

MultiTek[®] Total Sulfur & Total Nitrogen Analysis of Heating Oil by UV-Fluorescence & Chemiluminescence

- Rapid and Accurate Determination of Chemically Bound Sulfur & Nitrogen
- Fully Automated Combustion System
- Repeatable and Reliable Sample Analysis

Keywords:

MultiTek, Total Sulfur, Total Nitrogen, UVF, Chemiluminescence, Heating Oil, Accuracy, Repeatability, ISO 20846, DIN 51444

EXPERIMENTAL CONDITIONS

Instrumentation:

Antek MultiTek VNS,
with Liquid Autosampler,
Antek Model 758.



Introduction:

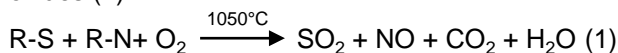
Heating oil is widely used in private houses and public facilities for heating. In order to limit the pollution of the environment by NO_x and SO₂, it is mandatory to control the total content of nitrogen and sulfur compounds in the heating oil product.

The application note shows, how the MultiTek analyzer system fulfills the requirements for analysis of total nitrogen & sulfur in heating oil, based on DIN 51444 (total nitrogen) & ISO 20846 (total sulfur).

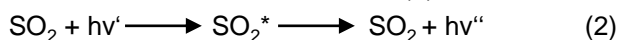
Two heating oil samples of an interlaboratory study (ILS)¹ were analyzed and compared.

Principle of Operation:

The hydrocarbon sample is introduced into a pyrotube at 1050°C. Any sulfur or nitrogen compounds present are combusted in an oxygen-rich atmosphere to SO₂, NO, CO₂, H₂O and other oxides (1):



Upon entering the detector cell, the SO₂ molecules are illuminated by UV-light, followed by the characteristic sulfur fluorescence (2):



The NO molecules are oxidized by O₃, which results in a specific chemiluminescence (3):



The intensity of the sulfur fluorescence and/or nitrogen chemiluminescence is used for calibration and quantification.

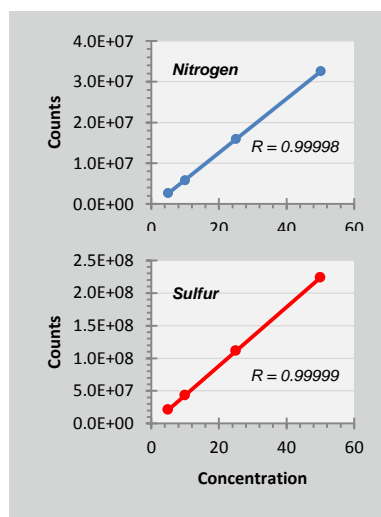
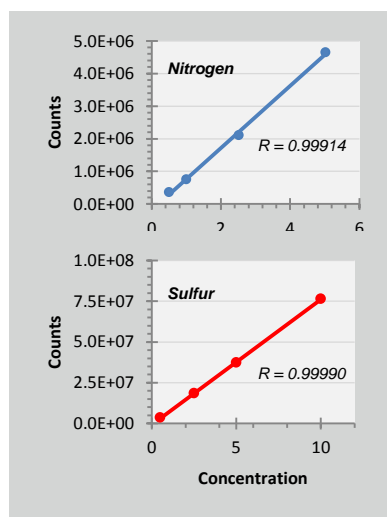
Instrument Parameters:

Syringe Size (µl)	25
Sample Fill Volume (µl)	15
GFC1 – Ar/He (ml/min)	130
GFC2 – Pyro O ₂ (ml/min)	400
GFC3 – Ozone O ₂ (ml/min)	25
GFC4 – Carrier O ₂ (ml/min)	30
Furnace (°C)	1050
Cycle Time (mm:ss)	5:00
S-PMT High Voltage (V)	600
N-PMT High Voltage (V)	600

¹ Annual Round Robin by Mineral Oil and Fuels Standardization Committee within DIN (FAM).
Heating oil samples originate from ILS 2012.

Calibration:

The MultiTek analyzer was simultaneously calibrated for total nitrogen and total sulfur, using mixed standards of Pyridine / Dibenzothiophene in Xylene. To cover the sample concentrations, two different working ranges were calibrated for each analyte, according to ISO 20846 and DIN 51444.



Results:

Table 1 shows the analysis results for each single injection and the calculated mean concentrations, which are comparable to the reported ILS mean results.

#Inject.	Sample HEL 777		Sample HEL 778	
	c(N) mg/kg	c(S) mg/kg	c(N) mg/kg	c(S) mg/kg
1	59.8	50.2	4.42	8.92
2	59.7	49.9	4.41	8.65
3	60.6	49.7	4.37	8.70
4	60.1	50.3	4.45	9.02
5	60.4	50.0	4.46	8.69
6	60.3	49.9	4.44	8.67
Mean	60.2	50.0	4.43	8.78
Mean SD	0.35	0.22	0.03	0.16
ILS Mean ²	60.5	49.6	4.47	8.59
ILS Mean SD	4.72	3.24	0.94	0.39
Z –Score ³	0.179	0.168	0.004	0.256

Table 1: MultiTek heating oil analysis and comparison to ILS results

Conclusion:

The analysis of heating oil demonstrates that the MultiTek fulfills the requirements of an accurate, precise and reliable analyzer system. The MultiTek offers a rapid and fully automated solution to monitor product specifications of final heating oil products.

The MultiTek is the only instrument on the market that combines sulfur, nitrogen, and halides analysis all in one. Compact, powerful, automated, and able to analyze gas, liquid, or solid samples, it's the perfect solution to today's increasing demand worldwide for fast, accurate detection and analysis of contaminants, and corrosive elements. Because MultiTek delivers precise results with high sensitivity and unmatched versatility, it's a valuable process optimization tool that will deliver faster ROI and a better bottom line.