

## MultiTek® Complete Elemental Analysis of Liquefied Petroleum Gas (LPG) 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, sulfur, nitrogen, n-butane, combustion ion chromatography (CIC), Ultraviolet Fluorescence (UVF), Chemiluminescence (CLND)**

## INTRODUCTION

The determination of halides, sulfur, and nitrogen in LPG is vital for numerous reasons. Hydrofluoric acid alkylation is a common technique for the production of high octane gasoline and LPG fractions such as n-butane and propane. Even though acid settlers and adsorbent beds are used to remove fluoride from the process, traces still manage to make it into the final product. Nitrogen monitoring in the form of nitrogen oxides is important for fuel reactivity, hazardous smog formation/acid rain, and water deterioration. Sulfur concentration monitoring is important for many of the same reasons. Sulfur is poisonous to catalysts in subsequent processing. The presence of sulfur can also result in the corrosion of metal surfaces which have adverse effects on pipeline components and pushes the need to monitor concentrations of these elements.

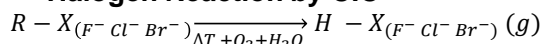
The MultiTek HNS-IC is the only instrument on the market that has the ability to determine total halogens, total sulfur and total nitrogen compounds in samples all in one instrument. Specifically n-butane was chosen, but heavier samples such as pentane and hexane are also applicable.

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<sub>3</sub>) and sulfate (SO<sub>4</sub>). 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.

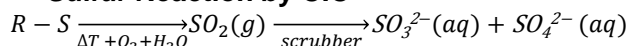
The oxidation products include CO<sub>2</sub>, H<sub>2</sub>O, NO, SO<sub>2</sub>, and various other oxides. The chemiluminescence emission is specific for nitrogen oxide and is proportional to the amount of nitrogen in the original sample. Therefore, only chemically bonded nitrogen is detected. The SO<sub>2</sub> is exposed to ultraviolet radiation of a specific wavelength. This radiation is released in the form of sulfur fluorescence. This fluorescence is detected by a photomultiplier tube and is proportional to the amount of sulfur in the original sample.

## REACTIONS

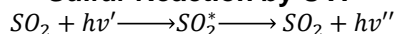
- Halogen Reaction by CIC



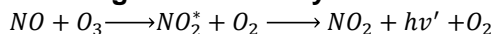
- Sulfur Reaction by CIC



- Sulfur Reaction by UVF



- Nitrogen Reaction by Cl



## EXPERIMENTAL CONDITIONS

- Instrumentation

Antek MultiTek HNS-IC, Antek Model 740 boat inlet system, Antek Model 735 syringe drive, Antek Model 734 Gas/LPG Sampler and suppressed IC system.



# APPLICATION NOTE



## Instrument Parameters

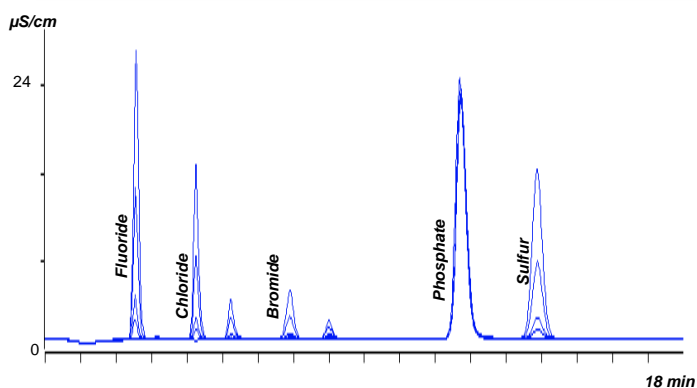
734 Sample Loop (µl)	15
IC Sample Loop (µl)	250
GFC1- Ar/He (ml/min)	130
GFC2- Pyro O <sub>2</sub> (ml/min)	450
GFC3- Ozone O <sub>2</sub> (ml/min)	35
GFC4- Carrier O <sub>2</sub> (ml/min)	30
734 Carrier- Ar or He (ml/min)	20
Furnace (°C)	1050

## Standards

Calibration was performed through entire sample flow path using certified n-butane matrix standard materials (0-300ppm). The compounds used for halide addition are fluorobenzene, chlorobenzene, bromobenzene. Dimethyl sulfide and acetonitrile used for sulfur and nitrogen addition.

## Linear Calibration 0-300ppm w/w

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



CIC; n-Butane Calibration Chromatography

## RESULTS

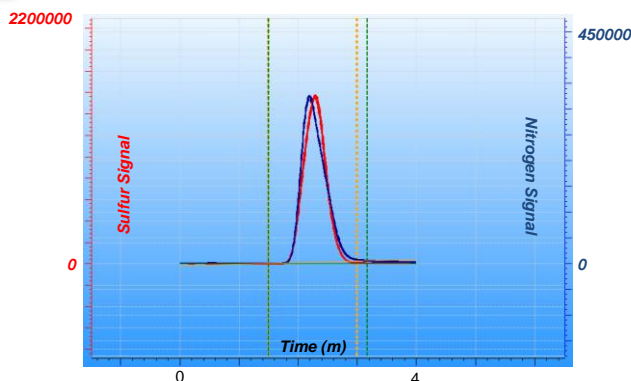
### Repeatability of Butane Sample (n=6)

	F	Cl	Br	S (CIC/UVF)		N
AVG ppm (w/w)	22	24	25	24	25	16
% RSD	1.0	2.3	1.8	4.4	0.8	0.7

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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 liquefied petroleum gas. This analysis will allow the refinery and other processes to monitor concentration levels to improve plant safety and efficiency.



UVF & CLND ; n-Butane Replicate 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.

