*Thermochimica Acta*, 175 (1991) 73–77
Elsevier Science Publishers B.V., Amsterdam



# VOLATILITY DETERMINATION OF OILS BY TG

B Gustavsson
Analytical Laboratory
Nynas Bitumen Technology AB
149 82 Nynaeshamn (Sweden)

## SUMMARY

A method, to determine the volatility of oils by thermal gravimetric analysis, is presented. Shown is that the loss of weight of mineral base oils, and blended engine oils, can be measured in a practical manner. The method correlates with the DIN 51581 standardized method. The effect of small changes in temperature of analysis, and purge-gas flow is described.

The method, which makes use of an oil with known weight loss, has a repeatability of 5%, and has been used during the past three years by our company.

## INTRODUCTION:

Mineral oils are still the most common lubricants for engines. The properties of lubrication should preferably perform equally well in case of a cold or a hot engine. If the liquid lubricant contains components with a relatively low boiling point, these fractions will evaporate, to a certain degree, at the higher operating temperature which occur during the running of the engine. This will result in an increased oil consumption, increased viscosity and decreased low temperature properties. Because of this it is important to choose base oils with low evaporation loss at increased temperature, and still low viscosity at low temperatures, to guarantee sufficient lubrication when starting the engine.

There are several different methods to determine the volatility of an oil, such as distillation, vapour pressure and weight loss as a function of time and temperature (Thermal Gravimetry). A method which describes a correlation between distillation and TG is found in "Fuel" 1984 (1). A common method to determine the volatility of a motor-oil or the base-oil to a motor-oil is the DIN 51581 standard, which also is referred to as the NOACK-method (2). The NOACK-method requires special apparatus and takes about one hour for one single analysis, resulting in high laboratory cost and investment.

A different method, making use of Thermal Gravimetric Analysis (TG) was reported by a group from British Leyland (3), in which a correlation between oil consumption and lubricant volatility was found. Our aim was to develop a new method, based upon TG, which could determine the volatility of mineral oils in a more practical and time saving manner. The objectives were to achieve accurate correlation with the existing NOACK-method, with similar or better repeatability and increased speed of analysis.

0040-6031/91/$03.50 

## EXPERIMENTAL

Aluminium oxide crucibles of Mettler standard size (70 µl) were used. They were filled with 50 ±5 µl of oil. The samples were introduced in the oven at a low temperature (50 °C). The temperature was then raised to the analyzing temperature and held there. The oven was purged with 0.20 l/min of high purity nitrogen to avoid weight change in the sample due to chemical reaction with atmospheric oxygen. During method development a range of temperature gradients and analyzing temperatures were evaluated, in search for optimum conditions.

It was also investigated how much a small change in analyzing temperature will influence the result and if changes in purge-gas flow will have an effect on the weight loss.

## RESULTS AND DISCUSSION

The samples that were used were five base oils with NOACK weight-loss ranged between 6,5 and 20 weight-%. Three of the oils were blended engine-oils, included one NYNAS Tarola 15W/40. One of the oils had a well determined NOACK weight loss of 14.1%, this oil is in the results labelled INTERNAL-standard. The INTERNAL-standard oil is also useful because of engine-oil specification.

### Correlation TG-NOACK

Method development showed that a correlation could be found between NOACK and TG weight loss for the TG-methods that were tested. The best result however was achieved by using a 40 °C/min temperature gradient from the insert temperature up to a analyzing temperature of 250 °C. The results are shown in Figure 1 and in Table 1.

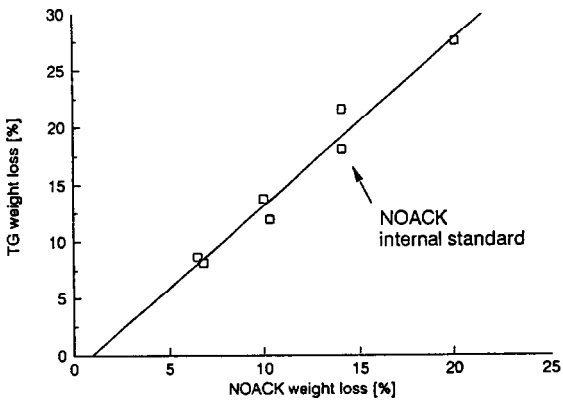


Figure 1 Diagram showing the correlation between "NOACK" and "TG" weight loss.
The linear correlation coefficient is 0.965.

Importance of small changes in analysis temperature.

The manufacturer of the TG equipment (Mettler) gives a guarantee that the oven has a diversion of maximum ±2 °C. To check how much a small diversion in analyzing temperature would influence the result, four oils and the standard were analyzed with a gradient end-point of 245 and 255 °C as well as 250 °C. The results are shown in Table 2 and in Figure 2.

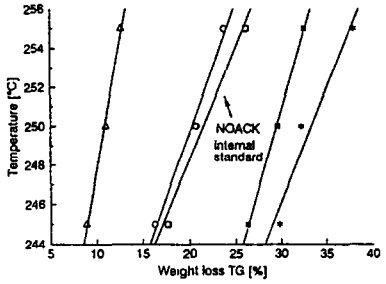


Figure 2 Diagram showing loss of weight at slightly different temperatures of analysis. A change in end point temperature of 2 °C caused a change in weight loss of about 5%.

Importance of changes in purge-gas flow

The manufacturer of the instrument recommends a purge-gas flow of 0.20 l/min. To check if a change from this flow would effect the weight-loss, three oils were analyzed at three different purge-gas flows. The gas-flows tested were 0,09, 0,20 and 0,34 l/min.

The results showed that a decrease in gas-flow from 0,20 l/min will only influence the weight loss marginally, 10% decrease in gas-flow will increase the weight loss about 3%. An increase in gas-flow may dramaticly decrease the weight loss. Before analysis the purge-gas flow must be adjusted to 0.20 ±0.01 l/min. The results are shown in Figure 3 and Table 3.

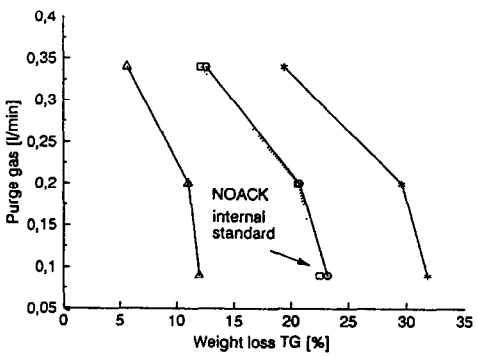


**Figure 3 Correlation between weight loss and change of the purgegas flow.**

## CONCLUSIONS

A good correlation between the TG and NOACK methods is found in the range between 6.5 and 20 wt% NOACK.

If the internal standard and samples are analyzed in duplicate the repeatability is at least that of DIN 51581 (NOACK) method, 5 relative %.

Even small changes in analyzing temperature have considerable impact on the weight loss.

A small decrease in purge-gas flow does not change the analyzeresult significantly.

Purgegas flows over 0,20 l/min should be avoided.

The TG method is up to three times faster than the NOACK-method

The TG-method consists of the following data:

| | | |
|---|---|---|
| Start temperature: | 50 | C |
| Temperature scan: | 40 | C/min |
| Analysis temperature: | 250 | C |
| Analysis time: | 15 | min |
| Purge-gas: | 0.20 | l/min |
| Sample: | 50 ±5 | µl |
| Crucible: | 70 | µl Al2O3 (Mettler N:r 24123) |

TABLE 1

Correlation between TG and NOACK weight loss

| Product | TG weight-loss% | NOACK weight-loss% |
|---|---|---|
| Internal standard | 18.1 | 14.1 |
| Tarola 15W/40 NYNAS | 8.2 | 6.8 |
| External product I | 12.0 | 10.3 |
| External product II | 27.6 | 20.1 |
| Base oil I | 21.6 | 14.1 |
| Base oil II | 8.7 | 6.5 |
| Base oil III | 13.8 | 10.0 |

TABLE 2

Importance of small changes in analyzing temperature

| Product | Analyzing temperature 245 C | 250 C | 255 C |
|---|---|---|---|
| Internal standard | 17.7 wt% | 20.6 wt% | 26.1 wt% |
| External product II | 26.3 " | 29.6 " | 32.5 " |
| Base oil II | 8.9 " | 11.0 " | 12.7 " |
| Base oil IV | 16.3 " | 20.7 " | 23.7 " |
| Base oil V | 29.8 " | 32.2 " | 37.9 " |

TABLE 3

Importance of changes in purge-gas flow

| Product | Purge-gas flow 0.09 l/min | 0.20 l/min | 0.34 l/min |
|---|---|---|---|
| Internal standard | 22.5 wt% | 20.6 wt% | 12.1 wt% |
| External product II | 31.8 " | 29.6 " | 19.4 " |
| Base oil II | 12.0 " | 11.0 " | 5.6 " |
| Base oil IV | 23.2 " | 20.7 " | 12.6 " |

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