WHITE PAPER

FUEL DILUTION IN ENGINE CRANKCASES: A FAST FIELD METHOD FOR THE WORKSHOP

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Synopsis

The Spectro Scientific Q6000 portable fuel dilution meter provides rapid and accurate measurements of fuel contamination in engine oil. Because of its small size, it is ideal for field use or use in the laboratory. This fuel dilution meter is designed for use by a technician or maintenance personnel, and requires no chemicals, making it inexpensive to use as well as fast and accurate.

The focus of this paper is to provide an overview of fuel dilution, details on the innovative combination of sampling and sensor design and validation data.

What is fuel dilution and why is it a problem?

Fuel dilution in oil is a condition caused by excess, unburned fuel mixing with engine oil in an engine crankcase. Hydrocarbon-based fuel, usually with a lower vapor pressure than the lubricant, has a thinning effect, lowering the oil viscosity. Oil film strength is reduced, increasing the cylinder liner and bearing wear.

Reciprocating engines based on Otto and Diesel configurations are not completely efficient in combusting fuel. Timing, injector quality, and fuel properties play a role in engine efficiency. In recent years, engine makers' efforts to meet stricter emissions requirements have led to widespread use of diesel particualte filter (DPF) designs with passive regeneration. Engine makers have tried several approaches to raise the exhaust gas temperatures to burn off the fuel soot. The most widely used approach is late in-cylinder fuel injection. Fuel is injected very late in the cycle, typically near the exhaust valve opening. The fuel vaporizes but does not oxidize in the cylinder, thus generating unburned hydrocarbons. The hydrocarbons will oxidize over the diesel oxidation catalyst (DOC) thereby raising the exhaust gas temperature. This hot exhaust gas then flows through the DPF and oxidizes the trapped soot. This late injection approach was quite popular in recent years as a low cost approach by engine OEMs. However, an increase in fuel dilution has been observed across user communities.

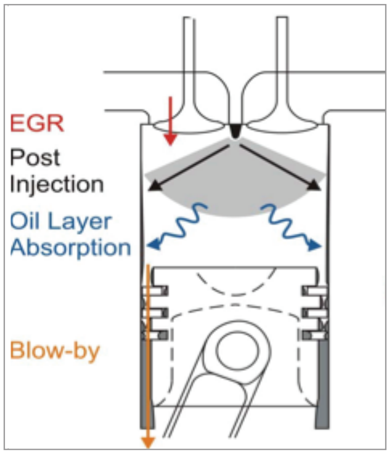


Figure 1: Sources of excess fuel in engine lubricants are EGR (exhaust gas recirculation, post injection (late) cycle, oil layer absorption and blowby past the

Existing Approaches to detecting fuel dilution

Gas Chromatography

Fuel dilution in an oil has a distinctive odor, readily identified by the human nose. Quantifying the amount of fuel in oil is difficult- the lighter molecular weight fuel is miscible in oil. Traditional approaches range from direct methods such as gas chromatography and SAW sensors to indirect methods such as flash point and viscosity measurement.

Most oil testing labs use gas chromatography (GC) to determine fuel content in oil and modified versions of ASTM standards. A GC Flame Ionizer Detector (FID) with headspace analysis is most commonly employed. GC-MS, a method that combines the features of gas-liquid chromatography and mass spectrometry, is also used to identify different substances within a test sample. It is considered the "gold standard" for forensic substance identification because it is used to perform a specific test. A specific test positively identifies the presence of a particular substance in a given sample. Gas chromatography is a laboratory technique that requires trained technicians and is not a portable tool that may be employed in the field.

Flash Point Measurement

As volatiles increase, the flash point of a lubricant will decrease. Manual flash point testers are often employed in a screening role in labs, with an improvised method to speed up the test, as the official methods can take 30 minutes to complete. Flash point testers are difficult to employ in a workshop with untrained operators as basic systems with a naked flame are discouraged in fueling areas and a low flash point result can be caused by oil sheardown, oil mixup, or masked by contaminants such as glycol/water in the the oil.

Viscosity Measurement

Viscosity measurement is effective when fuel dilution ranges are above 5%. The SAE viscosity range is broad enough that fuel dilution up to 5% can be tolerated. Synthetic motor oils maintain their grade better than mineral oils. For dual fuel engines that burn both low and high density fuels, the viscosity may not change at all, hiding a fuel dilution problem. Other lubricant conditions can also cause a drop in viscosity. Because of this, viscosity change alone is typically not a good indicator of fuel dilution, especially for low vapor pressure fluids.

Other methods:

FTIR Spectroscopy and Blotter tests are used to detect fuel dilution also. IR spectroscopy is very sensitive, and offered by service labs as part of the physical property/contamination screen. It is mostly used as a screen test, due to sampling challenges, and a confirmatory test such as GC is employed. Blotter tests are simple chromatography tools purely used as a non quantitative screening method.

Figure 2: First generation "fuel sniffer" fuel dilution test.

History of fuel dilution in the field and SAW sensors

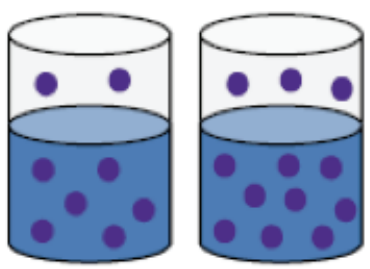
The need for fuel dilution measurement on board ships was identified by the United States Navy almost 20 years ago, and this need drove the Naval Research Lab to sponsor development of a product based on the Surface Acoustic Wave (SAW) sensor. Chemical micro sensors were first proposed as on-site and in-situ tools for engine and lubricant condition monitoring at the Joint Oil Analysis Program Conference in 1983. In the spring of 1990, the Naval Ship System Engineering Station (NAVSSES) initiated a program to develop a portable SAW-based instrument to measure the fuel dilution in shipboard diesel engine lubricants. Microsensor Systems Inc. and the U.S. Navy combined their experience and expertise to design and build a small, rugged, reliable instrument based on the SAW sensor. After an extensive evaluation and testing period, the US Navy purchased 100 fuel dilution meters "fuel sniffers" for use aboard ships and they are still in use today.

The first generation meter, known as a "fuel sniffer", is in widespread use globally oustide of military applications, such as mining operations and service labs. The fuel sniffer is used as a screening technique to determine if a more expensive GC analysis is required. The fuel sniffer is also used extensively by railroads and engine-based power plants. The fuel dilution meter satisfies the need for quick, easy direct measurement of volatile fuels and is simpler to use and operate than other methods to determine both field- and lab-based dilution (see Table 1).

TECHNIQUE	ADVANTAGE	LIMITS
Gas Chromatog-raphy	Referee Method (ASTM D 3525) Very accurate and repeatable	Not portable High cost Requires trained technicians
Flash Point	Flash point and fuel dilution correlation well known	Indirect method Open flame! Additives and anti-freeze can interfere
FTIR	Part of lab test screen	Sooty samples interfere, as do aromatics in oil
Blotter	Simple	Subjective, water and glycol interfere
Q600 Sniffer	Direct measurement, easy to use, accurate	Oil temperature caused imprecision

Table 1: Summary of Techniques

Why SAW Sensors for Fuel Dilution?



$$P_{gas}=k_H C$$

P_{gas}: partial pressure of gas in headspace
k_H: Henry's law constant
C: concentration of solute

Figure 3

Fuel dilution meters employ a Surface Acoustic Wave (SAW) vapor sensor to measure the concentration of fuel in used oil samples by sampling the "headspace" in a vial or bottle. The device assumes (based on Henry's Law) that the fuel concentration in the headspace vapor is directly related to the fuel present in the oil sample. A SAW sensor consists of a piezoelectric substrate that has an interdigitated electrode lithographically patterned on its surface. The surface of the SAW sensor has a polymer coating that is chosen to offer specific solubility to fuel vapors. The mechanism of detection is a reversible absorption of the fuel component into the polymer. When this device is excited by external RF (Radio Frequency) voltage, a synchronous Rayleigh wave is generated on the surface of the device. When fuel contamination comes in contact with the SAW sensor surface it will absorb into the polymer coating. This absorption into the polymer

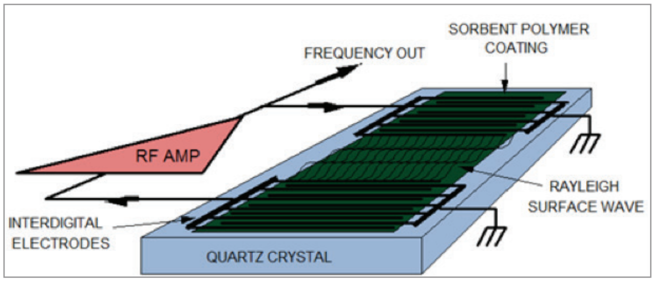


Figure 4: SAW sensors capture the response of a species specific coating and convert it to an electrical signal of equal magnitude.

causes a mass change which produces a corresponding change in the amplitude and velocity of the surface wave. When used in a self-resonant oscillator circuit, the change in Rayleigh wave velocity resulting from vapor absorption into the polymer coating causes a corresponding change in oscillator frequency. This change in frequency is the basis of detection.

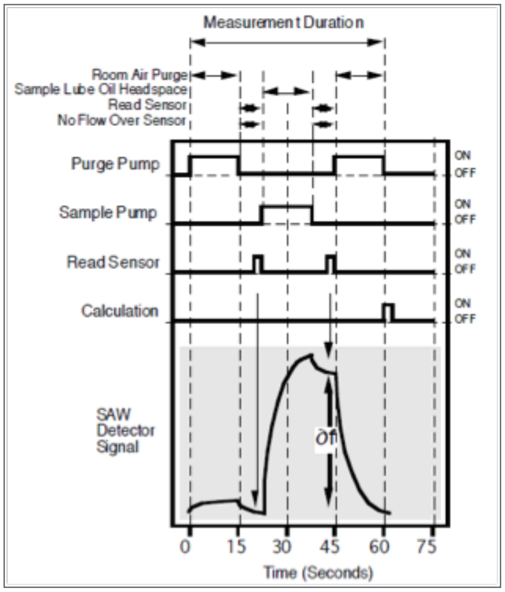


Figure 5: Typical measurement cycle and response time for fuel dilution meter

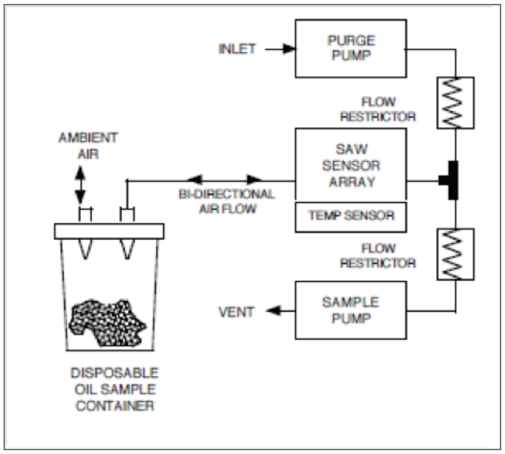


Figure 6

A newer design – Q6000 fuel dilution meter

Using the above concepts, a next generation fuel dilution meter was developed in 2014. The Q6000 FDM meter (Figure 7) was introduced based on the success of the first generation model and feedback from users in the field.

Figure 7

Headspace Innovation

A significant new feature of the Q6000 FDM design is the fang-like headspace sampling design as shown below. A common concern with the older design was the tendency for customers to saturate the SAW sensor with oil drawn into the tubing from overfilled bottles (little or no headspace). The solution to this was to change how the headspace is presented to the sensor. A small oil sample (0.5 mL) is dispensed into the sample bottle. The sample bottle has an absorbent layer on the bottom to hold the oil. The bottle cap is put on and the bottle is placed into the FDM. When the cover is closed, the fangs penetrate into the sample bottle and begin to collect vapor to analyze from the headspace above the oil sample. This patent pending design ensures the headspace is quickly and accurately measured with excellent repeatability and accuracy.

Calibration and Operation

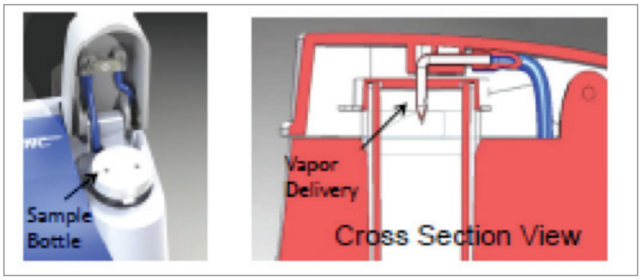


Figure 8: New headspace design improves repeatability and lowers risk of fuel carryover into sensor

The fuel dilution meter must be calibrated with an oil standard concentration containing a known content fuel in a lubricant base similar to the samples to be analyzed, e.g. 5% fuel in 15W 40 motor oil. Customers can prepare their own calibration standards as long as the oil and fuel used to create the standard is of the same matrix, or similar matrix, to that being measured. The instrument response to fuel dilution is linear over the measurement range. The fuel dilution meter can hold one or more calibrations depending on the model chosen. A single-point calibration, using a 5% fuel/oil standard, takes only minutes to perform, enabling quick measurements. The accuracy is comparable to that achieved using gas chromatography, but without all the time, expense and effort.

After measurement the percentage fuel dilution is displayed on the color touchscreen and can be sent to an external computer via mini USB output to record the measurements.

Repeatability and Accuracy

A validation study was performed using seven different Q6000 FDM analyzers (Figure 2) to mimic the situation that would be experienced by a large fleet with units deployed in multiple areas and with multiple users. Seven volunteers with both technical and non-technical backgrounds performed the testing on seven different units. There were 15 test samples randomly numbered with blind duplicates. The instrument has three slots to store calibrations. At the beginning of the test, each user calibrated the instrument with the calibration standards:

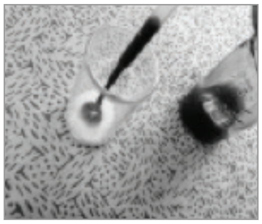


Figure 9

- A 5.0% gasoline in base oil standard
- A 5.1% diesel in clean 5W30 engine oil standard
- 5.0% JP-8 in MIL-PRF-23699. For each test sample, the user was instructed to use one of the stored calibrations: “Diesel”, “Gasoline”, or “Other”.

The users were instructed to prepare the sample vials by dispensing 0.5 mL of sample onto the felt disc using a graduated transfer pipette. The vials were capped and let to sit for at least a minute prior to analysis. It was noted that some users chose to make up several samples at once and other users chose to make each sample up one at a time using a stopwatch to carefully mark the one minute equilibration time.

Each user-instrument pair was treated as a separate laboratory for the purpose of the precision statistics. ASTM E691 was used as a guide (Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method). The precision statistics are shown in Table 2. For samples up to 3% fuel dilution, both the repeatability (r, 95%) and the reproducibility (R, 95%) were within 1% fuel dilution indicating that the measurement is very reliable.

PRECISION STATISTICS								
Fluid	Calibration	p	Average of User Averages	StDev of User Averages	Sr	SR	r, 95%	R, 95%
S1	Gas	7	2.20	0.15	0.17	0.19	0.46	0.53
S2	Gas	7	5.59	0.65	0.26	0.68	0.72	1.90
S3	Gas	7	11.64	0.57	0.42	0.64	1.17	1.79
S4	Gas	7	1.28	0.14	0.20	0.20	0.54	0.55
S5	JP-8	6	0.33	0.05	0.06	0.07	0.16	0.18
S6	JP-8	6	2.83	0.24	0.26	0.30	0.74	0.85
S7	JP-8	6	3.67	0.93	1.13	1.23	3.17	3.43
S8	JP-8	6	0.14	0.08	0.07	0.09	0.18	0.26
S9	Diesel	7	0.29	0.24	0.27	0.30	0.74	0.85
S10	Diesel	7	5.90	1.20	1.59	1.65	4.45	4.60
S11	Diesel	7	0.99	0.22	0.21	0.27	0.57	0.74
S12	Diesel	7	1.41	0.22	0.23	0.27	0.63	0.76
S13	Diesel	7	1.01	0.27	0.15	0.29	0.42	0.80
S14	Diesel	7	9.80	1.31	0.96	1.48	2.68	4.13
S15	Diesel	7	6.25	0.67	0.65	0.81	1.81	2.26

Table 2: Precision statistics of the internal r&R study performed on the Q6000 FDM. Fluid is the Sample # measured in blind duplicate by each user-instrument pair.

"Calibration" is the calibration standard used to calibrate the instrument.

"p" is the number of user-instrument pairs measuring that sample.

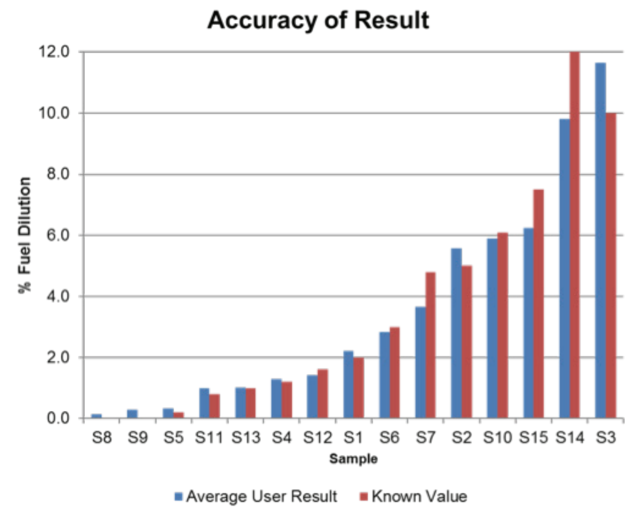
"Average of User Averages" and "StDev of the User Averages" are the average and standard deviation of the average sample results by each user-instrument pair.

"Sr" is the repeatability standard deviation.

"SR" is the reproducibility standard deviation.

"r, 95%" is the 95% repeatability.

"R, 95%" is the 95% Reproducibility.



Sample #	Fuel Type	Lubricant Notes	Ref. Value Source
S8	JP-8	-----	Gravimetric
S9	Diesel	-----	Gravimetric
S5	JP-8	-----	Gravimetric
S11	Diesel	Sooty	Gravimetric
S13	Diesel	-----	Gravimetric
S4	Gas	-----	Standard
S12	Diesel	Sooty	Gravimetric
S1	Gas	-----	Standard
S6	JP-8	-----	Gravimetric
S7	JP-8	-----	Gravimetric
S2	Gas	-----	Standard
S10	Diesel	Sooty	Gravimetric
S15	Diesel	-----	Gravimetric
S14	Diesel	-----	Gravimetric
S3	Gas	-----	Standard

Figure 10: Average user result compared to the known reference value for each test sample.

Discussion

Each test sample had a known fuel dilution reference value. Some of the test samples were commercially available standards, and others were prepared gravimetrically from new and used lubricants. The accuracy of the average user result compared to the known reference value is shown in Figure 10. Sensitivity to read samples <1% fuel dilution was achieved. Samples S8 and S9 were 0% fuel dilution samples. The published LOD is 0.2 % fuel dilution. Overall, the accuracy of lubricant samples with up to 3% fuel dilution was within 0.3% demonstrating that very good accuracy can be achieved by the average user. Even samples S10, S11, and S12 which were sooty used diesel engine oils that were measured reliably.

Conclusion

Measuring fuel dilution in engine oil is a continuing challenge for equipment owners. The need for a reliable field based method for accurate fuel dilution measurements is fulfilled by SAW sensor based tools. SAW sensor based devices have proven themselves over the years as effective screening tools to detect fuel dilution in lubrication oils. A next generation fuel dilution meter has improved on earlier designs by using smaller samples that are no longer prone to spilling, thus keeping the head space free of liquid, which can ruin the measurement. A multi-unit, multi-user study confirmed the robustness of the approach and device for meeting the specifications.

The Q6000 FDM provides a cost-effective, portable solution for detecting fuel dilution in oil samples.

Benefits of the technology

- Easy to use in the field or workshop

- Solvent free

- Fast and very repeatable

- Low limit of detection

- Range to 15%

- Calibrate to multiple fuels

It is substantially less expensive to use for single point calibrations than GC-MS and provides repeatable and accurate results. This technique requires no chemicals or expensive consumables and produces results more quickly than GC-MS without sacrificing precision.

In summary, the method is a low cost, easy to operate alternative to GC-MS and Flash Point methods for fleet operators looking to detect excessive fuel dilution in the field. It is ideally suited for customers requiring low cost, accurate and rapid analysis of fuel dilution in single point oil sample tests.

References

Wattrus, M., "Fuel Property Effects on Oil Dilution in Diesel Engines," SAE Int. J. Fuels Lubr. 6(3):2013

D. Ljubas, H. Krpan, I. Matanoviæ Influence of engine oils dilution by fuels on their viscosity, flash point and fire point NAFTA 61 (2) 73-79 (2010)

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