

## MultiTek® Nitrogen Analysis of Lubricating Oils by Horizontal Boat-Inlet Chemiluminescence

- **Rapid and Accurate Determination of Chemically Bound Nitrogen**
- **Fully Automated Combustion System**
- **Sample Versatility**
- **MultiTek® Performance Verification**

**Keywords:**

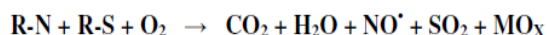
**MultiTek®, Total Nitrogen, Chemiluminescence, and ASTM D5762**

### INTRODUCTION

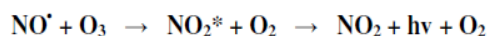
ASTM D5762 is an established method for the determination of nitrogen in lubricating oils by boat-inlet chemiluminescence. The application of this procedure is useful in determining the chemically bound nitrogen present in lubricating oils, this is significant because nitrogen components can contaminate refinery catalysts. The determination of nitrogen concentration in lube oils is a measure of the total nitrogen in various additives.

### Reaction:

The principal of operation for nitrogen analysis begins with the complete, high temperature oxidation of the entire sample matrix as illustrated in the first equation. The sample is combusted with oxygen at a temperature of 1050°C. The oxidation products include CO<sub>2</sub>, H<sub>2</sub>O, NO, SO<sub>2</sub>, and various other oxides (designated MO<sub>x</sub> below). The combustion gases are routed through a membrane drying system to remove all water and then to the nitrogen detector module for quantification.



According to the second equation, the NO is contacted with O<sub>3</sub> (ozone), produced by an onboard generator, to form NO<sub>2</sub> (metastable nitrogen dioxide).



### EXPERIMENTAL CONDITIONS

#### Instrumentation:

Antek MultiTek® Horizontal, Antek Model 740 boat inlet system, Antek Model 735 syringe drive, and Antek Model 748 Autosampler.

#### Instrument Parameters:

Syringe Size (µl)	25
Sample Fill Volume (µl)	5
GFC1 – Ar/He (ml/min)	130
GFC2 – Pyro O <sub>2</sub> (ml/min)	450
GFC3 – Ozone O <sub>2</sub> (ml/min)	35
GFC4 – Carrier O <sub>2</sub> (ml/min)	25
Furnace (°C)	1050
Cycle Time (mm:ss)	4:30
N-Detector Cooler (°C)	5
N-PMT High Voltage (V)	675

#### Calibration:

A calibration was performed from 0-100 µg/ml, using the recommended calibration standards listed in ASTM D5762.

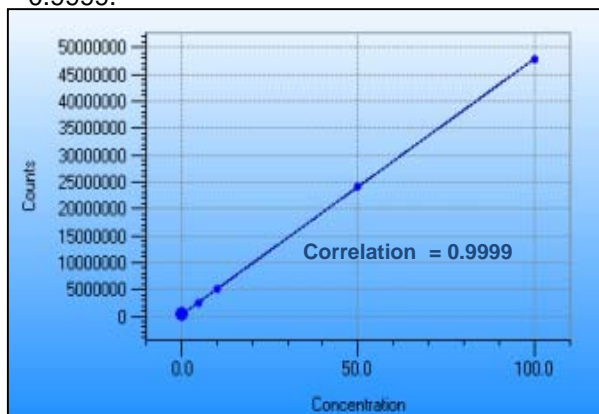
# APPLICATION NOTE



The samples were then prepared by dilution, according to the expected concentration/number of area counts that would fall within the method calibration range of 0-100 ppm ( $\mu\text{g/ml}$ ).

Calibration Standard Concentration ( $\mu\text{g/ml}$ ):	Average Area Counts (n=7):
0	502839
1.0	649422
5.0	2519953
10.0	5112004
50.0	24061623
100.0	47863193

The MultiTek HNS was calibrated in the range of 0-100 mg/kg using acridine, in a toluene matrix. The resulting correlation coefficient was 0.9999.



## Sample Preparation:

The samples tested were diluted in a solvent (xylenes) using the sample weight(s) shown below.

Sample ID:	S <sub>x</sub> Weight (g):	Solvent (ml):
LU1101	0.6213	20
Lube Oil AA	0.7308	20
Lube Oil BB	0.7435	20
Lube Oil J	0.7599	20

*The Antek 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.*

Sample ID:	Nitrogen Conc. ( $\mu\text{g/ml}$ ):	%RSD (n = 7):	Area Counts (n = 7):
ASTM LU1101	902	6.27	13623788
Lube Oil BB	822	4.17	14837023
Lube Oil J	830	2.00	15302946
ASTM LU1101	886	1.24	13393489
Lube Oil BB	834	1.34	15048787
Lube Oil J	837	1.15	15430231

## Correlation of Test Data:

The sample ASTM LU1101 was evaluated, as a check standard, for the sample data compiled. The statistical results for the program has been captured below. The data was used to calculate the acceptability of the data set by Z-score.

Sample ID:	Deviation by Z-score
LU1101	-1.18
LU1101	-1.34

## CONCLUSION

The analysis of lubricating oils demonstrates that exemplary data can be generated by the implementation of ASTM D5762 method parameters. This method also demonstrates versatility in quantifying a wide range of nitrogen containing lubricating oils.