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 deabsorption 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,[6,7] and the test is known as the TGA Noack test. The TGA Noack Test, in this application, has been developed to improve reproducibility and taken into account better test conditions considerations to closely meet ASTM D6375-09.

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 TGA8000, TGA4000 or STA6000 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 TGA8000 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, normalizing all other sample weights to the first.
- Nitrogen 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.

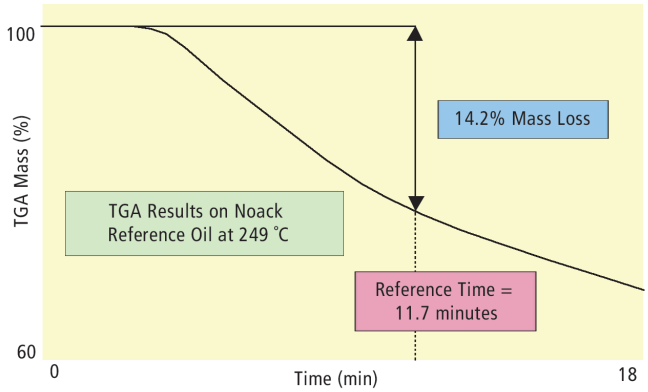
The TGA instrument should be 'burned out' periodically (e.g., every 10 runs) by heating the instrument (no sample present) to 500 °C and holding for a 10 minute period under an oxygen purge.

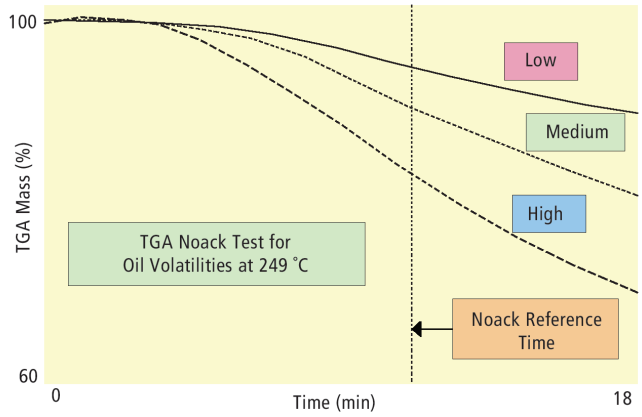
For improved reproducibility, the certified reference standard, RL-N, should be run after every fifth sample. An average of three to five runs of the reference material should be used to get a mean and standard deviation.

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 oxygen purge. The higher volatility oils will generate a larger mass loss after the Noack reference time at 249 °C. The PerkinElmer TGA8000, TGA4000 or STA6000 provides a sensitive, easy to use instrument for the TGA Noack test.

The low mass furnace associated with the TGA8000, TGA4000 and STA6000 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.

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