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APPLICATION NOTE 42485

Elemental Analysis: Nitrogen determination of lubricants by combustion method using a single reactor

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Goal

To assess the performance of the elemental analyzer for the nitrogen determination of lubricants using a single reactor.

Introduction

In a typical production process of mineral oils, the nitrogen content is periodically monitored and tested for quality control. The concentration of nitrogen is an indicator for the presence of nitrogen-containing additives. Information about nitrogen concentration can be used to predict the performance of the lubricant. The method for the analysis of nitrogen in lubricants is described in ASTM D5291.

The method described in ASTM D5291 is based on combustion analysis.

A simple and automated technique allowing fast analysis with excellent reproducibility, without the handling of toxic chemicals is required by laboratories, looking at reduced analysis time and confident results. The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1), based on the dynamic combustion method (modified Dumas method), provides rapid and automated nitrogen determination without use of hazardous chemicals and offers advantages over traditional methods.

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Figure 1. Thermo Scientific Flash*Smart* EA

Methods

The standard N Lubricant configuration is based on a double reactors system: first reactor for combustion and catalytic oxidation of the combustion gases, the second is used to reduce nitrous oxides as N_2. The superior performance of the Flash*Smart* EA allows the reduction of the amount of oxidation catalyst needed for nitrogen analysis using a single combustion/reduction reactor (25 mm diameter). The reactor filled with less amount of oxidation catalyst and copper ensures the complete conversion of gases produced by the combustion. It also offers advantages for higher number of analyses.

The Flash*Smart* EA operates according to the dynamic flash combustion of the sample. Samples are weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific™ MAS Plus Autosampler together with oxygen. After combustion, the produced gases are carried by a helium flow to a layer of copper, then swept through CO_2 and H_2O traps, a GC column and finally detected by a Thermal Conductivity Detector (TCD) (Figure 2). A complete report is automatically generated by the Thermo Scientific™ Eager*Smart*™ Data Handling Software and displayed at the end of the analysis.

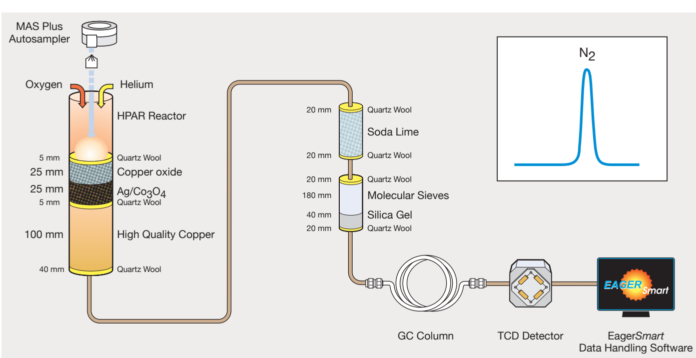

Figure 2. N Lubricant single reactor configuration

Results

The performance of the Flash*Smart* EA N Lubricant using a single reactor was evaluated through the analysis of the Thermo Scientific Lubricant Reference Material and three lubricant samples coming from an ASTM International Interlaboratory Program. The data was compared with the Reference Material certifícate and with the nitrogen accepted range indicated in the ASTM reports.

The Thermo Scientific Lubricant Reference Material was analyzed 10 times The calibration was performed with 4–5 mg atropine or tocopherol nicotinate, using K factor as calibration method. The sample weight was 8–10 mg. Atropine is one of the standards suggested in the ASTM 5291 Official Method and it is the standard with the lowest nitrogen percentage (4.84 N%), however this standard is classified as toxic material. Tocopherol nicotinate (2.61 N%) can be considered as an alternative to atropine. Table 1 shows the experimental data obtained of the Thermo Scientific Lubricant Reference Material using different standard for the calibration. Data are comparable and the repeatability is acceptable. The certificate Thermo Scientific Lubricant Reference Material data are: 1.12 N% (range ±0.10).

Table 1. Nitrogen data of Thermo Scientific Lubricant Reference Material

Standard used for calibration	Atropine	Tocopherol nicotinate
N%	1.068	1.073
	1.068	1.084
	1.059	1.071
	1.071	1.065
	1.073	1.071
	1.063	1.075
	1.073	1.082
	1.071	1.075
	1.065	1.070
	1.071	1.078
Average N%	1.068	1.074
Std.Dev.	0.0046	0.0057
RSD%	0.432	0.534

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Table 2 shows the ASTM lubricant nitrogen data obtained and compared with the accepted range of the ASTM International Interlaboratory Program. Samples were analyzed 10 times. The calibration was performed with 4–5 mg atropine as standard using K factor as calibration method. The sample weight was 8–10 mg. Data obtained are inside the range indicated in the ASTM reports.

Table 2. Nitrogen data of ASTM lubricants

ASTM lubricant sample	1	2	3
N%	0.922	0.725	0.583
	0.925	0.722	0.591
	0.926	0.722	0.583
	0.927	0.723	0.590
	0.934	0.728	0.582
	0.925	0.716	0.589
	0.940	0.715	0.590
	0.923	0.727	0.597
	0.928	0.729	0.598
	0.935	0.726	0.594
Average N%	0.928	0.723	0.590
Std.Dev.	0.006	0.005	0.006
RSD%	0.633	0.658	0.966
Accepted ASTM range N%	0.854 – 0.974	0.650 – 0.747	0.500 – 0.606

Another two oil samples were analyzed five times to show the repeatability. The calibration was performed with 4–5 mg atropine as standard using K factor as calibration method, the sample weight was 8–10 mg.

Oil sample	A	B
N%	1.007	0.779
	1.017	0.787
	1.016	0.781
	1.010	0.779
	1.009	0.781
Average N%	1.012	0.781
Std.Dev.	0.004	0.003
RSD%	0.439	0.421

Conclusions

The Flash*Smart* EA is a valuable solution for the analysis of nitrogen in lubricants in compliance to the ASTM D5291 Method.

Using one large reactor for nitrogen determination instead of the classical two reactors configuration, extends the analytical capabilities of the Flash*Smart* EA. No matrix effect was observed when changing the configuration, indicating the complete combustion of the sample.

The tocopherol nicotinate can be considered an alternative to other standards: it contains lower nitrogen and higher carbon and hydrogen content, and the element concentration is more similar to the lubricants composition. Besides, tocopherol nicotinate is not a hazardous standard and it is widely used, also in commercial products.

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