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WHITE PAPER

# Techniques for Measuring Soot In Lubrication Oil

Photo courtesy of www.underhoodservice.com

Example of extreme soot build up

## Background

Since no engine is 100% efficient, products other than carbon dioxide and water will be formed during combustion. One such product produced from incomplete combustion is soot. Soot is a mass of mainly carbon particles that are typically spherical in shape. As soot levels rise, the soot particles begin to clump together and become more dangerous. The soot levels will continue to increase and the particles clump together until it reaches a level great enough to precipitate out of the oil. This precipitation will both increase the viscosity of the oil and attach itself to the engine surfaces which will significantly increase wear on the engine. This precipitation can also lead to filter plugging. Performing regular soot checks can realize cost savings by extending drain periods, reducing used oil disposal, and extending the life of diesel engines.

## Methods for testing soot in lubrication oil


Spectro Scientific InfraCal 2 Soot Meter

### FIXED FILTER IR ANALYZER

Fixed Filter IR analysis of soot samples is an ASTM approved test method (D7686) that is becoming a more popular technique. Using a fixed filter at a specific wavelength allows the analyzer to determine the percentage of soot in diesel engine oil up to 15% by weight. For this method, a fixed filter at 3.9 µm and a Horizontal Attenuated Total Reflectance (HATR) crystal are used for the measurement. The HATR crystal allows for much easier cleaning between samples. To complete a measurement, a sample of clean oil is applied to the HATR crystal and a background measurement is taken. The clean oil sample is removed from the crystal and then the sample of interest is homogenized, applied to the crystal and measured. It is important to measure the sample immediately since the soot will tend to settle onto the crystal and produce artificially high readings.

The instrument calculates the absorbance of the sample which is then converted to a concentration reading based on an internal

ADVANTAGES

- Provides fast analysis
- Can measure up to 15% soot
- Can be used in the lab or the field
- Does not require trained personnel
- Both analyzer and testing are inexpensive
- Follows ASTM D7686

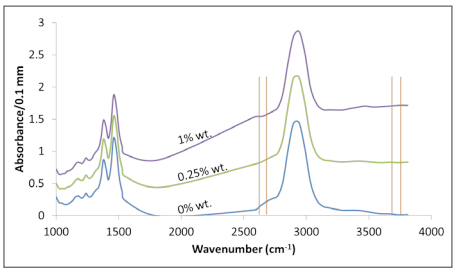
DISADVANTAGES

- Requires clean oil sample for background
- Sample must be homogenized
- Requires careful measurement of the sample
- Only measures soot

calibration. Done correctly this method is very precise, however not homogenizing the sample or allowing the sample to settle on the crystal can produce poor results. Overall, the use of a fixed filter and HATR crystal allows for quick, easy, and inexpensive measurement that can be done either in a lab or in the field.

Spectro Scientific FluidScan portable grating spectrometer

## GRATING IR SPECTROMETER




Four point calibration for soot measurement by D7889

Similar to an FTIR, grating spectrometers measure the light transmitted by a sample throughout the mid-infrared region. However, they use a diffraction grating to separate the light which allows for a system with no moving parts that is rugged, portable and inexpensive. Since grating spectrometers are measuring the same region of light as an FTIR, they can be used to measure many of the same oil properties as an FTIR. Following the ASTM method D7889, the soot concentration in the diesel engine oil is measured using a 4-point multivariate calibration. A weighted baseline is also used to minimize any errors from improper cell background collection and sample characteristics such as undissolved water and high TBN. When measuring a sample, a background of the blank cell is done first. Then the sample is placed in the cell and measured. The soot concentration is calculated by the instrument software using the multivariate calibration. The time required for a full analysis is less than three minutes.

ADVANTAGES
- Provides fast analysis
- Handheld
- Can measure multiple parameters
- Follows ASTM D7889

DISADVANTAGES
- Multivariate calibration susceptible to differences in base oil
- Limited range (up to 5%)

## THERMAL GRAVIMETRIC ANALYSIS (TGA)

Perkin Elmer Thermogravimetric Analyzer

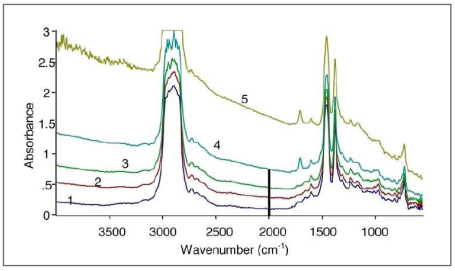
TGA is the traditional method for soot analysis as well as an approved test method from ASTM (D5967). The method uses heat to force reactions and physical changes in the sample, in order to provide a quantitative result of soot. The sample is placed into an oven where the weight is continuously monitored throughout the process. The sample is heated in the oven until all the organic material has evaporated and only the insoluble particles remain. The sample is kept under a blanket of nitrogen to prevent oxidation from taking place. Next, the nitrogen is replaced with oxygen and the sample is heated until the soot is burned off. All that is left at this point are the inorganic oxides. The soot concentration is determined by calculating weight difference of the sample before the oxygen is introduced and subtracting the weight of the sample after the soot is burned off. This method provides very precise results (within 0.1%) but it is a time consuming and labor intensive method.

ADVANTAGES
- Traditional method for the analysis
- Extremely precise
- Follows ASTM D5967

DISADVANTAGES
- Cannot be used in the field
- Requires trained personnel
- Analysis is expensive
- Time consuming
- Only measures soot


Spectro Scientific Q400 FTIR

## FTIR SPECTROMETER




Measurement of soot by ASTM D7844

FTIR (Fourier-Transform Infrared Spectroscopy) is an ASTM accepted (D7844) field technique for monitoring soot level in diesel engine oils that is both inexpensive and fast. FTIR spectrometers measure all the light transmitted by a sample in the mid-infrared region of the light spectrum (4000-400 $cm^{-1}$). This allows the instrument to measure the soot content of the oil, and to measure other properties such as oxidation, nitration, sulfation, water and glycol content. The method works by first placing a clean oil sample in an IR cell and measuring it on the spectrometer to establish a baseline. The clean oil is removed from the cell, replaced with the used oil sample, which is then measured on the spectrometer. Because the method measures the amount of IR light scattered rather than absorbed, the difference in baseline between the sample and the clean oil at 2000 $cm^{-1}$ is used to determine the soot value. This technique provides both fast and highly repeatable results, but will not always provide good precision. Because of this, the FTIR method is best suited for trend analysis in the field when monitoring soot.

ADVANTAGES
- Provides fast analysis
- Less expensive than TGA
- Can measure multiple parameters
- Follows ASTM D7844

DISADVANTAGES
- Best suited for trend analysis
- IR cell can be difficult to clean
- Limited range (up to 5%)

## Summary

There are several methods available for both laboratory and field analysis of soot in diesel engine oil. The choice of methods depends on budget, precision, location of testing, time, and other factors. The most important thing is to consistently monitor soot contamination in the diesel engine oil in order to avoid unexpected downtime and equipment failure.

Resources


- ASTM Standard D5967, 2013, "Standard Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine," ASTM International, West Conshohocken, PA, 2013, DOI: 10.1520/D5967-13 www.astm.org.
- ASTM Standard D7686, 2011, "Standard Test Method for Field-Based Condition Monitoring of Soot in In-Service Lubricants Using a Fixed-Filter Infrared (IR) Instrument," ASTM International, West Conshohocken, PA, 2011, DOI: 10.1520/D7686-11, www.astm.org.
- ASTM Standard D7844, 2012, "Standard Test Method for Condition Monitoring of Soot in In-Service Lubricants by Trend Analysis using Fourier Transform Infrared (FT-IR) Spectrometry," ASTM International, West Conshohocken, PA, 2012, DOI: 10.1520/D7844-12, www.astm.org.
- ASTM Standard D7889, 2013, "Standard Test Method for Field Determination of In-Service Fluid Properties Using IR Spectroscopy," ASTM International, West Conshohocken, PA, 2013, DOI: 10.1520/D7889, www.astm.org.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