



The TGA Noack Test for the Assessment of Engine Oil Volatility

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Background

Researchers in the automotive and petrochemical industries have studied the effects of oil volatility on engine emissions and oil consumption over the past decade. It is generally accepted that reducing oil volatility should have a positive impact on emissions and consumption. As emissions and consumption levels are both being reduced for environmental concerns, engine oil volatility has become an increasingly more important specification for motor oils.

The standard test for assessing oil volatility has been the Noack [1,2] and gas chromatographic techniques [3,4]. The Noack test conditions, in particular, seems to be recognized as being more representative of those experienced by oils in engines during operation.

The Noack volatility of an oil is defined as the weight loss of the oil when it is held under isothermal conditions at 250 C for a period of 1 hour under a constant flow of air. This test does expose the oil to relatively high temperatures and to air and simulates or approximates the conditions in the area around the top piston rings of engines. It is believed that the majority of the emissions are a result of exposure of the oil to the piston ring region.

While the Noack test does provide valuable information on oil volatilities, there are concerns about the test due to health or safety reasons. These stem around the fact that the Noack test utilizes a metal alloy, for heat transfer purposes, comprised of lead, bismuth, tin and cadmium, the vapors of which are toxic. The Noack test is also imprecise,

labor intensive and time consuming to conduct. All of these factors contribute to the need for the development of a safer, less time-consuming test to assess oil volatility.

The other common test is the use of gas chromatography (GC) to indirectly assess oil volatility. The GC method has the oil absorbed onto a packed column in an inert atmosphere (helium or nitrogen). The column is then heated to 600 C and the desorption is then monitored. The oil volatility is defined as the amount of oil desorbed from the column at a temperature of 371 C, relative to the total oil desorbed or relative to a known quantity of some internal standard. The advantage of the GC method is that it is safer and less hazardous than the traditional Noack test. The disadvantages are that the GC test is conducted in an inert atmosphere, as compared to air, and does not truly simulate the conditions to which the oil would be subjected to in real life conditions.

It was desired to have a test that would combine the real life conditions used in the traditional Noack test with the safety and precision of the GC test. Such a method was developed by E.F. de Paz and C.B. Sneyd and utilizes thermogravimetric analysis, TGA [5,6], and the test is known as the TGA Noack test. The test is currently in the process of being adopted as an ASTM test procedure for the characterization of the volatilities of oils.

TGA Noack Test Procedure

The TGA Noack test offers the precision and safety of the GC test while simultaneously providing real

life conditions (exposure to air at an elevated temperature) of the traditional Noack test. In addition, the TGA Noack method is fast and easy to perform.

The PerkinElmer Pyris 1 TGA or TGA 7 provides an excellent means of characterizing oil volatilities using the TGA Noack test. The PerkinElmer Pyris for Windows software offers a turnkey approach known as the Pyris *Player* software which will take the TGA Noack test from start to finish (including running, analysis, plotting and assessment of 'pass/fail' criteria) at the touch of a button. This makes the TGA Noack test more standardized and user-friendly, especially when multiple shifts and numerous operators are necessary. The Pyris 1 TGA features a state-of-the-art autosampler, which provides unattended sample loading and unloading. With the autosampler, oil samples can be analyzed overnight without the presence of an operator.

The recommended conditions for the TGA Noack test are:

- Sample mass of 36 to 40 mg placed in aluminum liner using injection pipette
- Air purge at a flow rate of 150 mL/min
- Heat sample from 50 to 249 C at 65 C/min
- Hold sample at 249 C for 15 minute isothermal period
- Measure mass loss (%) at certain time interval, *Noack Reference Time*, as specified by analyzing a Noack reference oil (RL-N)

The “Noack reference time” is determined by analyzing a sample of the reference oil, RL-N, under the conditions specified above. The time that it takes for the reference oil to reach a specified mass loss (14.2% for oil RL-N) then becomes the Noack reference time that is used as the standardized reference time for the assessment of mass losses during subsequent measurements.

The TGA instrument should be ‘burned out’ periodically (e.g., every 10 runs) by heating the instrument (no sample present) to 1000 C and holding for a 10 minute period under an air purge.

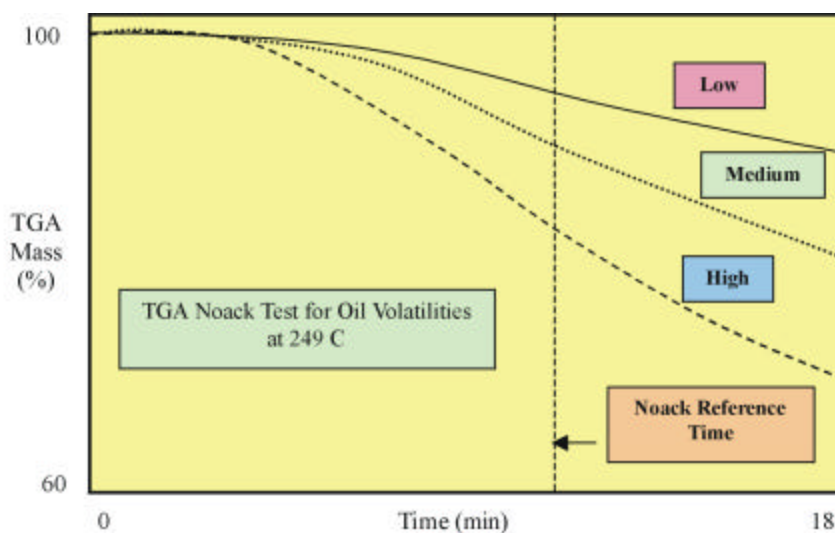
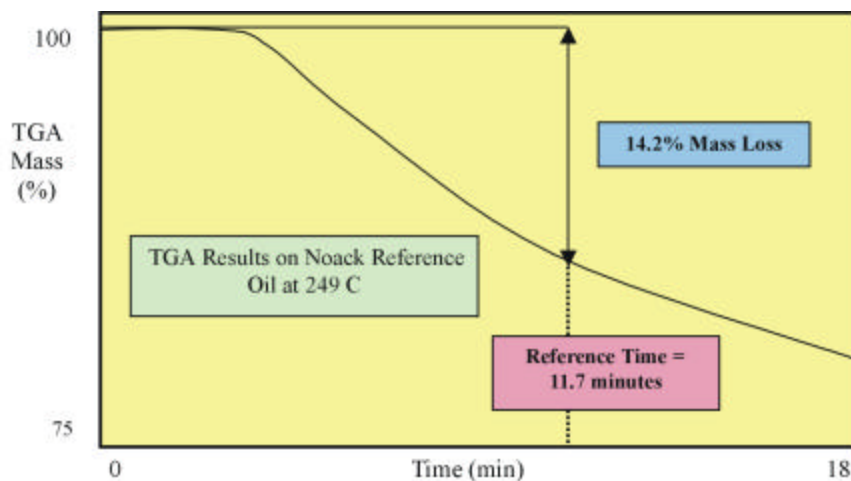
Displayed in the following figure is the assessment of the Noack reference time based on the TGA mass loss (defined as 14.2%) for the Noack reference oil.

Shown in the following figure are the TGA results obtained on a series of three motor oils with different volatilities.

The oil with the higher degree of volatility exhibits the greatest loss in weight after the Noack reference time interval at 249 C.

Summary

The TGA Noack test provides a fast, sensitive, safe and reproducible means of assessing the volatilities of engine oils. The TGA test correlates very well with the traditional Noack and gas chromatography (GC) test, while providing enhanced sample throughput and precision. The test is conducted by holding a bulk quantity of the oil at 249 C under isothermal conditions for 30 minutes with an air purge. The higher volatility oils will generate a larger mass loss after the Noack reference time at 249 C. The PerkinElmer Pyris 1 TGA or TGA 7 provides a sensitive, easy to use instrument for the TGA Noack test.



The low mass furnace associated with the Pyris 1 TGA and TGA 7 permits the instrument to cool back quickly to room temperature decreasing the sample analysis time. In addition, the Pyris *Player* software provides an easy-to-use, automated means of analyzing the oil samples.

References

- [1] D.S. Orrin, B.W. Coles, “Effects of Oil Composition on Oil Consumption”, SAE Paper 710141
- [2] F.D. Didot, E. Green, R.H. Johnson, “Volatility and Oil Consumption of SAE 5W-30 Engine Oil”, SAE Paper 872126

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- [3] ASTM D5800-96, "Evaporation Loss of Lubricating Oils by Noack Method"
- [4] ASTM D2887-93, "Boiling Range Distribution of Petroleum Fractions by Gas Chromatography"
- [5] E.F. de Paz, C.B. Sneyd, "The Thermogravimetric Noack Test: A Precise, Safe and Fast Method for Measuring Lubricant Volatility", Subjects in Engine Oil Rheology and Tribology, SP1209, International Fall Fuel and Lubricants Meeting, San Antonio 1996
- [6] *Hart's Lubricant World*, pages 20-21, December, 1996
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