil.

Chemical degradation and incorporation of particulate matter negatively affect the performance of a lubricant. Some of this degradation is visually obvious, such as the darkening of lubricants during use, but much of it occurs at the elemental and molecular level. This necessitates the use of sensitive analytical techniques for monitoring.



Test Methods for Lubricant Analysis

Most of the industry-standard practices follow test methods set forth by ASTM International. The table below outlines some of the common lubricant analysis test methods and their target analytes and instrumentation.

Condition Assessment		Instrument	Standard(s)
Deterioration	Oxidation	FTIR	ASTM E2412
	Nitration		
	Sulfate by-products		
Contamination	Water	FTIR	ASTM E2412
	Soot		
	Gasoline	GC	ASTM D3525
			ASTM D7593
		FTIR	ASTM E2412
	Diesel	GC	ASTM D3524
			ASTM D7593
		FTIR	ASTM E2412
	Coolant (B, Na, K)	ICP-AES	ASTM D5185
		FTIR	ASTM E2412
	Antifreeze (Na)	ICP-AES	ASTM D5185
	Dust (Si)		
	Seal / Gasket Materials (Si)		
Wear	Metals (e.g., Al, Fe, Cu, Cr, Ni, Zn, etc.)	ICP-AES	ASTM D5185
Additives	Anti-Oxidants (Zn, Cu, B)	ICP-AES	ASTM D4951
		FTIR	ASTM E2412
	Anti-wear Agents (B, Cu, K, S, Zn, etc.)	ICP-AES	ASTM D4951
		FTIR	ASTM E2412
	Detergents (Ba, Mg, Ca, etc.)	ICP-AES	ASTM D4951
	Corrosion Inhibitors (Ba, Zn)		
	Anti-rust Agents (K, Ba)		
	Friction Modifiers (Mo)		

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Lubricant and Additive Degradation

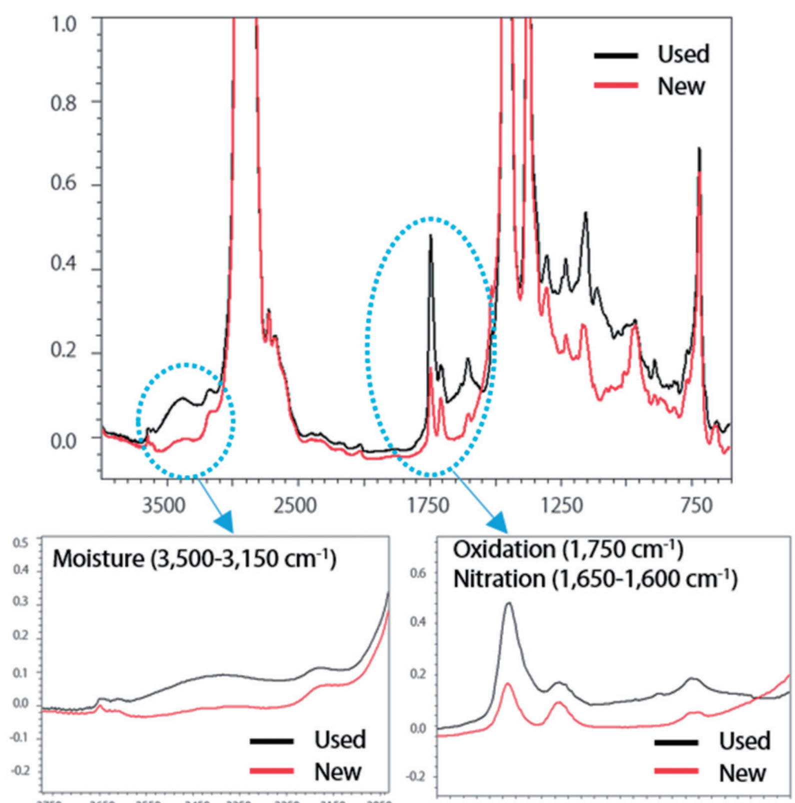
Due to thermal and physical stresses, the molecular composition of lubricants changes as they are used. Common molecular changes include oxidation, nitration, sulfonation, and hydration. Sacrificial additives, such as anti-oxidants, help mitigate these changes, but their chemistry changes over time as they perform their intended role. Fourier Transform Infrared Spectroscopy, or FT-IR, is a fast and simple technique that allows for analysis of these molecular changes in lubricants.

Fourier Transform Infrared Spectrometer IRSpirit™

FT-IR analyses are commonly used to assess lubricant chemistry because they are quick, easy to perform, and can target multiple analytes in a single analysis. About the size of a typical ink jet printer, Shimadzu's IRSpirit features a small, space-saving design with the widest sample compartment in its class. It can be equipped with a variety of accessories, such as the Pearl™ Liquid Analyzer and the Q-ATRS attenuated total reflectance prism, to simplify lubricant analysis.

The Pearl™ Liquid Analyzer is easily inserted into the optical path of the IRSpirit and allows for a simple way to analyze liquid samples by FT-IR with no sample pretreatment. The construction of the Pearl Liquids Analyzer ensures a consistent and repeatable pathlength through the samples, even after changing samples. The analyst simply places a small amount of the liquid on the sampling cell, closes the lid on the liquid cell, slides the cell into the light path, and the instrument is ready for analysis. Changing samples is made simple with a solvent and lens paper.

The figure to the right shows incorporation of water into used lubricant, as well as oxidation and nitration due to thermal stresses. By analyzing lubricants at known wavelengths for O-H, C-O, and C-N bonds, among others, qualitative and quantitative assessment can be quickly performed on lubricant samples using the IRSpirit.



Specac Pearl™ Liquid Analyzer

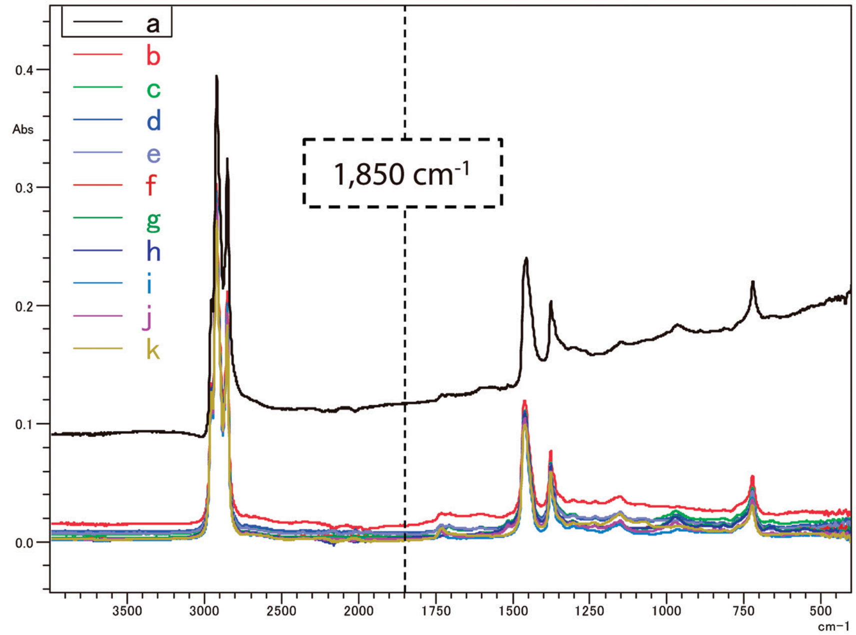
Soot Contamination

Incomplete combustion of fuels results in the production of soot, carbon-rich particulates in the exhaust stream. This soot can be incorporated into the lubricant, visibly darkening its color through use. Soot accumulation in the lubricant negatively affects its properties and fouls oil filters over time. Excess soot particles in lubricants may indicate problems with combustion, air-fuel mixtures, or timing of the engine.

Fourier Transform Infrared Spectrometer IRSpirit

The use of FT-IR with an ATR (attenuated total reflectance) prism allows quick and easy quantify the amount of soot incorporated into lubricating oils. Because dark-colored carbon within oils strongly absorbs light in the range of 1850-2000 cm^{-1}, quantification of soot particulates in oil is simple.

Increasing baseline levels with increasing soot content as measured by FTIR



With the use of the IRSpirit equipped with the QATR™-S prism, analysis of soot is by FT-IR is as simple as placing the sample on the stage, tightening the "lid", and beginning analysis. Because the QATR-S is installed seamlessly and its design prevents overtightening, fast, repeatable results are within reach.

Additive Elements and Wear Metals Analysis

Certain elements and their compounds are specifically added to lubricants for their unique properties. For example, zinc and phosphorous compounds are added to lubricants as antioxidants and corrosion inhibitors. Ensuring those additive compounds are added to lubricants at the optimal concentrations ensures their proper functioning.

Metals particles may also be incorporated into lubricants by slow erosion of metallic engine components. So-called wear metals can act as an abrasive, causing further erosion of components. Analyzing for wear metals is important for indicating the lifespan of a lubricant or diagnosing mechanical problems causing excessive wear.

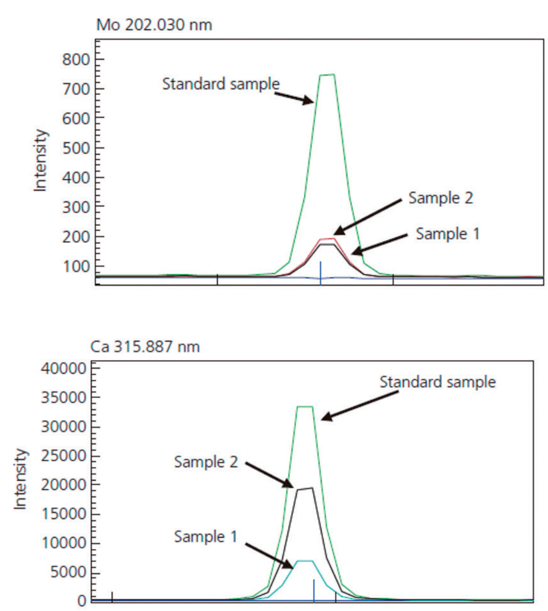
Regardless of whether the metals and metallic compounds are added intentionally or incorporated through abrasion of moving components, analysis for them is a crucial aspect of any lubricant testing program.

Inductively Coupled Plasma Atomic Emission Spectrometer ICPE-9800 Series

Elemental analyses for additives and wear metals in lubricating oils are commonly performed by ICP-AES instruments due to their sensitivity, robustness, and rapid sample throughput. The ICPE-9800 series features a vacuum-purged optical bench to minimize operating costs and increase sensitivity at low wavelengths. The vertically-oriented torch minimizes carry-over between samples and is more amenable to high-TDS and organic samples by minimizing accumulation of matrix material on the torch. LabSolutions™-based software contains assistant functions that enable the user to easily identify interferences and select optimal wavelengths for analysis.

The table presented here shows quantitative results of elemental analysis of used and unused lubricant, including spike-and-recovery results for low-concentration analytes and dilution results from high-concentration analytes. Using Shimadzu's ICPE-9800 series, highly-stable and sensitive results are generated with ease for both trace and major metallic contaminants.

Element	Used Lubricant (µg/g)	Used Lubricant Spike Recovery (%)	Used Lubricant Dilution Test (%)	Unused Lubricant (µg/g)	Detection Limit (µg/g)
Ag	n.d.	100	-	n.d.	0.02
Al	10	101	-	6.51	0.3
B	65.9	-	98	121	-
Ba	0.123	101	-	n.d.	0.02
Ca	3970	-	98	2250	
Cr	1.03	101	-	n.d.	0.01
Cu	0.65	100	-	n.d.	0.02
Fe	10.8	101	-	0.43	0.01
K	22.1	99	-	n.d.	0.6
Mg	10.4	100	-	5.48	0.02
Mn	0.618	101	-	0.139	0.002
Mo	184	-	98	183	-
Na	2.5	100	-	n.d.	0.4
Ni	n.d.	102	-	n.d.	0.05
P	756	-	99	731	-
Pb	n.d.	100	-	n.d.	0.5
S	3980	-	100	3810	-
Si	8.96	103	-	5.07	0.03
Sn	n.d.	100	-	n.d.	0.5
Ti	n.d.	100	-	n.d.	0.01
V	n.d.	103	-	n.d.	0.022
Zn	872	-	97	882	-



These figures show optical emission spectra of calcium and molybdenum, elements in common additive compounds, in lubricating oils.

Fuel Dilution

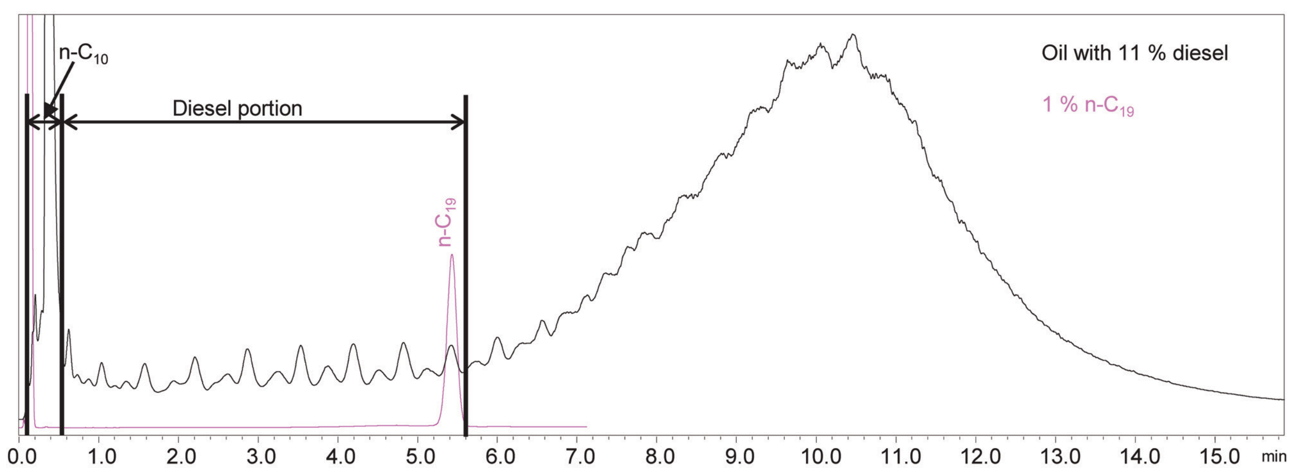
Unburned liquid fuels can be integrated into lubricating oil and can change its viscosity, its ability to disperse sludge and deposits, and its lubricating ability. Quantifying the amount of fuel in the oil, also called fuel dilution, can be indicative of improper fuel-to-air ratios or failure of sealants in the engine, being diagnostic of the need for mechanical maintenance.

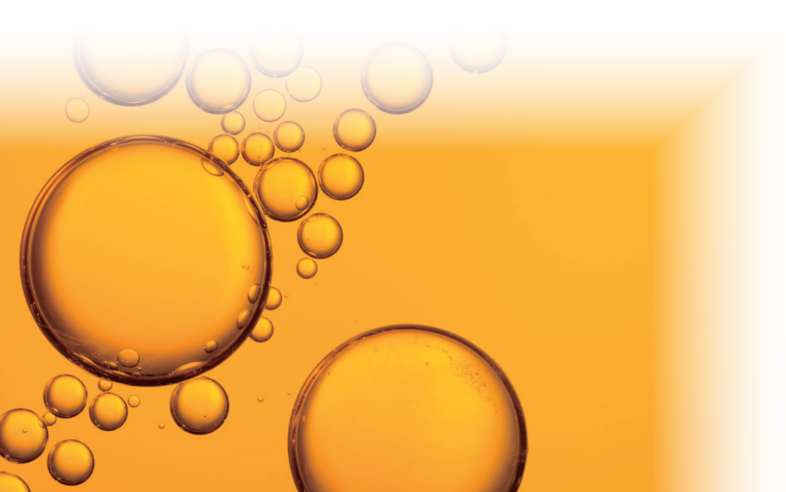
Gas Chromatograph GC-2030

Shimadzu's state-of-the-art GC-2030 gas chromatograph is compliant with industry-standard methods for quantifying the amount of common fuels, such as gasoline and diesel, in lubricants. It features a large, color touch-screen for easy operation, proprietary ClickTek™ one-push column connectors to simplify maintenance, and easy conversion between common carrier gases such as helium, hydrogen, and nitrogen.

It can also be outfitted with a range of autosamplers, including Shimadzu's AOC™-6000, which fully automates sample preparation, pre-treatment, and injection. Though FID is used for fuel dilution analysis, the GC-2030 can be equipped with several other detectors to expand the scope of its applicability.

This figure shows a chromatogram of 11% diesel fuel diluted into engine oil using nitrogen carrier gas.





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