

Using a Portable Infrared Analyzer to measure soot in diesel engine oil

By Paul Wilks

The soot is maintained in suspension by the dispersant additives of the lubricant. As soot levels increase, the viscosity of the oil also increases. At high levels, this suspended soot can become abrasive, and can result in wear in areas of high load, such as in the cam/cam follower region. Also, at higher levels, the soot may exceed the capacity of the dispersant additive, causing the soot to deposit in critical oilways, and in the oil filter - eventually leading to clogging of the oilways and plugging the filter. Thus, the soot concentration in the lubricating oil of a diesel engine is one of the key indicators as to the condition of the oil.

With the demand for extended drain times, combined with more stringent emission requirements, soot levels are increasing and the need to measure easily and accurately these higher levels is more important than ever.



Soot Level Testing Methods

There are five main methods in current use that can provide a measurement for soot content - either directly or indirectly. Each has its pros and cons - both in terms of success for soot measurement, and relative to use as a field test method.

#1 - The blotter test is a screening tool for visually evaluating residual dispersancy and soot level. It is subjective in evaluation and is limited for field use.

#2 - The total insolubles method is non-specific and may be a poor indicator for soot if other materials are present. It requires solvents, a centrifuge, an oven and an accurate laboratory balance.

#3 - Thermogravimetric analysis (TGA) tends to be the standard method of choice - and is the only direct measurement of soot as element carbon. For some, it is too involved and too lengthy to be considered as a routine method. Also, it tends to be a specialist technique and therefore has limited potential beyond the laboratory.

#4 & #5 - Optical methods (visible and IR) can be limited by the optical density of the medium, unless a sampling procedure is used to limit the level of light attenuated by the soot. Calibration against a laboratory method - such as TGA - is required.

Infrared Spectrum of Carbon

Carbon is a universal absorber of radiation over the entire visible and infrared wavelength regions. This is unique as most other materials only absorb in specific regions of the spectrum. This is the case in the infrared region where the organic and inorganic materials present in fully formulated lubricating oil provide a unique signature. This signature is known as the infrared spectrum, and is correlated to the specific molecular species present (in terms of frequency, cm^{-1}), and their relative concentrations - displayed in % transmittance to identify the species but converted to absorbance which is proportional to concentration for quantitative determination.

When comparing the spectra of a series of oils taken over time during the operation of an engine, it is possible to observe fine detail changes which are related to changes in chemical composition as the oil ages, and as different contaminants enter the lubricating system. When soot is formed in a diesel engine, it is observed in the lubricating oil spectrum as an overall suppression of the background. The effect increases in time as the soot content increases (Fig. 1).

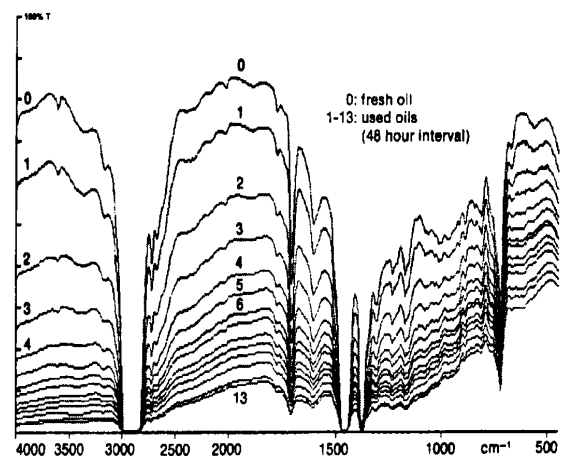


Fig.1. Mack Engine Test: demonstrating decrease in %T with soot content.

IR-ATR Permits High Concentration Soot Sampling

The InfraCal Soot Meter makes use of an infrared sampling technique called Attenuated Total Reflection (ATR) which has a number of advantages over visible and other infrared transmission methods. Because soot absorbs energy very strongly, extremely short path length sample cells are required in order to have enough signal transmitted to measure concentration level. Even using the thinnest practical absorption cells (about 100 μ m) the maximum measurable level of soot by visible or infrared transmission is less than 10% and sample dilution is often required. Thinner cells are possible, increasing the maximum soot level, but such cells are typically too thin to be easily usable in a production laboratory or on-site. The ATR method on the other hand, results in a very short effective path length and hence the ability to analyze samples in the 12% and higher concentration range which still maintaining sensitivity at low concentration.

(Fig. 2)

The InfraCal Soot Meter uses a sample platform fabricated from cubic zirconia, which is an extremely durable material nearly as hard as a diamond. With a built-in calibration, and simple push-button control, the analyzer is a turnkey device, requiring no special training or expertise in infrared analysis. The result is a field based system capable of producing results correlated to a standard method, such as TGA.

Soot measurements are made by coating the sample platform with a used oil sample from a dipstick or other sample collecting device. The results are displayed to the nearest 0.1% in less than 30 seconds. The infrared technique utilized in the InfraCal Soot Meter correlates well with TGA or FT-IR laboratory analyzed samples. Linear calibrations can be achieved out to 12% soot loading and beyond.

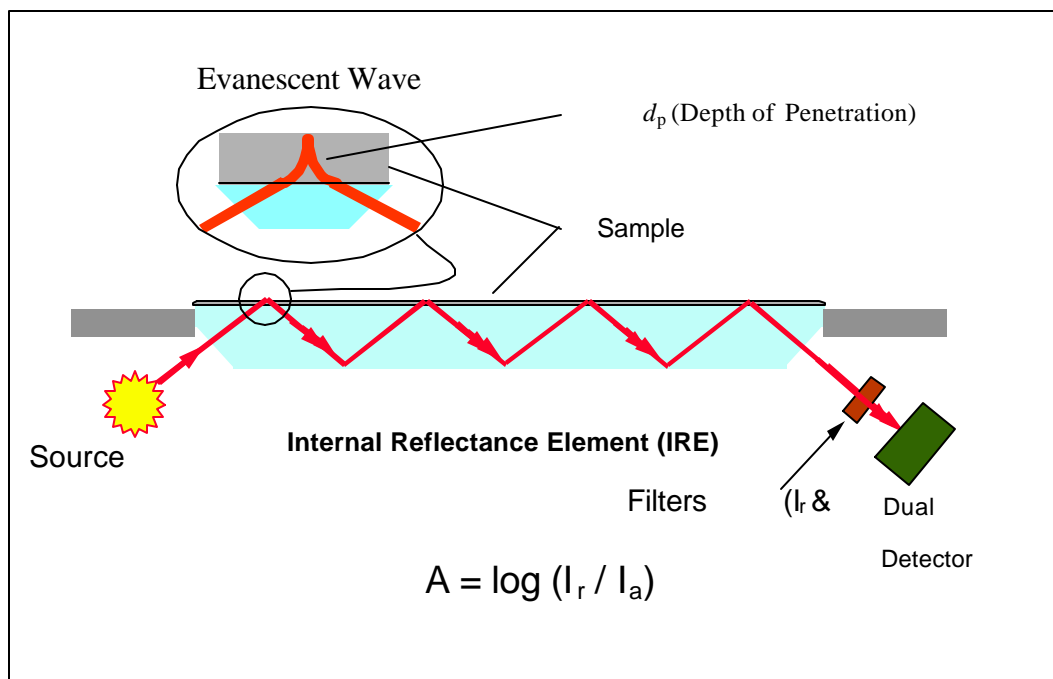


Fig. 2 - Optical Schematic of the IR-ATR sample system. Range and sensitivity are determined by the number of reflections which can be changed by varying the length or thickness of the internal reflection element.

Comparison of Data

IR-ATR vs. TGA and FT-IR data on a series of samples is shown in tables in Fig. 3. The correlation of the InfraCal Soot Meter with both methods is quite good. A similar comparison of measurements made by the InfraCal Soot Meter and an infrared spectrometer carried out at Texaco FLTD (Equilon) is shown in Fig. 4. The chart shows the linearity of the Soot Meter over the entire range. The results illustrate an impressive 99% correlation (R^2).

Time Per Analysis

The time per analysis with the InfraCal Soot Meter is less than 30 seconds. Users in oil testing laboratories report the Soot Meter easily handles samples 1500 to 2000+ samples per week.

Because of the analytical wavelength used, most oils have the same background absorption so that frequent re-zeroing of the instrument is not necessary. The sample is applied to the platform from a disposable syringe, a squeeze bottle or an engine dipstick. The platform is cleaned with a cloth or paper towel. There are no cells to clean and no solvents requiring special ventilation or expensive waste disposal.

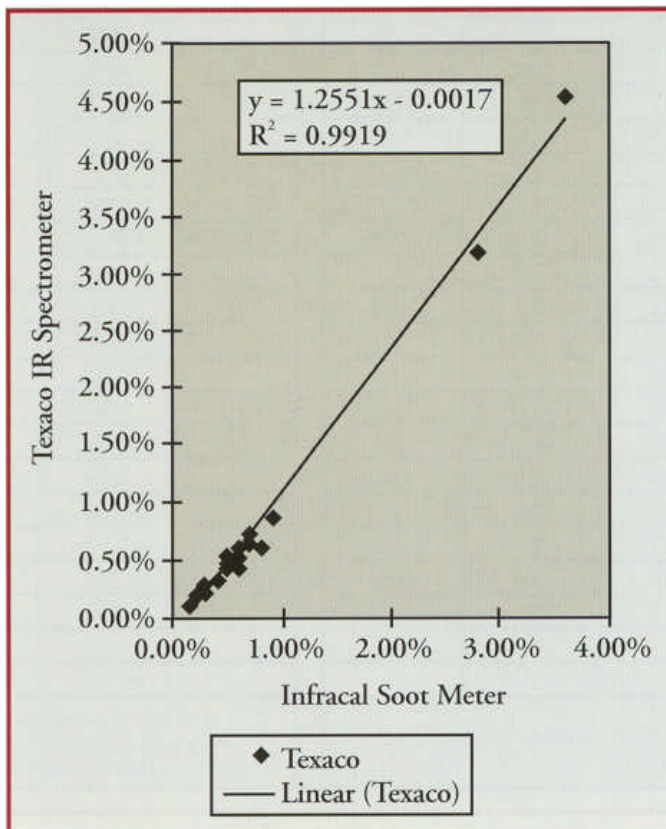


Fig. 4 - Comparison of InfraCal Soot Meter to near infrared analysis method.

| Field Test Samples | | | | | Lab Engine Test Samples | | | | |
|--------------------|-------|--------------|--------------|--------------|-------------------------|--------------|--------------|--------------|--|
| Sample ID | FT-IR | Soot, % mass | | | Engine Hours | Soot, % mass | | | |
| | | TGA | Soot Meter 1 | Soot Meter 2 | | TGA | Soot Meter 1 | Soot Meter 2 | |
| A1 | 1.46 | | 1.4 | 1.4 | Mack T-8 | | | | |
| B1 | 1.59 | | 1.6 | 1.6 | Hours: 0 | 0.13 | 0.1 | 0.0 | |
| B2 | 1.82 | | 1.8 | 1.9 | 50 | 1.76 | 1.7 | 1.7 | |
| B3 | 1.94 | | 1.9 | 2.0 | 100 | 3.13 | 3.2 | 3.1 | |
| B4 | 2.56 | | 2.5 | 2.5 | 150 | 4.48 | 4.5 | 4.4 | |
| A2 | 2.74 | 2.65 | 2.6 | 2.6 | 175 | 5.16 | 5.0 | 5.0 | |
| B5 | 2.68 | | 2.7 | 2.7 | 200 | 5.80 | 5.8 | 5.8 | |
| B6 | 2.81 | 2.76 | 2.8 | 2.9 | 225 | 6.38 | 6.1 | 6.0 | |
| B7 | 2.82 | 2.90 | 2.6 | 2.7 | 250 | 6.92 | 6.1 | 6.2 | |
| B8 | | 3.15 | 3.0 | 3.1 | DDC Series 60 | | | | |
| B9 | | 3.21 | 3.2 | 3.2 | Hours: 0 | 0.09 | 0.0 | 0.0 | |
| B10 | | 3.53 | 3.3 | 3.1 | 24 | 0.83 | 0.8 | 0.8 | |
| A3 | | 3.54 | 3.5 | 3.6 | 48 | 1.40 | 1.4 | 1.4 | |
| A4 | | 4.05 | 4.0 | 4.0 | 72 | 1.98 | 1.9 | 1.9 | |
| A5 | | 4.40 | 4.3 | 4.4 | 96 | 2.52 | 2.5 | 2.5 | |
| A6 | | 4.99 | 4.5 | 4.6 | 120 | 3.05 | 3.0 | 2.9 | |
| A7 | | 6.12 | 5.1 | 5.1 | 144 | 3.58 | 3.5 | 3.5 | |
| A8 | | 6.47 | 5.7 | 5.7 | 168 | 4.10 | 4.0 | 4.1 | |
| A9 | | 7.00 | 6.0 | 6.1 | 192 | 4.57 | 4.5 | 4.6 | |
| | | | | | 216 | 5.03 | 4.8 | 4.9 | |
| | | | | | 240 | 5.33 | 5.0 | 5.0 | |

Note: A & B Series Samples are different oil formulations
 Note: Soot Meter data was obtained using 2 production models.
 Each was calibrated to 5% soot, which accounts for measurement discrepancy in samples containing higher percentage.

Fig. 4 - Comparative soot measurements on used diesel oil samples*FT-IR, TGA and InfraCal Soot Meter.
 *Provided by Mobil Oil Co., Paulsboro Technical Center

Portability

The InfraCal Soot Meter is portable, weighs less than 5 lbs., and is roughly a 6-inch cube in size. Its rugged construction which is sealed against external contamination, plus no moving parts, make the InfraCal Soot Meter the perfect instrument for field-based operation. The use of a 12-volt power supply enables the InfraCal Soot Meter to be used literally anywhere - in the testing laboratory or right along side the engine. (Fig. 5) Field-based soot analysis can supplement for added assurance of equipment or vehicle performance while in service. With rapid and easy field screening made possible with the InfraCal Soot Meter, cost savings can be realized by extending drain periods, reducing needless disposal of used lubricants before the end of their useful life.

Determining “Time to Drain”

According to one fleet operator, it costs roughly \$1,500 in lost time to take a truck off the road to change the oil. Furthermore, the routes vehicles travel vary tremendously in engine loading, i.e. mountain routes vs. flat plains. Therefore, using “miles driven” as the criteria to drain does not make sense or is the most cost effective.

Other characteristics of used lubricating oils, such as TBN depletion or oxidation levels are certainly important in determining its condition. Because of increasing levels of soot, its concentration is probably the single most important condition that determines time to drain. Furthermore, with the IR-ATR sampling method, soot is the easiest oil characteristic to measure on-site - in maintenance shops, service stations, fleet parking areas, onboard ships or anywhere that rapid soot analysis is required.



Fig. 5 - InfraCal Soot Meter in the lab or on-the-road.

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