APPLICATION NOTE

Thermal Analysis

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Soot Content Determination of In-Service Lubricants of Diesel Engine as Per ASTM D5967 Annex A4

Introduction

Internal combustion in an engine produces soot as a result of incomplete fuel combustion. Normally in diesel engines, fuel and air does not get thoroughly mixed before ignition. This produces fuel dense pockets that produce soot when ignited. The majority of soot passes out through the exhaust and some gets past the piston rings and then gets mixed with the oil. The soot deposits deplete the quantity of dispersant additives which may cause an increase in viscosity which in turn increases engine wear. These soot particles adversely affect the engine performance and can lead to damage of the engine parts. Hence, timely monitoring of engine oil soot content is a necessity for the smooth working of the engine.

This application note demonstrates the quantification of soot content as well as the repeatability of the measurement using both the PerkinElmer Pyris™ 1 TGA and the PerkinElmer TGA 8000™ (shown in Figure 1) as per the ASTM D5967 method. ASTM D5967 is a test method which covers engine test procedures for evaluating diesel engine performance characteristics including viscosity and soot concentrations. Annex A4 is a recommendation on how to measure soot in engine oils. The Pyris 1 TGA has been adopted in many laboratories for this type of measurement. It's replacement, the TGA 8000 is shown to give equivalent results for these tests. Results are presented from both instruments.

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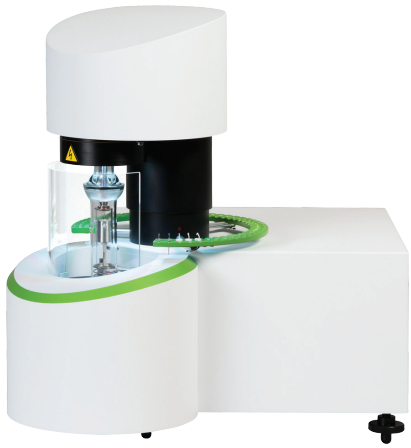

Figure 1. The PerkinElmer TGA 8000

Materials Used

1. Wilks enterprise Soot standards
2. Test sample from industrial manufacturer of lubricant

Result and Discussion

The following TGA analysis conditions were set for the analysis, following Annex A4 recommendations

Table 1. TGA Experimental Conditions.

INSTRUMENT CONDITIONS	
Temperature Program	1. Hold for 1 min at 50 °C 2. Heat from 50 °C to 550 °C at 100 °C/min 3. Hold for 1 min at 550 °C 4. Heat from 550 °C to 650 °C at 100 °C/min 5. Heat from 650 °C to 7500 C at 100 °C/min 6. Hold for 5 min at 750 °C
Pan Type	Platinum
Balance Purge	40 ml/min
Sample Purge	N_2, 30 ml/min for step 1 to 4 O_2, 30 ml/min for step 5 and 6
Sample Quantity	Around 10 mg

TGA thermogram:

The first weight loss that occurs as the sample is heated is the evaporative loss of the lubricating oil and any other volatile materials present. All that remains will be soot and other solid residues present in the sample. Switching the purge gas from nitrogen to oxygen will remove any soot present in the sample.

The calculation of soot content was performed for the weight loss that commences after switching over the purge gas from nitrogen to oxygen at 650 °C up to the temperature where constant residue was obtained around 750 °C.

The thermograms in Figure 2 show the quantification of soot in certified reference materials of 15W40 grade used oil.

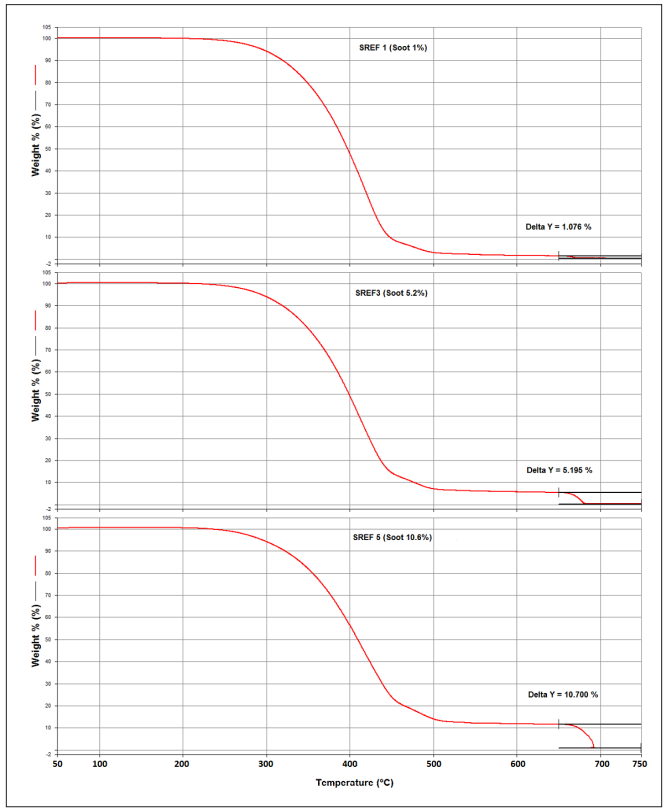

Figure 2. Thermograms obtained from the three certified reference materials.

TGA analysis report:

Table 2 shows the comparative TGA analysed value and reference standard value.

Table 2. Results obtained from the Pyris 1 and the TGA 8000 compared to the Certified Reference values.

SAMPLE	SOOT% BY PYRIS 1 TGA	SOOT% BY TGA 8000	SOOT% STANDARD VALUE
1% Soot Standard	1.076	1.055	1.0
5.2% Soot Standard	5.195	5.210	5.2
10.6% Soot Standard	10.700	10.680	10.6

The analysed certified reference materials show very low deviation (close to 0.1%) with respect to the standard values and complies with Annex A4.

Figure 3 shows the correlation between Pyris 1 TGA analyzed soot value and reference standard value.

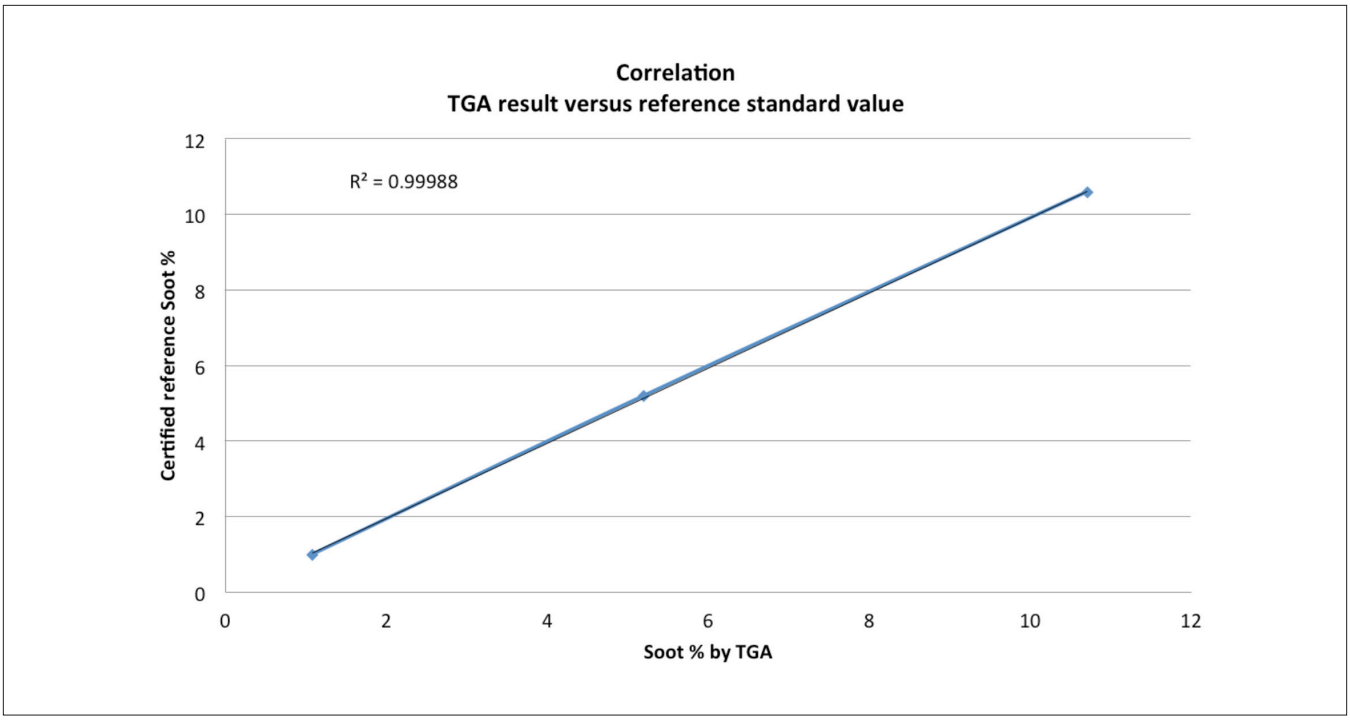

Figure 3. Comparison of TGA results versus the Certified Reference values for the standards.

Figure 3 shows that there is a linear correlation between results obtained by TGA and the certified reference values with regression coefficient of 0.9999. This shows the feasibility and accuracy of TGA for the estimation of soot as per Annex A4.

To study the repeatability of the soot content measurement by TGA, an unknown test sample was analysed in triplicate and the resulting thermograms shown in Figure 4.

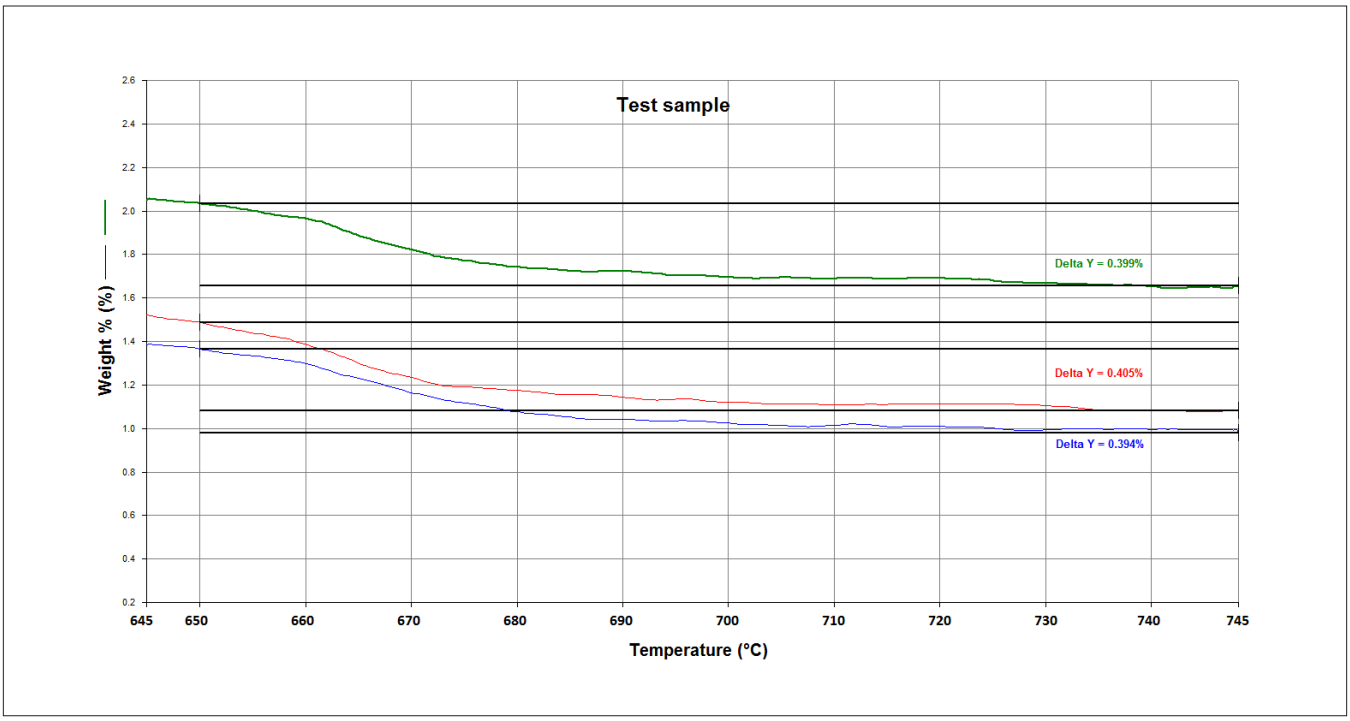

Figure 4. Repeatability of measurements of the test sample.

Table 3 shows the tabulated results of the repeatability test.

Table 3. Repeatability test results.

TEST SAMPLE	SOOT% BY TGA	DEVIATION (%)
Run 1	0.405	0.006
Run 2	0.399	0
Run 3	0.394	0.005
Average	0.399	

The repeatability test shows that the deviation is less than or equal to 0.006% for the quantification of soot as per Annex A4 using TGA.

Conclusion

There are many adverse effects of soot in terms of performance and durability of engines. Hence, it is extremely important to monitor soot on a timely basis. The certified reference materials analysed by TGA show a low deviation of around 0.1% and correlation of 0.9999 with respect to standard value. The repeatability test shows a deviation of less than or equal to 0.006%. Thermogravimetric analysis using the Pyris 1 TGA and it's successor, the TGA 8000, demonstrates easy, accurate, and reproducible quantification of soot in used lubricating oils as per ASTM D5967 Annex A4. Use of an autosampler on the TGA 8000 allows for higher sample throughput and better productivity in the laboratory.

Reference

ASTM D5967 – Standard test method for evaluation of diesel engine oils in T-8 diesel engine

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