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WHITE PAPER

Guide to Measuring Fuel Dilution in Lubricating Oil

Background

Fuel dilution in oil can cause serious engine damage. High levels of fuel (>2%) in a lubricant can result in decreased viscosity, oil degradation, loss of dispersancy, and loss of oxidation stability. Fuel dilution is one of the most important lubricant failure modes in internal combustion engines. It usually occurs due to improper fuel-to-air ratio. Fuel dilution can also occur due to excessive idling, piston ring wear, or defective injectors and loose connectors.

Measurement Techniques

GC-MS courtesy of Thermo Scientific

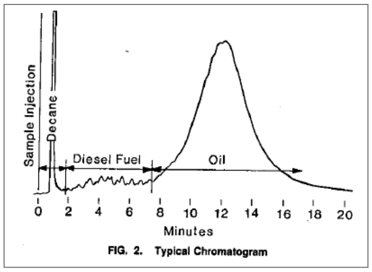
GAS CHROMATOGRAPHY

The most widely accepted direct method for analyzing fuel dilution in lubricants is Gas Chromatography (GC) according to ASTM methods D3524, D3525, and more recently, D7593.1-3 This method involves injecting a portion of an oil sample into a gas chromatograph. The GC vaporizes the sample and passes it through an analytical column that separates the sample into its component hydrocarbons in order of boiling point. Quantitation is obtained by integrating the area of fuel peaks as detected by an FID (Flame Ionization Detector). A calibration curve is created which associates the peak area to mass% of fuel in oil. Sometimes an internal standard is used (like decane for diesel) so a ratio of the integrated area of fuel peaks compared to the area of the internal standard peak is calibrated.

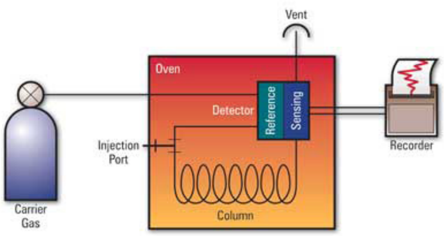
These methods can provide highly accurate results and are designed for high volume labs due to the fact that they are expensive and require experienced technicians. Getting the best result using GC analysis can be very time consuming since there are cases in which there is overlap in boiling points between fuel and certain engine oil formulations. These overlaps can introduce errors of up to 2% fuel dilution if left unchecked. In these cases, the temperature, pressure, or column may need to be optimized for the specific oil type. Many commercial laboratories have modified the traditional ASTM methods in favor of a faster and more robust method developed by a leading GC manufacturer.[4]

PROS:
- The industry standard
- Accurate
- Ideal for high volume labs
- Can detect biodiesel and ethanol

CONS:
- Must be performed in a lab
- Requires expensive equipment and gases
- Requires an experienced operator
- Can be time consuming for best results



ASTM D3524: Typical gas chromatogram of diesel fuel contamination in oil.



Schematic of a gas chromatograph. The sample is injected into the instrument and vaporized. It is then carried through the analytical column by an inert gas that separates the sample according to molecular structure (boiling point). The presence of each analyte is detected as it passes through the detector and exit vent of the instrument and is converted to signal peaks on a chromatogram.

VISCOSITY

Viscosity tests are routinely performed on a lubricant already as part of the basic suite of testing for lubricant condition. There are many methods and instruments available for both lab and field testing, including the Spectro Q3050 portable kinematic viscometer. The presence of fuel dilution in a lubricant can be detected indirectly through viscosity and will appear as a change from uncontaminated oil. However, if a lubricant exhibits a change in viscosity it does not exclusively conclude a fuel dilution issue. Many other lubricant problems also occur with a change in viscosity: lubricant degradation, contamination (water, coolant, soot), topping off with the wrong oil, etc. Viscosity is best used as a screening test to promote further testing if necessary on problem samples.

PROS:

- Lab instruments and portable instruments are available
- Already a routine test for lubricant condition
- Good screening test for possible fuel dilution
- Can detect biodiesel and ethanol

CONS:

- Cannot definitively point to fuel dilution problem
- Requires a careful operator

FLASH POINT TESTING

Setaflash Series 3 Closed Cup Flash Point Tester (ASTM D3828)

Flash point testing is a standard method that has been used for decades to evaluate both new and used lubricant.5 In a used oil sample, it can detect the presence of fuel dilution. The flash point is the lowest temperature at which an ignition source causes the vapors of the sample to ignite under set conditions. In the presence of lighter hydrocarbon fuel components in a lubricant, the flash point temperature will be decreased.

One popular method is ASTM D3828 also known as the Small Scale Closed Cup procedure. In this procedure, only 2 mL of sample is used, and the sample is heated quickly to a temperature lower than the new oil baseline by a preset amount for a pass/fail test (for example 25°C). Once the target temperature is reached, the ignitor is applied. If a flash is obtained the test indicates possible fuel dilution. For more quantitative results, multiple temperature points can be attempted and correlation tables that were generated based on known samples are used to convert the flash point temperature to a fuel dilution %. Because the fuel type or engine oil type may not be known, it is difficult to determine the actual percentage of fuel dilution that is present. For this reason, flash point testing is typically used as a qualitative pass/fail test.

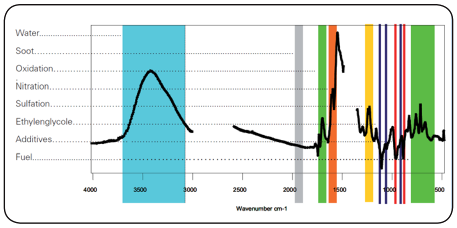
PROS:

- Pass/fail result is adequate for most applications
- Can detect ethanol
- Some instruments require only 1-2mL

CONS:

- Cannot detect biodiesel
- Requires a careful operator
- Requires the user to know the oil/fuel type for quantitative measurement
- Some danger due to heating fuel-laden samples
- Excessive water or glycol contamination can give false readings

FTIR SPECTROSCOPY



The spectrum above shows the difference spectrum of used-oil versus a new-oil reference. The areas (as marked) are used for the determination of the compounds.

Reference: "Used Lubricating Oil Analysis by FT-IR", Bruker Application Note #AN57.

FTIR spectroscopy offers a fast, user-friendly measurement for fuel dilution, however it is not easy to distinguish between fuel hydrocarbons and those present in the base oil. It is very difficult to achieve accurate results. Calibration requires large datasets of a narrow scope of sample type in order to develop complex algorithms correlating the spectrum to fuel dilution. For this reason, FTIR is not widely used for fuel dilution measurement.

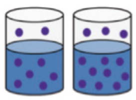
PROS:

- Low cost per sample after initial equipment purchase
- Test is quick

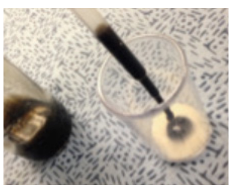
CONS:

- Requires expensive equipment
- Calibrations tend to be specific to a narrow sample type

Spectro Scientific Fuel Dilution Meter



The concentration of fuel vapor present in the headspace of a closed bottle at equilibrium is proportional to the amount of fuel present in solution.



Sample vial used to test small amounts of oil

SURFACE ACOUSTIC WAVE SENSING

The Spectro Q6000 Fuel Dilution Meter (FDM) uses a surface acoustic wave sensor (SAW) that reacts specifically to the presence of fuel vapor.6 It works by the principle of Henry's law. In a closed sample container, the amount of fuel diluted in the oil is directly proportional to the amount of fuel vapor in the headspace of a closed sample vessel at equilibrium.

One half mL of sample is placed onto a small pad of felt in the bottom of a disposable vial. The vial is left to equilibrate for one minute, and then the FDM uses a fang design to pierce through the cap and sample the vapor in the headspace. It is a simple, direct, portable instrument that can be used either in the laboratory or in the field. It can easily be used by reliability professionals without detailed training or experience. The range is from 0 – 15% fuel dilution with an LOD of 0.2% fuel dilution. The repeatability is <5% RSD and the accuracy is ±10% of the measurement (minimum 0.2% fuel dilution).

PROS:

- Portable
- Easy to use
- Requires only 0.5 mL of sample
- Test is quick
- Less expensive than GC
- Easily adapted to different oil/fuel types

CONS:

- Cannot measure biodiesel
- Must be calibrated with a reference fluid

Summary

Fuel dilution is a critical lubricant contamination issue that can result in expensive engine damage. There are several methods available to measure fuel dilution. Viscosity is a great screening method that is traditionally performed as part of the testing suite for used lubricants. Direct methods include GC, flash point testing, and SAW sensing. The best method to use depends on the application need.

References

1. ASTM Standard D3524-14. Standard Test Method for Diesel Fuel Diluent in Used Diesel Engine Oils by Gas Chromatography.
2. ASTM D3525-04(2010). Standard Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Gas Chromatography.
3. ASTM D7593-13. Standard Test Method for Determination of Fuel Dilution for In-Service Engine Oils by Gas Chromatography.
4. Noria Corporation. "Gas Chromatography: The Modern Analytical Tool," Machinery Lubrication. http://www.machinerylubrication.com/Read/352/gas-chromatography
5. Timothy D. Ruppel, Gerald Hall, and Andrew Tipler. "A Novel Method for High-Speed Determination of Fuel Diluents in Lubricating Oils," Field Application Report. Perkin Elmer, 2005, Pages 1-3.
6. Jim Fitch. "The Enduring Flash Point Test," Machinery Lubrication. http://www.machinerylubrication.com/Read/19/flash-point-test
7. Randi Price. "Accurate Fuel Dilution in the Field Using the Q6000 FDM: A Comparison to Gas Chromatography," Application Note, Spectro Scientific.

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