APPLICATION NOTE

MultiTek® Complete Elemental Analysis of Mineral Oil by CIC / UVF / CLND Detection

- **Fully Automated Combustion System**
- **Rapid and Accurate Determination of Halides , Chemically Bound Nitrogen and Sulfur**
- **Sample Versatility**

Keywords:
MultiTek HNS-IC, pyrohydrolysis, halides, chloride, sulfur, nitrogen, mineral oil, combustion ion chromatography (CIC), Ultraviolet Fluorescence (UVF), Chemiluminescence (CLND)

INTRODUCTION

Mineral Oil is an alkane that is a byproduct of the fractional distillation of crude oil. It requires further refining to remove nitrogen, sulfur and both organic and inorganic salts before passing on to the consumer. Some of the final product uses include laboratories with oil emersion microscope slide preparation, food preparation as protectant for wooden kitchen utensils and in industrial tools such as hydraulic fluid. Mineral oil also plays rolls in the cosmetology and veterinary fields.

The determination of chlorine, sulfur, and nitrogen in mineral oil is a necessity to prevent corrosion in industrial equipment and to meet regulatory requirements for local, state and federal agencies. The presence of these contaminants push the need to monitor concentrations. The MultiTek HNS-IC is the only instrument on the market that has the ability to determine total halogens, total sulfur and total nitrogen compounds in samples all in one instrument.

Halides determination starts with pyrohydrolysis of the samples. In the pyrotube with the help of steam, halogen containing compounds are converted into an acid gas state. Sulfur containing compounds are oxidized in a unequalled occurrence ultimately converting to sulfite (SO_3) and sulfate (SO_4). Gases are condensed, absorbed, and then transferred by the MultiTek to an injection system on the Ion Chromatograph (IC) for conductivity analysis.
The principal of operation for nitrogen and sulfur analysis begins with high temperature oxidation and combustion of sample. The oxidation products include CO_2, H_2O, NO, SO_2, and various other oxides.

The chemiluminescence emission is specific for nitrogen oxide and is proportional to the amount of nitrogen in the original sample. Therefore, only chemically bonded nitrogen is detected. The SO_2 is exposed to ultraviolet radiation of a specific wavelength. This radiation is released in the form of sulfur fluorescence. The fluorescence is detected by a photomultiplier tube and is proportional to the amount of sulfur in the original sample.

REACTIONS

- **Halogen Reaction by CIC**

$$R - X_{(F^-\ Cl^-\ Br^-)} \xrightarrow[\Delta T + O_2 + H_2O]{} H\ - X_{(F^-\ Cl^-\ Br^-)}\ (g)$$

- **Sulfur Reaction by CIC**

$$R - S \xrightarrow[\Delta T + O_2 + H_2O]{} SO_2(g) \xrightarrow[scrubber]{} SO_3^{2-}(aq) + SO_4^{2-}(aq)$$

- **Sulfur Reaction by UVF**

$$SO_2 + hv' \longrightarrow SO_2^* \longrightarrow SO_2 + hv''$$

- **Nitrogen Reaction by CI**

$$NO + O_3 \longrightarrow NO_2^* + O_2 \longrightarrow NO_2 + hv' + O_2$$

EXPERIMENTAL CONDITIONS

- **Instrumentation**

Antek MultiTek HNS-IC, Antek Model 740 boat inlet system, Antek Model 735 syringe drive, autosampler and suppressed IC system.

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- **Instrument Parameters**

Injection Volume (µl)	20
IC Sample Loop (µl)	250
GFC1- Ar/He (ml/min)	130
GFC2- Pyro O_2 (ml/min)	450
GFC3- Ozone O_2 (ml/min)	35
GFC4- Carrier O_2 (ml/min)	30
Furnace (ºC)	1050

- **Standards**

Calibration was performed through entire sample flow path using certified mineral oil matrix standard materials (0-25ppm). The compound used for halide addition is chlorobenzene. Dibutyl sulfide and pyridine are used for sulfur addition and nitrogen addition.

- **Linear Calibration 0-25ppm w/w**

Detection	Cl	S	N
	Correlation Coefficient		
CIC	0.999	0.999	
UVF		0.999	
CLND			0.998

RESULTS

- **Repeatability of 10ppm Mineral Oil Sample (n=6)**

Injection	Cl	S (CIC/UVF)		N
1	9.5	9.8	10.2	9.5
2	9.7	10.1	10.0	9.1
3	9.8	9.6	9.9	9.2
4	9.1	9.5	9.5	9.3
5	9.0	9.1	10.2	9.4
6	9.0	9.5	10.0	9.3
% RSD	4.0	3.8	2.6	1.4

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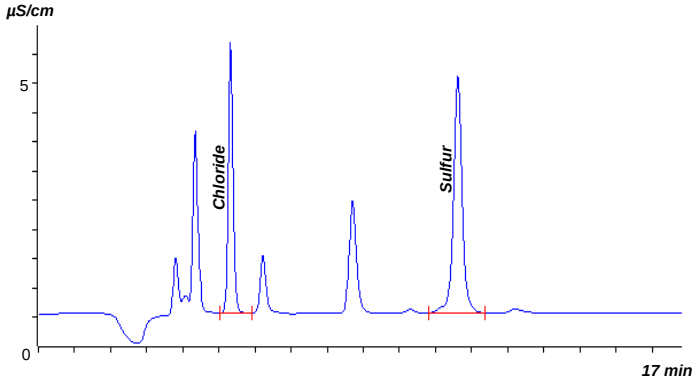
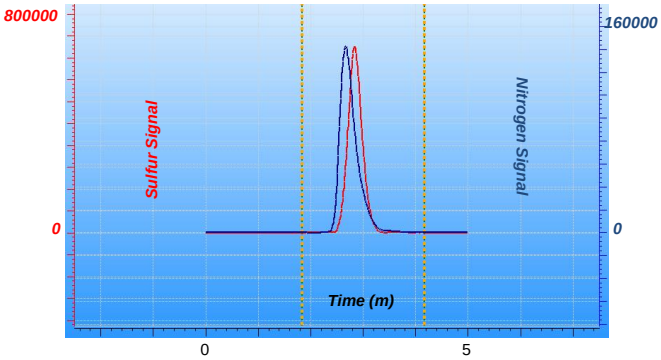
CONCLUSION

The MultiTek HNS-IC is the only instrument on the market that has the ability to determine total halogens, total sulfur and total nitrogen in samples all in one instrument. The results demonstrate that the MultiTek Analyzer coupled with Ion Chromatography provides a sensitive, automated and reliable elemental analysis of mineral oils.



CIC; 10ppm Mineral Oil Chromatography



UVF & CLND ; 10ppm Mineral Oil Chromatography

Anteks MultiTek® is the only instrument on the market that combines testing sulfur, nitrogen, and halides all in one. Compact, powerful, automated, and multi-configurable, it's the perfect solution to today's increasing demand worldwide for fast, accurate detection and the analysis of unwanted chemicals, pollutants, 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.

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