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era**flash** & fuel dilution

Comparison of using well-weathered or fresh diesel fuel for fuel dilution

Introduction

OCM – oil conditioning monitoring – describes processes involved in monitoring lubricants used in machinery, such as gear boxes or combustion engines. One important aspect of OCM is testing for contaminants, which are an early sign of equipment failure. Most common contaminants are wear-and-tear metals, typically iron or aluminum and other alloy metals thereof, or other contaminants which can accumulate over time like soot or water.

Another class of contaminants of considerable importance are fuels in lubricants, due to three reasons:

- Fuel contamination increases the fire hazard of the lubricant
- It is a clear sign of a failing gasket
- The lubrication properties can be negatively affected by a fuel contamination

According to ASTM E2412 (*Standard Practice for Conditioning Monitoring of In-Service Lubricants by Trend Analysis Using FT-IR*) fuels differ from lubricants especially in their molecular weight (boiling range) and their higher percentage of aromatic materials. While there are potentially many measuring techniques available for diesel fuel in lubricant detection, most of them do not provide a reliable solution. Because either diesel and lubricants can hardly be distinguished (IR, density), which is especially the case for synthetic fuels containing practically no aromatics, or the fuel concentration is typically too low to be safely detected for the measuring technique (distillation).

A quick and reliable approach is the flash point measurement. It uses the increased volatility of contaminated lubricants and exhibits an excellent reproducibility even at concentration levels far below 5%. Modern flash point testers typically provide a special measurement program called “fuel dilution” for this application. The test result is then expressed not only by the flash point but it also contains the fuel concentration as a secondary result.

The relationship between the flash point and the fuel concentration is non-linear, therefore it cannot be easily described by a simple correlation formula. Instead, a table is created containing various fuel concentrations, correlating a measured flash point to a known fuel concentration. For establishing this correlation table, several solutions with different known fuel concentrations have to be prepared and measured on a flash point tester.

While the flash points of fresh and used oils lacking any fuel contamination usually remain stable over time, any fuel observed as a contaminant is subdued to fundamental changes when exposed to elevated temperatures, e.g. in the lubricant of a combustion engine. The fuel typically loses a significant amount of its more volatile components, meaning its flash point is also affected.

Therefore, using fresh fuel for establishing the correlation table would potentially result in a systematic bias. Instead, *weathered* fuel is used for this purpose. This application note intends to discuss the difference between using well-weathered diesel fuel and fresh diesel fuel for the fuel dilution.

Preparation

An example how to prepare weathered fuel is described in section A2.2.7.4 of ASTM E2412-23a:

Diesel fuel is brought to approximately 107 °C then held at this temperature for 30 min. This represents a total loss of around 5% of the original volume.

A bottle containing 500 mL commercially available B7 diesel fuel (EN 590) was treated according to this procedure, which was then used for spiking lubricant samples in several concentrations ranging from 1 to 50 Mass%. Analogously, untreated diesel fuel was used for the preparation of the corresponding samples as well. For the sample matrix a typical 5W-40 C3 combustion engine lubricant was used.

Measurements

First, the flash points of the neat materials were measured to estimate the total temperature range of the contamination samples. Commercially available B7 diesel fuel has an expected flash point of approximately 60°C, therefore the neat diesel samples were directly measured according to the test method ASTM D7094 without getting a preliminary SCAN flash point result first. The flash point of engine lubricants is approximately 200°C but can vary depending on the lubricant type and manufacturer. Thus, the lubricant was first measured with the SCAN test method and a temperature range of 150-250°C.

This SCAN test method is based on ASTM D7094, but uses a faster heating rate of 10°C/min and an ignition interval of every 5°C. It is used for determining an approximate flash point of unknown samples. ASTM D7094 features a heating rate of 2.5°C/min with an ignition interval of every 1°C. It is therefore more precise than the SCAN test method but requires a known expected flash point due to the relatively small method defined temperature range of 28°C.

All the following measurements for the contaminated samples were conducted in the same sequence:

1. The SCAN method (range 60-220°C) was used to get a preliminary flash point.
2. The result obtained by SCAN was then taken as the expected flashpoint (*T exp*) for the subsequential and more precise measurement according to ASTM D7094.
3. To estimate the repeatability, a second ASTM D7094 measurement was done for each sample.

The results of all measurements are displayed in the graph below and also in the table on the following page (figures 1 and 2):

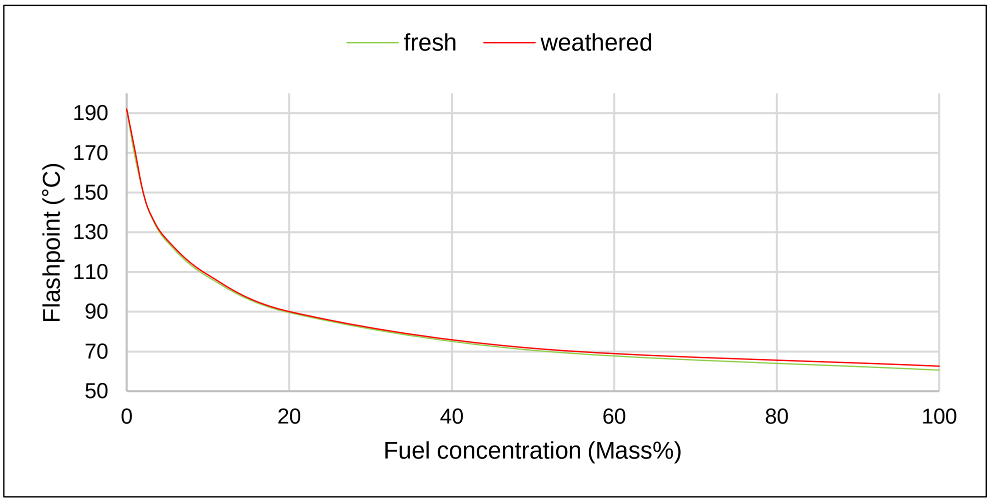

Figure 1: measured flash points in dependance of the fuel concentration

Sample	SCAN (°C)	1st D7094 (°C)	2nd D7094 (°C)	Average (°C)
5W-40	195.7	191.6	192.6	192.1
1% fresh	170.7	168.6	168.7	168.7
1% weathered	170.7	172.7	170.7	171.7
2% fresh	150.7	150.7	150.7	150.7
2% weathered	150.7	150.7	150.7	150.7
3% fresh	140.7	137.7	138.7	138.2
3% weathered	140.7	138.7	138.7	138.7
5% fresh	125.7	124.7	125.7	125.2
5% weathered	125.7	126.6	125.6	126.1
10% fresh	110.6	107.6	107.6	107.6
10% weathered	110.6	108.6	108.6	108.6
20% fresh	90.6	89.6	89.6	89.6
20% weathered	95.6	90.6	89.6	90.1
50% fresh	70.6	70.6	70.6	70.6
50% weathered	75.6	71.6	71.6	71.6
fresh B7 diesel	-	60.6	60.6	60.6
weathered B7 diesel	-	62.6	62.6	62.6

Figure 2: flash point results obtained by SCAN and ASTM D7094

The mean flash point result of the two ASTM D7094 measurements was calculated for both the fresh and weathered samples and is displayed in the column “Average”. This average value was used for creating the fuel dilution table.

After specifying the temperature range and the dilution table in the profile settings on the analyzer, a quick verification measurement was done with the 2% weathered sample and the SCAN test method. The test result was spot-on:

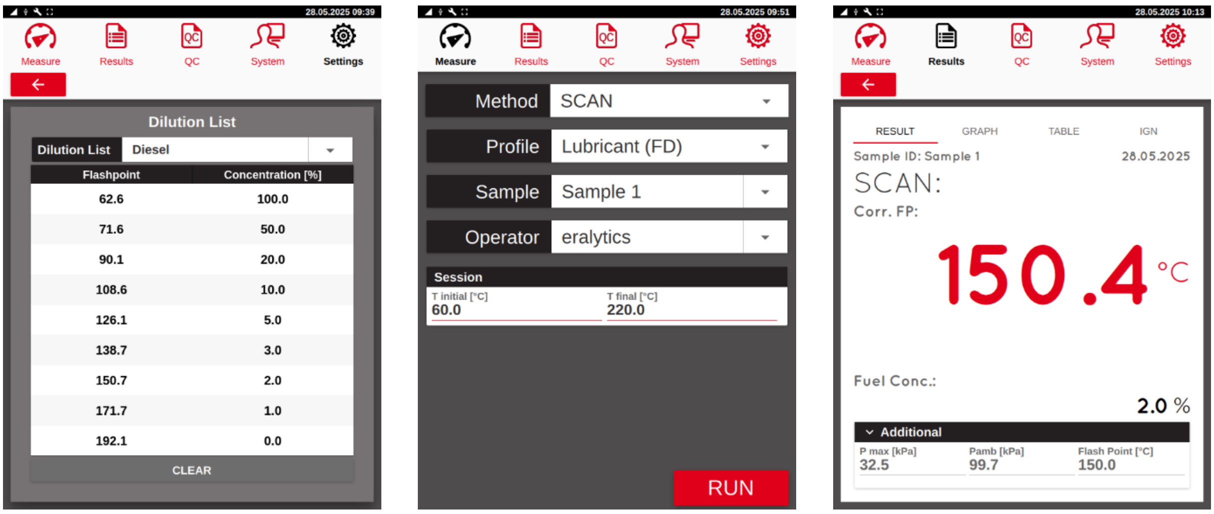

Figure 3: screenshots of era**flash** application software used for a fuel dilution measurement

Visible in the screenshot to the right of figure 3, the flash point is shown as the main result, and the corresponding determined fuel concentration is displayed as the secondary result below.

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Discussion

Clearly visible in the table and the graph (figure 1 and 2), there is hardly any difference between the flash points of the fresh and the weathered diesel samples. The preparation procedure described in ASTM D2412 mentions a loss of 5 Vol% upon treating the diesel for 30 min at 107°C, but a loss of volume was not observed.

At least for the investigated diesel fuel it does not make any difference whether to use it “as is” for establishing the fuel dilution table, or to artificially weather it. It can be argued that the results obtained by unmodified diesel will be accurate enough for this application, but to be on the safe side better use the treated diesel.

The repeatability of both the fresh and weathered samples is exceptionally good, and the preliminary SCAN results are very close to the more accurate ASTM D7094 test results, in some cases even identical.

Therefore, for this application – getting an estimated fuel concentration in used oils for OCM – the SCAN test method provides a more than acceptable accuracy in combination with a fast flash point measurement over the total temperature range of 60-220°C. The advantage of using SCAN instead of ASTM D7094 is that it allows for using *one* profile with the specified concentration table, independent from the expected flash point of a sample with an unknown concentration.

Conclusion

Using era**flash** for OCM – oil conditioning monitoring – features several advantages over other measuring techniques, and the findings in this application note can be summarized as follow:

- The flash point is an easy and reliable way to determine the fuel concentration in used oils for OCM.
- The flash point is much more accurate compared to FT-IR measurements in this regard.
- The small thermal mass and the unique PBT™ installed in era**flash** allow for very fast measurements and short turnaround times in general.
- Due to the inherently safe apparatus design of MCCCFP testers (modified continuously closed-cup flash point = ASTM D7094) contaminated samples can be measured in high precision and without any fire hazard.
- Dependent on the diesel type untreated diesel can be used for establishing the concentration table.
- Modern flash point testers such as era**flash** automatically provide the fuel concentration as the secondary test result, making the actual measurement and data interpretation a simple task.