Designation: D8004 – 15

Standard Test Method for Fuel Dilution of In-Service Lubricants Using Surface Acoustic Wave Sensing[1]

1. Scope

1.1 This test method describes a means for determining the amount of fuel dilution present in an in-service lubricant. This is achieved by drawing into a surface acoustic wave (SAW) sensor vapor from the lubricant. Fuel vapor will be absorbed by the SAW sensor's polymer coating. The amount of absorbance is then related to fuel content in the lubricant.

1.2 The range of fuel dilution capable of being measured by the test method is from 0.1 % to 10.0 % by mass fuel dilution.

1.3 This test method is specifically tailored to determining the fuel dilution of in-service lubricants, including newly utilized lubricants. The method is applicable to contamination with diesel, gasoline, and jet fuels.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*See Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E456 Terminology Relating to Quality and Statistics
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
D7235 Guide for Establishing a Linear Correlation Relationship Between Analyzer and Primary Test Method Results Using Relevant ASTM Standard Practices
D7593 Test Method for Determination of Fuel Dilution for In-Service Engine Oils by Gas Chromatography
D7669 Guide for Practical Lubricant Condition Data Trend Analysis
D7720 Guide for Statistically Evaluating Measurand Alarm Limits when Using Oil Analysis to Monitor Equipment and Oil for Fitness and Contamination

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *fuel dilution sample holder, n*—a bottle that contains the lubricant to be analyzed. For example, this may be a standard 125 mL bottle (see Fig. 1, example configuration A) or a standard 30 mL laboratory vial (see Fig. 2, example configuration B).

3.1.2 *fuel dilution sample inlet, n*—a tube that connects the sample to the SAW sensor.

3.1.3 *fuel dilution sample stand, n*—mechanical device for holding the bottle of lubricant in the SAW fuel dilution apparatus in a way such that the headspace from the bottle is directly fed into the SAW element.

3.1.4 *fuel dilution seal, n*—a mechanism that seals the fuel dilution sample holder to the vapor path leading to the SAW sensor.

3.1.5 *SAW fuel dilution apparatus, n*—a device that measures fuel dilution using surface acoustic wave (SAW) technology. This is achieved by drawing vapor from the lubricant into a surface acoustic wave (SAW) sensor. The fuel dilution apparatus measures the concentration of contaminating fuel vapor present in the air "headspace" over the lubricant. The fuel dilution apparatus assumes that this headspace fuel vapor concentration is directly proportional to the fuel present in the oil. This relationship is based on Henry's Law. As fuel contamination builds up, a vapor concentration will be established in the headspace that is directly proportional to the concentration dissolved in the oil. The fuel dilution apparatus uses a SAW sensor to make these measurements.

3.1.6 *SAW sensor, n*—consists of a piezoelectric substrate that has an interdigitated electrode lithographically patterned on its surface. The surface of the SAW sensor has a polymer

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.96.02 on Chemistry for the Evaluation of In-Service Lubricants.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

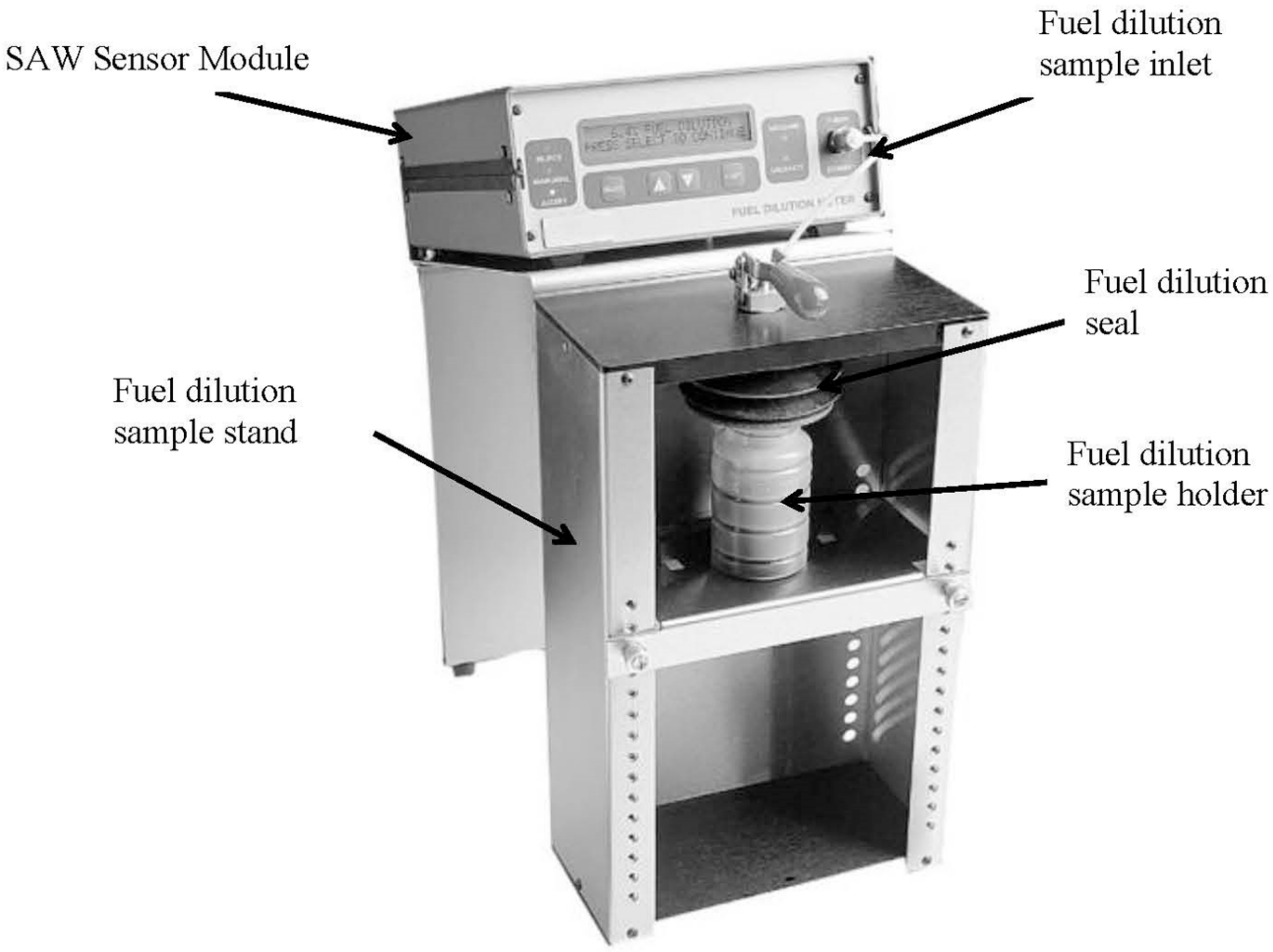

FIG. 1 Example SAW Fuel Dilution Apparatus—Configuration A

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 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 of the fuel dilution apparatus.

3.1.7 *surface acoustic wave (SAW), n*—a mechanical deformation travelling on the surface of a material; such a deformation may be converted into electrical signals using a piezoelectric material, which generates a voltage in response to a mechanical deformation.

4. Summary of Test Method

4.1 A liquid sample is placed into the fuel dilution sample holder (see Figs. 1 and 2) of the SAW fuel dilution apparatus and fuel dilution (percent by mass) is determined.

4.2 The vapor headspace of the sample, equilibrated at room temperature, is drawn into the SAW sensor by means of a diaphragm pump, which draws the vapor into the chamber of the SAW sensor.

4.3 The SAW sensor registers the buildup of mass on its polymer absorbent coating over a period of approximately one minute.

4.4 Based on this mass buildup and a calibration, fuel dilution (percent by mass) for the sample is determined.

5. Significance and Use

5.1 This test method provides a means for a reliable field determination of fuel dilution that is quick and preparation-free. Results are obtained in approximately 1 min. Such a

FIG. 2 Example SAW Fuel Dilution Apparatus—Configuration B

method is used, for example, at remote railroad depots where it is impractical to carry out a standard laboratory method for determination of fuel dilution, such as described in Test Method D7593, but it is a critical need to determine if fuel has contaminated the lubricant. If fuel has contaminated the lubricant, this is significantly detrimental to the machinery and it is typically serviced immediately. Further, the fuel can ignite at the high temperatures encountered in machinery lubricant paths.

6. Interferences

6.1 Departures in temperature between the sample under test and the sample which was used in the calibration by more than 2 °C will affect the precision of the results. This is due to the fact that the fuel vapor pressure is temperature dependent. For example, if calibration was performed with the fuel standard at 20 °C and the measured samples that are at 30 °C, the fuel dilution determination would be approximately 50 % high.

6.2 Creating a standard using fresh, rather than aged, fuel will cause the method to underreport actual measured values.

6.3 Possible interferences include the introduction of biodiesel variations or other additives in the fuel after it has been calibrated on the SAW fuel dilution meter. If the sample is calibrated with fuel containing the expected amount of biodiesel or additive, no error in the measurement should occur. If measured with an unexpected amount of biodiesel higher than that calibrated with, the apparent reading will be lower. This is because the biodiesel is typically less volatile than the fuel being measured. For example, if a calibration was performed on a 5.0 % fuel sample with no biodiesel, then a sample of that same fuel containing 10 % biodiesel (B10) is diluted into the lubricant at a 5.0 % level, the reading will be approximately 4.5 %. Thus, unexpected additional amounts of biodiesel can lead to false negative indications of fuel contamination if the apparatus is not calibrated with that same fuel. Similarly, unexpected lesser amounts of biodiesel can lead to false positive indications of fuel content when the apparatus is not calibrated with that fuel.

6.4 Other interferences include the possibility of the calibration not being performed with the source fuel and lubricant material under test. This can significantly reduce the precision of the method.

6.5 Other interferences can arise from fuel dilution sample holders that are not completely dry or with a cleaning compound residue.

6.6 Samples that have very high water content (>1.0 % by mass) may also degrade the precision of the measurement.

7. Apparatus

7.1 The SAW fuel dilution apparatus (see Figs. 1 and 2) consists of the following components:

7.1.1 *SAW Sensor Module, Fuel Dilution Sample Stand, Sample Inlet, and Seal*—These components work in conjunction to extract a sealed vapor headspace from the sample being analyzed, and gauge fuel contamination in the in-service lubricant sample.

7.1.2 *Fuel Dilution Sample Stand Holder*—This is a disposable bottle into which the in-service lubricant is added and placed into the apparatus.

7.2 *Optional*—A *thermometer*, for example a thermocouple, which allows for the determination of the lubricant temperature. A thermometer is not required because users may elect to calibrate the instrument each time before a series of measurements, minimizing the temperature effects discussed in 6.1.

8. Reagents and Materials

8.1 *As-prepared Fuel Dilution Calibration Standards.* These are prepared from samples of the fuel and lubricant under use.

8.2 *Fuel Dilution Sample Holder,* such as a plastic bottle or laboratory vial.

8.2.1 If glassware is to be used as the fuel dilution sample holder, ensure that it is carefully cleaned and dried to avoid interferences.

9. Hazards

9.1 Since pure fuel is mixed with lubricant during calibration, care should be taken to avoid any contact with electronic equipment or spark sources to avoid fuel ignition.

9.2 Hazardous materials precautions, as appropriate, should be followed when handling both the fuel and lubricant under test.

9.3 Typical hazards considerations for electronic equipment should be followed in accordance with manufacturer's instructions.

10. Sampling, Test Specimens, and Test Units

10.1 A sample of the liquid should be obtained. The amount of liquid depends on manufacturer's instructions. If obtained directly from the equipment, it should be allowed to reach room temperature before further action. If such equipment is available, the sample may be obtained as described in Practice D4057.

11. Preparation of Apparatus

11.1 Place an appropriate amount of sample into the fuel dilution sample holder in accordance with manufacturer's instructions.

11.2 If the fuel dilution sample holder has a fuel dilution seal placed on by the operator, place the seal onto the holder and wait 1 min before proceeding to the next step.

11.3 Ensure that the SAW fuel dilution apparatus is free of residual vapor by inspecting the current displayed fuel content to see that it reads 0.0 % to 0.1 % by mass. If not, the performance of the method will be altered and biases can be expected.

11.4 Ensure that the temperature of the sample is within 2 °C of that of the calibration sample. If not, either wait for the sample to reach this temperature range or recalibrate.

11.5 Follow the manufacturer's instructions for the operation of the instrument.

12. Calibration and Standardization

12.1 The SAW fuel dilution apparatus should be calibrated with the operator's expected oil and fuel at the mid-range (5.0 % by mass) of dilution prior to operation of the apparatus on a given day or before a set of measurements is run. Such a sample may be prepared using a standard scale. An empty bottle is placed on the scale, zeroed, and the needed amount (for example, 50 g) of lubricant is added to the bottle. The corresponding amount (2.65 g) of the fuel that is being tested for is added to the bottle.

12.2 It is critical that a calibration standard include a representative sample of the fuel to be detected, in addition to representative lubricant itself. To ensure a representative calibration sample, the standard should be mixed and allowed to equilibrate for at least 1 h for diesel fuel and at least 4 h for more volatile gasoline fuel or jet fuel. Alternatively, a fresh open bottle of pure fuel may be allowed to age and then be mixed for immediate use. In this case, the sample should be allowed to outgas for a period of time until a volume decrease of 15 % is noted, at which point the sample is considered aged. The bottle should remain uncapped during the equilibration period to ensure a representative calibration sample. This is done to offgas the light end hydrocarbons that are present in fresh fuel samples. This is consistent with an actual engine oil sample, since it would have been exposed to heat during operation that drives off the light end gases.

12.3 The apparatus should be turned on and running for at least 15 min before the calibration sample is run.

12.4 Calibration measurements are performed in accordance with normal device operating instructions as outlined in Section 14.

12.4.1 *Optional*—Record the temperature of the sample before calibrating.

12.5 *Optional*—Validate the calibration by rerunning the 5.0 % calibration sample and another known sample in the range of <0.1 % to 10.0 % by mass. The values should agree to within ±0.5 %. If the values do not agree to within this limit, recalibrate.

12.6 Note that no equivalence (in accordance with, for example, Guide D7235 or Practice D6708) to standard laboratory test methods such as Test Method D7593 is required, since the apparatus is calibrated with the same sample lubricant and same fuel that is to be tested, in predetermined amounts.

13. Conditioning

13.1 As long as the meter reads 0.0 % to 0.1 % by mass fuel before measurement, no conditioning of the apparatus is required. If non-zero readings persist, continue to pump air through the apparatus to remove residual vapor.

14. Procedure

14.1 Place the sample, after it reaches room temperature, in the fuel dilution sample bottle.

14.2 Put the fuel dilution sample bottle onto the fuel dilution stand, and use the lever to engage the fuel dilution seal.

14.2.1 Initiate the measurement, which will begin to draw vapor into the SAW sensor through the fuel dilution sample inlet.

14.3 Once the measurement is complete, remove the fuel dilution sample bottle.

14.4 If an erroneous measurement is detected by the instrument, follow the manufacturer's instructions regarding troubleshooting. An erroneous measurement can arise when a generated waveform due to the introduction of the sample does not decay, for example, due to the fact that the liquid sample has been introduced into the vapor lines.

14.5 If removing the fuel dilution sample bottle does not cause the reading on the apparatus to read between 0.0 % to 0.1 % by mass after several minutes, inspect the fuel dilution sample stand, sample inlet, and seal for liquid residue. If accessible, wipe these areas clean. If not, replace the appropriate component (for example, the tubing in the fuel dilution sample inlet). If a high reading persists, contact the manufacturer.

15. Calculation or Interpretation of Results

15.1 The results are calculated automatically by the SAW fuel dilution apparatus control system and displayed on the LCD screen to the user. If an erroneous measurement occurs, the user is notified of this by means of the LCD screen. Interpretation of results depends on the nature of the liquid being analyzed.

15.2 Results may be used for immediate action due to the presence of fuel contamination or may be trended and analyzed over time in accordance with Guides D7669 or D7720 in the case where trace fuel contamination may be occurring.

16. Report

16.1 The fuel dilution in percent by mass is reported to one decimal place in the range of 0.1 % to 10.0 % by mass.

17. Precision and Bias

17.1 A full precision and bias statement gleaned from an interlaboratory study will be provided within the time parameters established by ASTM for such statements. A preliminary precision statement was established using seven operators, each with a different apparatus. Fifteen samples were analyzed in blind duplicate, comprising diesel, gasoline, and jet fuel contamination in diesel lubricant, mineral oil, and synthetic gas turbine lubricant, respectively. The samples ranged in concentration from 0 % to 12 % by mass fuel dilution. Further statistics gleaned from this test are discussed in Appendix X1.

17.2 *Preliminary Repeatability*—Preliminary examinations of repeatability have shown that the difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$r = 0.4722(X+0.0001)^{0.6126} \text{ \% by mass (fuel dilution)} \quad (1)$$

where:

X = the average of the two test results.

18. Keywords

18.1 fuel dilution; fuel sniffer; headspace; SAW; surface acoustic wave

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL PRECISION INFORMATION

X1.1 *Intermediate Precision*—An intermediate precision statement in accordance with Terminology E456 was established by means of the same test described in 17.2. In particular, the variation in results obtained by the seven operators, each with a different instrument, was studied in order to establish an intermediate precision statement. Each of the operators performed the blinded tests in the same facility at different times. The time between each operators' test varied between 4 h and 48 h. Fifteen samples were analyzed by each operator in blind duplicate, comprising diesel, gasoline, and jet fuel contamination in diesel lubricant, mineral oil, and synthetic gas turbine lubricant, respectively. These preliminary examinations have shown that the difference between two single and independent results obtained by different operators working on different apparatus in the same laboratory on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$0.6574(X+0.0001)^{0.6126} \text{ \% by mass (fuel dilution)} \quad (X1.1)$$

where:

X = the average of the two test results.

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