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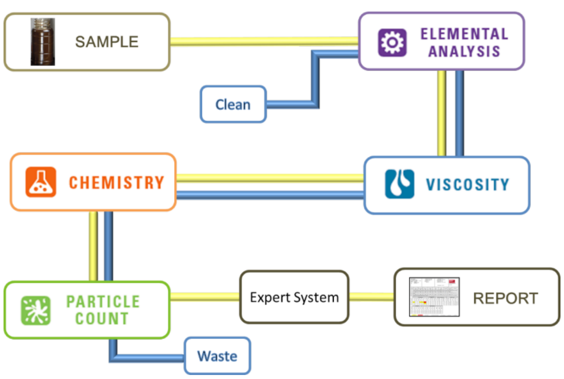
Confidence in knowing

WHITE PAPER

MicroLab® Series – Fully Automated Oil Analyzer

Introduction

The MicroLab® is a fully automated, bench-top analyzer specifically designed for the analysis of oil samples to provide comprehensive analytical and diagnostic results of engine, generator, gearbox, hydraulics, power steering and transmission fluids. The MicroLab was developed to bring oil analysis capability to any company that maintains equipment and seeks to improve their overall equipment readiness. Companies can send oil samples out to a traditional lab but that process can take days or weeks for results. The MicroLab provides results in less than 15 minutes allowing the mechanic to take immediate maintenance action. The MicroLab is well suited for over-the-road and off-road vehicles, generators and other reciprocating machinery.



The MicroLab is the ideal trending tool to allow companies to identify mechanical problems before they become catastrophic equipment failures. The ability to track the serviceable life of the oil to know when they can safely extend the oil drain interval helps save money on oil consumption, oil disposal and labor costs associated with unnecessary oil drains.

The MicroLab is the first compact analyzer that combines multiple measurement modules, automated fluidics and interactive software to provide non-laboratory users the capability to obtain analytical results along with diagnostic interpretation. The MicroLab's innovative design and methods hold multiple patents and the analyzer complies with the ASTM standard for a Multi-Functional Oil Analysis Instrument, ASTM D7417. It combines four separate test components in an all-in-one, fully automated analyzer. The MicroLab includes an infrared spectrometer (IR) for six parameters for oil chemistry and contamination, an optical emission spectrometer (OES) to report up to 20 elements for wear metals, contaminates and oil additives, a dual temperature viscometer (DTV) to report viscosity at 40° and 100°C along with viscosity index, and an optional particle counter.

This paper serves as a guide to the technology and design considerations for the MicroLab and includes what to expect when comparing it to laboratory methods and results.

ELEMENTAL ANALYSIS

Elemental analysis is the backbone of any oil analysis program and is designed for detection of wear metals and other impurities as well as oil additive levels to provide information on equipment condition and oil health. MicroLab 30 analyzes 10 basic metals: aluminum, chromium, copper, iron, lead, molybdenum, potassium, silicon, sodium, and tin. It is upgradable to include all 20 elements. MicroLab 40 analyzes 20 elements including the basic metals and the extended metals: barium, boron, calcium, magnesium, manganese, nickel, phosphorous, titanium, vanadium, and zinc.

Principle of Operation

The MicroLab employs Optical Emission Spectroscopy (OES) to quantify elements from mechanical wear, oil additives or sources of contamination. OES relies on the fact that every element has a unique atomic structure. When an atom is excited with enough energy it will emit light of discrete wavelengths (or colors) based on its atomic structure. Since no two elements share the same pattern of emitted wavelengths, the emission spectrum is a fingerprint that can be used to identify the elements that are present in a sample as shown in figure 1. Also, the intensity of the emitted light can be correlated to the concentration of that element in a sample.

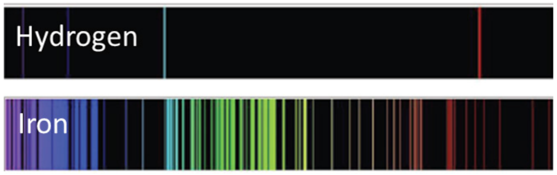
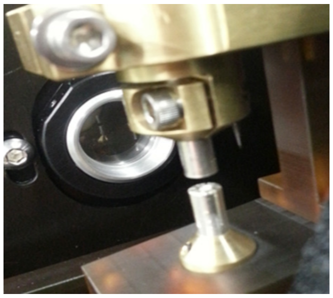


Figure 1. The optical emission spectra of hydrogen and iron.

An optical emission spectrometer consists of three parts: (1) the excitation source, (2) the optical system, and (3) the readout system. The two most popular methods of elemental analysis for oil analysis available on the market differ primarily in the excitation source of the sample: Inductively Coupled Plasma Spectroscopy (ICP) and Arc/Spark/Rotating Disk Electrode Spectroscopy.



In the MicroLab OES, the spark module consists of a spark stand, two electrodes and a high voltage power source. As shown in Figure 2, high voltage from a computer-controlled pulsed power source is applied to the upper and lower electrodes of the spark stand to generate electrical spark and create plasma. The oil sample is pushed through the hole to the top of the lower electrode where it is heated, vaporized and atomized. The atomized elements are subsequently excited by their collision with the high energy particles in the plasma flume. Some atomized elements are ionized and the formed ions are excited. When these excited atomic or ionic elements return to their ground states, they emit light which bears the signature of the emitting element. The emitted light is collected with the lens and delivered with an optical fiber to the optical spectrometer, and the data of emission spectrum is collected. The optical spectrometer collects the spectrum in the ultraviolet (UV) and the visible (Vis) light region.

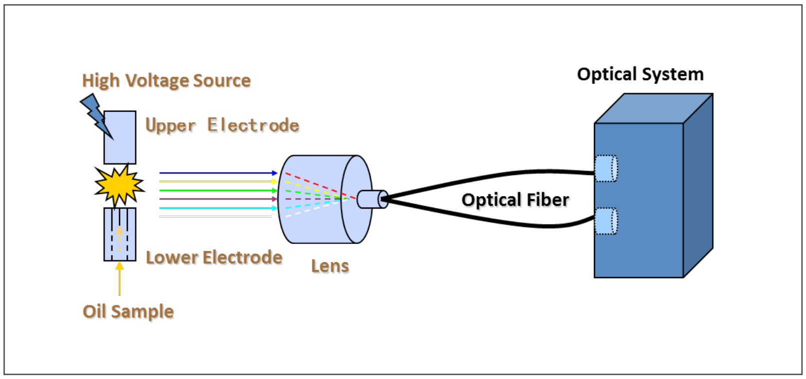


Figure 2. Schematic of MicroLab OES system and a picture of spark stand (above).

The collected sample emission spectrum is analyzed with a sophisticated data analysis procedure to identify and quantify the elements. Figure 3 below shows a representative emission spectrum (in red) of a customer used oil sample, and for comparison an emission spectrum of white mineral oil is shown which contains no metal element at all. The emission lines (or peaks) for iron (Fe), Silicon (Si), and copper (Cu) are identified based on their wavelengths. By measuring the peak area of

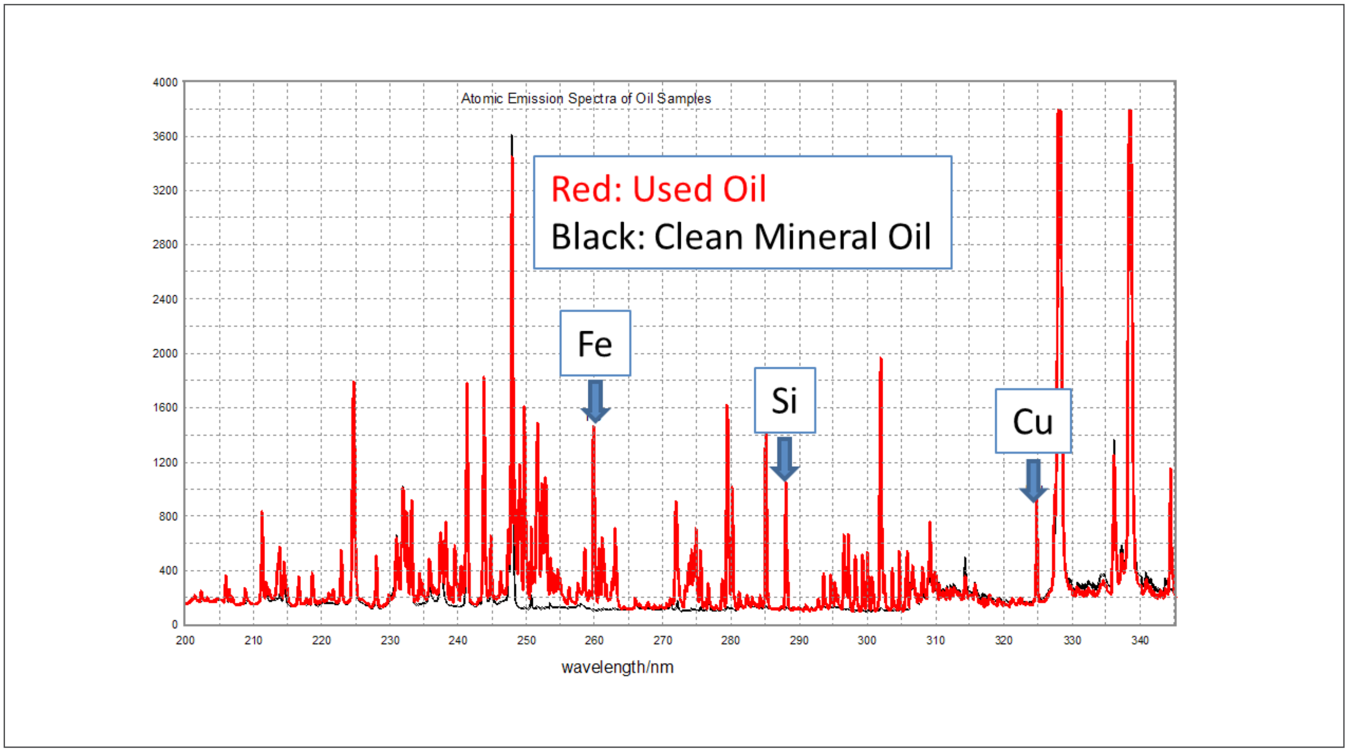


Figure 3. Representative emission spectra from two oil samples: the spectrum in red is from a customer used oil sample and the one in black is white mineral oil shown here for comparison.

each element and comparing to the peak area of the standard samples measured at the time of instrument calibration during production, the concentration (in ppm) of the element is then determined.

Several parameters of the OES system, such as spark frequency, voltage, gap between the electrodes, and oil sample flow rate, can affect the plasma characters and consequently influence the accuracy of the concentration measurements for the sample. These parameters are optimized during production, and then the OES is calibrated with a set of standard samples with known concentrations of the elements. The calibration coefficients for each of the elements are stored in the computer and used later to calculate the element concentrations for unknown customer samples.

Comparison to Other Techniques

MicroLab typically correlates well to other OES methods and provides similar trending information. The principle of operation is the same for all optical emission techniques; however, there may be some differences due to matrix, plasma temperature, or particle size. The MicroLab module is a dedicated fixed electrode spark spectrometer, specifically chosen for the application. The plasma temperature of the MicroLab OES is the lowest of those typically employed by used oil analysis:

ICP: approx. 6000 to 8000 K
RDE: approx. 5000 to 7000 K
MicroLab OES: approx. 3000 to 5000 K

In oil labs, ICP analysis is typically a “dilute and shoot” approach. The oil sample is diluted with kerosene and nebulized with a spray chamber to produce an aerosol spray which is introduced to the plasma to produce the emission spectrum. Superb accuracy

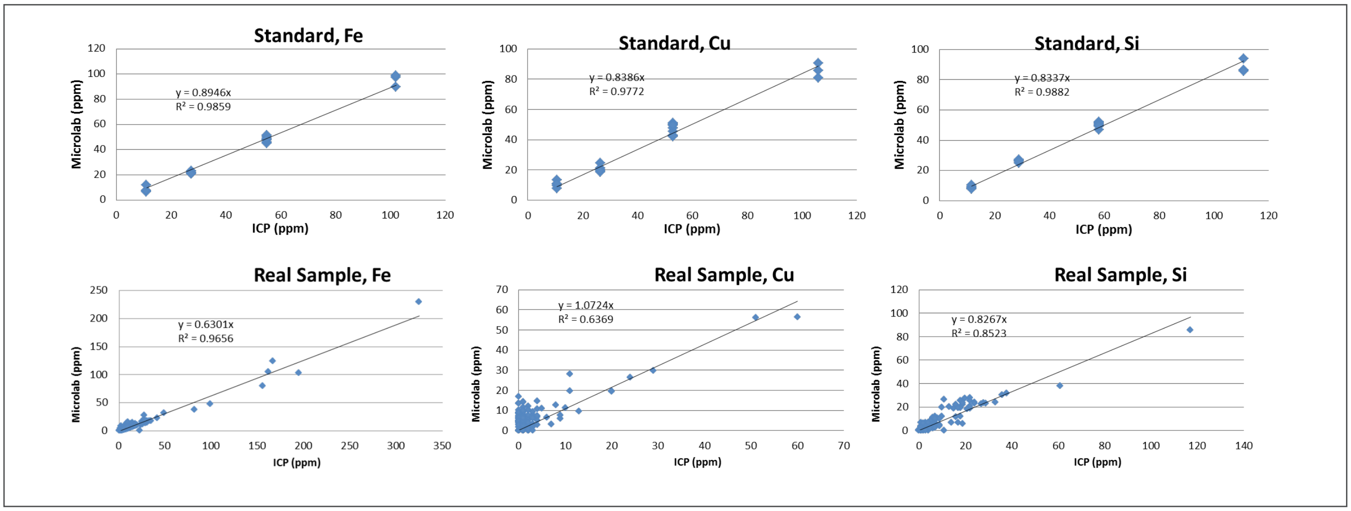


Figure 4. MicroLab results compared to Lab ICP-OES for elemental standards and several real in-service oil samples.

and repeatability can be achieved for particle sizes <5 μm. Acid digestion can be used when larger particle sizes are needed. The advantages of ICP are reduced matrix effects due to the sample dilution, analysis of larger particles with acid digestion, and the superb analytical performance. The main disadvantages of ICP are the cost and sample preparation required.

Figure 4 shows MicroLab OES results compared to Lab OES-ICP results for elemental standards in mineral oil as well as for a series of authentic in-service heavy duty engine oils. Although the MicroLab is systematically lower than the ICP results, the trend is maintained in both the standards and in-service samples. The elemental standards show excellent correlation over the calibration range, whereas for the real samples there is significantly more scatter at lower ppm range. Most of this variation compared to ICP is likely due to matrix effects in the real samples. ICP generally has a dilution factor of approximately 10, so any matrix effect is minimized.

Figure 5 compares the MicroLab results on a variety of real samples to a more similar method, the SpectrOil RDE-OES. The reported MicroLab results are lower in magnitude likely due to a difference in plasma temperature, but it still provides excellent trending information compared to this technique.

To summarize, the MicroLab is designed for routine fleet-based oil analysis and provides results that when taken together with other measurement methods provide an effective picture of what is occurring in the equipment. The results may not be equal to those obtained from RDE or ICP, but they will trend in the range of interest in order to diagnose common issues.

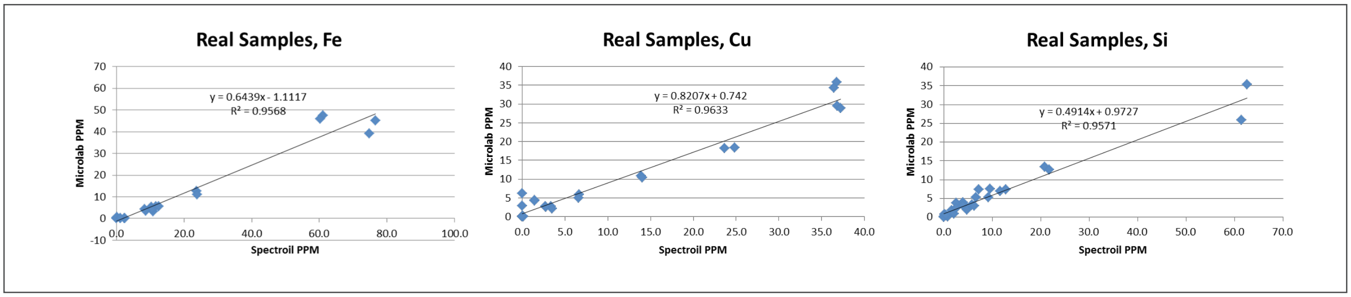


Figure 5. MicroLab results compared to SpectrOil RDE-OES for several real in-service oil samples

CHEMISTRY

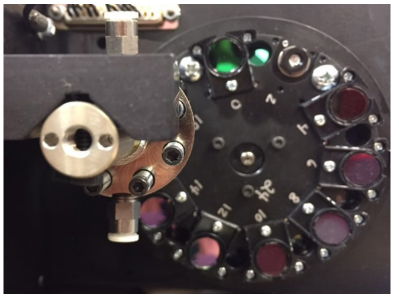
A change in oil chemistry can affect the ability of the lubricant to perform its function. The MicroLab uses Infrared (IR) Spectroscopy to measure six key physical parameters to assess oil degradation and contamination:

- TBN - Total Base Number, measure of alkaline reserves in oil
- Oxidation, Nitration – represent oil degradation process due to chemical reaction of oil with air at high temperature
- Soot – nanometer size hard carbon particles as combustion by-product
- Glycol – contamination from coolant leak
- Water – moisture in oil or contamination from coolant leak

Principle of Operation

In the MicroLab, the IR module uses a filter based mid-infrared spectroscopic analysis technique to differentiate and quantify the components in an oil sample. The module uses a proprietary set of optical band-pass filters mounted on to a filter wheel to measure the amount of IR beam that is absorbed by an oil sample at each specific filter as shown in Figure 6. The amount of light absorbed is proportional to the concentration of the component(s) in the oil sample. The sample is introduced into the MicroLab's IR module sample cell and the filter wheel is rotated to each of the 15 filters where the scans are taken to collect the absorbance data.

The mid-infrared range of 800-4000 cm^{-1} is the region of interest for routine oil analysis. Using built-in calibrations for the different parameters, the MicroLab analyzes the collected IR spectrum and provides trendable, quantitative data. Oxidation and nitration products appear as peaks in the IR spectrum between 1600 and 1800 cm^{-1}. Because there are no absolute reference standards for oxidation and nitration, the results are always compared to those of new oil and trended over time. For example, if the nitration peak around 1650 cm^{-1} becomes significantly more intense as an engine oil is sampled over a specified time, then nitration has occurred which may indicate improper air/fuel ratio. Total Base Number can be monitored by the TBN additive depletion which appears as a decrease in the absorbance peaks in the region of 1000 to 1900 cm^{-1}. Water that is dissolved in oil shows a characteristic absorption peak between 3200 and 3800 cm^{-1}.



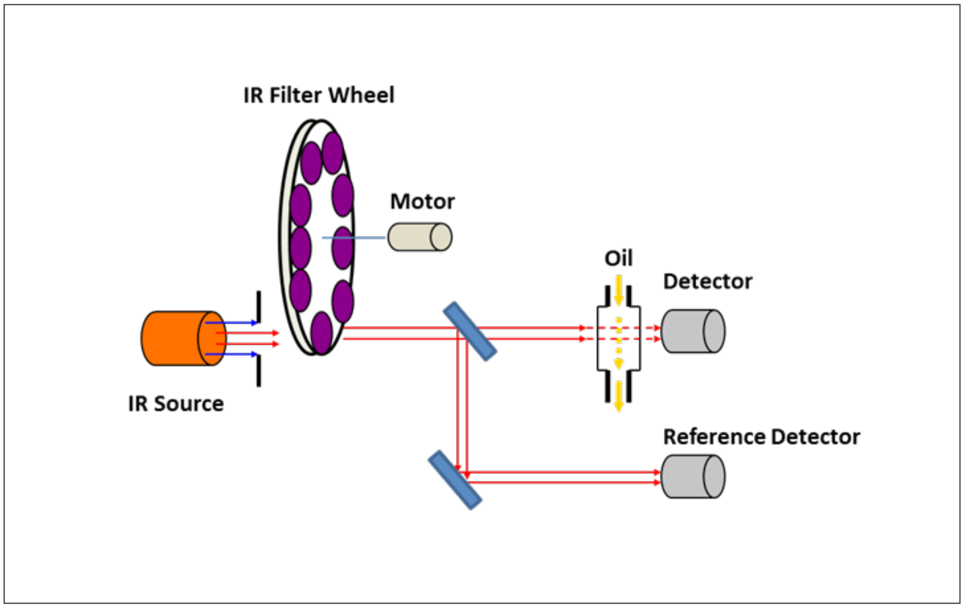


Figure 6. The IR module inside the MicroLab.

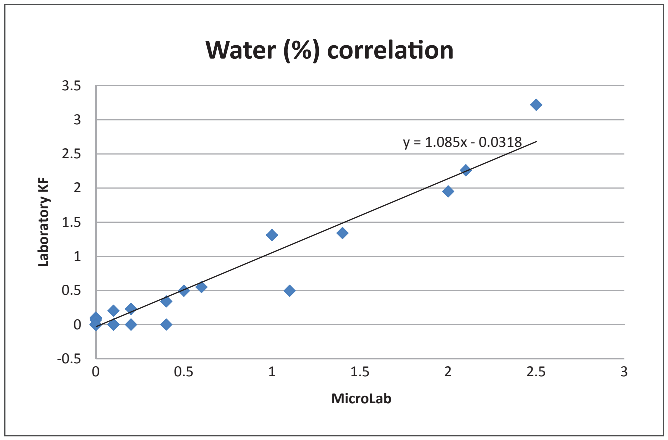


Figure 7. Water (%) correlation between MicroLab and Karl Fischer.

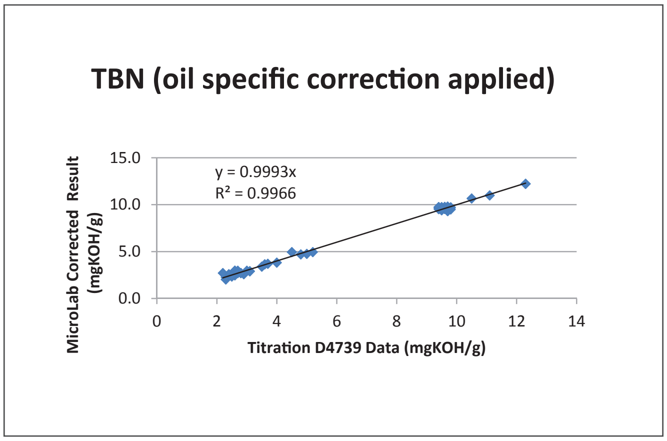


Figure 8. Applying an oil-specific slope and offset to TBN results improves the accuracy and correlation to the laboratory D4739 method.

Comparison to Other Techniques

Infrared technology is a common tool for oil analysis and well-accepted methods exist for laboratory grade FTIR measurement as well as for portable field testing. Two examples are ASTM E2412 which describes the standard practice for FTIR measurement of these properties and ASTM D7889 which describes a test method for portable IR measurement. In addition, specific test methods have been defined for oxidation (D7414) and nitration (D7624). Each ASTM method can be specific to the IR analytical design (FTIR vs IR), but the result output of the different methods can be similar (parameter, units of measure, etc). The MicroLab's IR spectrometer uses a dedicated design in order to arrive at these oil analysis parameters that match or correlate with existing laboratory methods.

Titration methods are used as the benchmark for TBN and water analysis. Potentiometric titration is the accepted method for TBN analysis by ASTM D2896 or ASTM D4739. These titration methods are designed only for laboratories and use a variety of solvents and reagents. The MicroLab uses a chemometric algorithm which reports TBN based on correlating the raw IR spectrum to the lab methods. For water, Karl Fischer titration ASTM D6304 is the referee method. As shown in Figure 7, the water measurement in the MicroLab shows correlation with Karl Fischer measurement.

The calibration models used in the MicroLab are universal, meaning the same calibration model is applied to every kind of sample. This was a compromise for customer convenience. The trade-off is that the measurement accuracy may vary depending on the kind of oil analyzed. If the absolute accuracy is important, it is possible to apply an oil specific slope and offset to the MicroLab result in order to improve the accuracy (Figure 8).

VISCOSITY

Viscosity is an important oil analysis test to perform because it determines the load carrying ability of the oil, as well as how easily it circulates.

Kinematic viscosity is defined as the resistance to flow under gravity (a constant force). MicroLab employs a Dual Temperature Viscometer (DTV) module to measure the sample viscosity at 40 °C and 100°C (and VI) which can help identify potential oil degradation or contamination. The innovative flow-through approach is a unique design to the MicroLab.

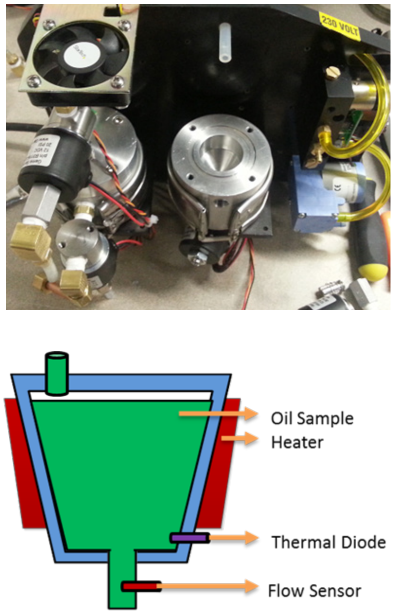


Figure 9. The dual viscosity module in the MicroLab.

Principle of Operation

The DTV works on the principle of sample flow under a constant pressure. As shown in Figure 9, the sample oil is pumped into two reservoir chambers, one with temperature regulated at 100 ºC and the other at 40 ºC. After the sample temperature reaches the equilibrium with the chamber temperature, the sample is then pushed out by the compressed air at constant pressure through a precision orifice at the bottom of the chamber. The time to push out a fixed amount of the sample is directly related to the sample viscosity. Therefore by accurately measuring the time the sample flows out, the sample viscosity can be calculated. The DTV is calibrated with a set of standard fluids during production, and standardized in the field by the customer at a given period or usage with the provided two standard fluids.

Comparison to Other Techniques

The most commonly referenced method for viscosity of lubricating oils is ASTM D445, however there are many other methods (ASTM D7279, D8092, etc). MicroLab DTV employs a technology that is very similar to ASTM D445. Both measure the sample viscosity based on the sample flow time. The difference is that in MicroLab the sample flows through an orifice under fixed or constant pressure, while in D445 the sample flows down a capillary glass tube under gravity. The MicroLab viscosity measurement agrees well with ASTM D445. Indeed as shown in Figure 10 below, MicroLab demonstrates excellent agreement with D445 measurements for a series of Shell Omala fresh oils with grade from ISO 46 up to ISO 320.

Figure 11 shows the comparison between MicroLab and D445 for the real world customer in-service oil samples. Not only does the MicroLab trend the viscosity variation, but it also has a satisfactory correlation and agreement with D445.

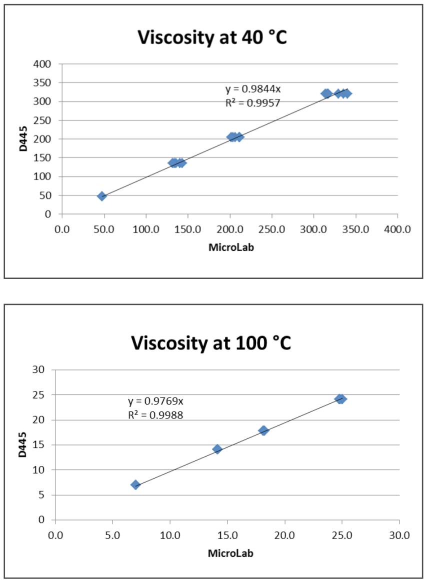


Figure 10. Comparison of Viscosity measurements between MicroLab and Lab D445 for a series of fresh Shell Omala oils with grade from ISO46 to ISO 320..

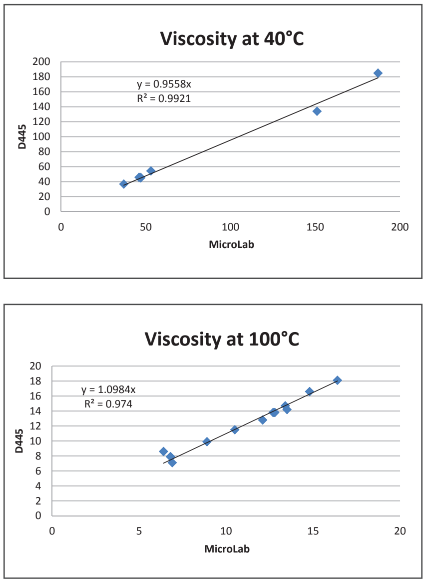
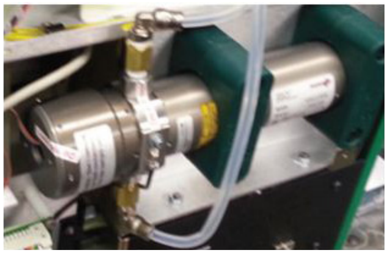


Figure 11. Real world samples measured by the MicroLab vs ASTM D445.

PARTICLE COUNT

The MicroLab 40 model is equipped with a particle counter module which employs a standard ISO 11171 light blocking technique to measure gross particle count and ISO particle size particle contamination. Particle counting is a critical part of any machine condition monitoring program for maintaining hydraulic systems, compressors and turbines to track the quantity and severity of contamination in an oil. Particles can be present in oil due to either external contamination such as dirt or from machine wear.



Principle of Operation

The MicroLab uses a commercially available light-blockage sensor diode laser and detector as shown in Figure 12. The liquid flows through the measuring cell of the sensor. On one side of the measuring cell, there is a light beam, on the other side there is a photodetector. If there are particles in the sample the light beam hits the particles and as a result, the shadow of the particle is shown on the photo-detector. The surface of the shadow causes a voltage change on the photodetector and indicates the size of the particle flowing through the sensor cell. The particle counter translates the number of shadows on the photo-detector to the quantity of particles in the liquid. Furthermore, the particle sizes are distributed in different size classes. The particle measurement results are reported according to the cleanliness class standards which are relevant for the specific application (e.g. ISO 4406). The MicroLab reports up to ISO code 25.

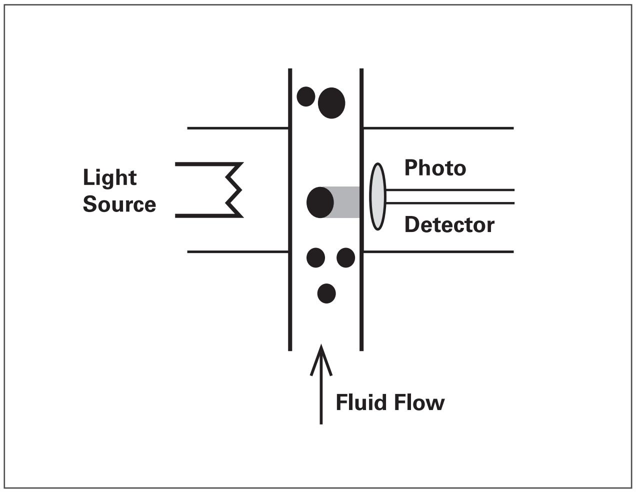


Figure 12. The light blocking particle counter module in the MicroLab.

Comparison to Other Techniques

The specific application and type of particles will often govern what is the best particle counting technique for the analysis. The three most commonly used particle counting techniques are light blocking (as used by the MicroLab), pore blockage, and direct imaging. Light blocking particle counters are widely used for many applications and are the traditional instruments used for in-service oil analysis. Pore blockage particle counters pass an oil sample through a fine mesh substrate. Constant flow or pressure pushes the sample through the mesh and the change in either the flow or pressure is measured as particles accumulate on the mesh. Direct imaging particle counters, like the LaserNet Fines (LNF) incorporate a solid-state laser configured with a CCD array in order to count particles and also classify according to size and shape.

MICROLAB REPORT AND EXPERT SYSTEM

The MicroLab report compiles all of the analytical data generated by the analyzer and includes up to three sets of historical results for trending. The results are color-coded to indicate abnormal or severe conditions. The report also provides diagnostic statements with recommended maintenance actions.

The Expert System of the MicroLab interprets the analytical data. It is built from over 20 years of industry knowledge and more than 10,000 individual software scripts to generate diagnostics that are specific to each application, the type of equipment and the type of oil. The system utilizes Rule Sets which are specific to each component type. These Rules Sets contain the threshold levels and diagnostics statements that are applied to sample results to generate maintenance recommendations. The system automatically factors

Sample history for trending analysis

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Account:	**MICROLAB SYSTEM TEST**
Address:	**1 EXECUTIVE DR**
	CHELMSFORD MA 01824
Phone:	
Email:	
Vehicle ID:	OVER-ROAD
Vehicle Make:	FORD
Vehicle Model:	EXPLORER LIMITED
Vehicle Year :	2017
Component ID:	OVER-ROADGSGENERAL
Component Type :	GASOLINE ENGINE
Oil Brand:	MOBIL
Oil Type:	MOBIL 1 FORMULA
Oil Weight:	5W30
Sump Capacity:	6 QUARTS
Viscosity Limit 40 Deg C	50 - 68
Viscosity Limit 100 Deg C	9.3 - 12.5

Diagnosis for current sample

HEAVY CONCENTRATION OF WATER PRESENT. CHECK FOR SOURCE OF WATER ENTRY. OIL DRAIN AND REFILL MAY BE NECESSARY. CONSULT SERVICE PROVIDER FOR FURTHER RECOMMENDATIONS. TO CONFIRM, RESAMPLE AT 5,000 MILES (8,000 KM) OR 100 HOURS.

Diagnostic statements with maintenance recommendations

Legend

ABNORMAL | SEVERE | X = NOT TESTED / NOT APPLICABLE | - = NOT DETECTED

NA = NOT AVAILABLE | C = CALCULATED | M = MEASURED

Analysis Results:	***Units***	***Current Sample***			
Sample ID		6	5	4	3
Date Analyzed		9/19/2017	9/19/2017	9/19/2017	9/19/2017
Date Sample Taken		9/19/2017	9/19/2017	9/19/2017	9/19/2017
Top Up	qt/gal/L				
Miles on Oil		7500	2500	7500	3850
Miles on Component		15000	10000	7500	3850
Oil Changed	Y/N	No	Yes	No	No
Oil Condition:					
Nitration	abs	<2.0	<2.0	8.1	-
Oxidation	abs	<2.0	<2.0	8.1	-
Total Base Number	mg KOH/g	8.9	9.4	3.9	3.9
Viscosity @ 100°C (M)	cSt	10.3	11.0	7.6	8.2
Viscosity @ 40°C (M)	cSt	64	65	46	48
Viscosity Index		148	162	132	145
Contamination:					
Glycol	%	-	-	-	-
Potassium	ppm	<2	<2	<2	<2
Silicon	ppm	<2	<2	<2	3
Sodium	ppm	<2	<2	31	24
Soot	%	<0.1	<0.1	<0.1	<0.1
Water	%	2.0	<0.1	3.0	>3
Wear Metals:					
Aluminium	ppm	<2	<2	<2	<2
Chromium	ppm	<2	<2	<2	<2
Copper	ppm	<2	3	8	12
Iron	ppm	<2	<2	15	17
Manganese	ppm	0	0	0	1
Molybdenum	ppm	<2	<2	52	59
Nickel	ppm	0	1	6	0
Lead	ppm	<2	<2	<2	<2
Tin	ppm	<2	<2	<2	<2
Titanium	ppm	0	0	0	0
Vanadium	ppm	1	0	0	0
Additives:					
Barium	ppm	0	0	0	0
Boron	ppm	41	31	25	27
Calcium	ppm	1524	1162	1508	1360
Magnesium	ppm	41	22	0	0
Phosphorus	ppm	744	663	814	756
Zinc	ppm	1004	779	1243	877
Additional Tests:					
Fuel Dilution	%	0.7	1.0	4.8	4.5
Total Acid Number	mg KOH/g	5.0	3.0	6.0	6.2
Total Ferrous	ppm	250.0	200.0	925.0	920.0
Water	%	2.0	0.0	3.0	3.2

Color-coded alarm limits

Results from external devices

in the usage time on the component and oil, break-in periods if applicable, and other important factors before comparing the results to custom threshold levels stored in the system's database when grading the results and providing the diagnostic statements.

For example: Given the same results for wear metals but varying the time on the component and oil, the resulting diagnostic statements will be different.

Report Results: **25 ppm Lead, 20 ppm Tin**

Time on Engine	Time on Oil	Alarm	Diagnostic Statement
35,000 miles	15,000 miles		NORMAL WEAR METALS DETECTED FOR BREAK-IN OR OVERHAUL PERIOD
375,000 miles	15,000 miles	Yellow	ABNORMAL BEARING WEAR DETECTED
375,000 miles	5,000 miles	Red	SEVERE BEARING WEAR DETECTED

Conclusion

Oil analysis is a critical part of any maintenance program, providing valuable information on the condition of the oil and then health of the equipment. To be most effective, oil analysis must be used when the equipment is being serviced so the technician can take immediate action. An oil analysis program lowers the risk of an unexpected failure and eliminates the costs involved with having equipment out of service. Oil analysis also allows mechanics to make decisions about oil drain extensions but this can only occur when the oil condition results are available at the time the equipment is being serviced – not days or weeks later.

The MicroLab is a useful trending tool, putting the information in the hands of the operators when they need it. The fully automated measurement, diagnostics and cleaning of the MicroLab make it possible for any company to have an on-site oil analysis program.

The MicroLab does not have the same detection capability as sophisticated laboratory equipment, but those analyzers require highly trained operators and need special sample prep and dedicated facilities. The key advantages of the MicroLab versus a traditional lab are rapid results for immediate decision making and the automation that gives anyone the ability to run the instrument. The MicroLab has demonstrated the ability to provide reliable trending data compared to outside laboratories and allows operators to make decisions faster by providing results in less than 15 minutes.

Spectro Scientific | One Executive Drive, Suite 101, Chelmsford, MA 01824-2563
978-431-1120 | www.spectrosci.com | sales@spectrosci.com | An ISO 9001:2008 company

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