APPLICATION NOTE

MultiTek® Halide and Sulfur Analysis of Aromatic Hydrocarbons by CIC Detection

- **Fully Automated Combustion System**
- **Rapid and Accurate Determination of Halides and Sulfur**
- **ASTM 7359**
- **Sample Versatility**

Keywords:
MultiTek HS-IC, Trace Halides Kit, ASTM D7359, Vacuum Gas Oil (VGO), pyrohydrolysis, trace halides, fluoride, chloride, sulfur, aromatics, combustion ion chromatography (CIC)

INTRODUCTION

The determination of halides and sulfur in aromatic hydrocarbon matrices is imperative for numerous reasons. The presence of these components in materials such as vacuum gas oils (VGO) can cause severe and costly corrosion to downstream pipeline components. In addition, harmful emissions and catalyst poisoning can reduce the efficiency of the catalytic process. All of these push the need to monitor concentrations of these elements.

The MultiTek HS-IC complies with ASTM D7359 and is the only instrument on the market that has the ability to determine total fluoride, chloride and total sulfur 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. Ultimately, sulfur containing compounds are oxidized in an unequalled occurrence to sulfite (SO_3) and sulfate (SO_4). Gases are condensed, absorbed, and then transferred by the MultiTek to a preconcentration column injection system on the Ion Chromatograph (IC) for conductivity analysis.

REACTIONS

- **Halogen Reaction by CIC**

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

- **Sulfur Reaction by CIC**

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

EXPERIMENTAL CONDITIONS

- **Instrumentation**

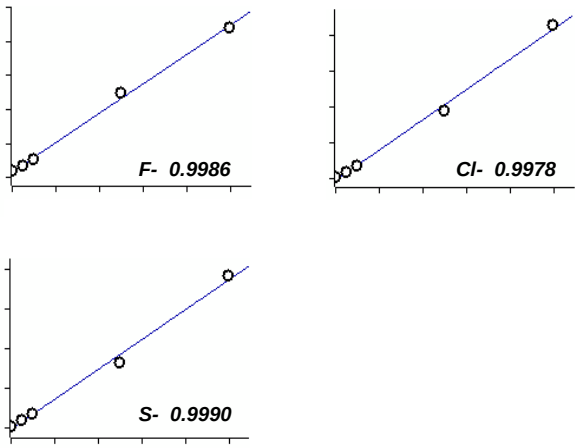
Antek MultiTek HS-IC, Antek Model 740 boat inlet system, Antek Model 735 syringe drive, autosampler and suppressed IC system equipped with a trace halides kit.

- **Instrument Parameters**

Autosampler Inj Volume (µL)	20
Preconcentration Volume (mL)	2.75
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

APPLICATION NOTE

CALIBRATION



- **Standards**

Calibration was performed through entire sample flow path using certified standard materials. Calibration standards were prepared with Fluorobenzoic acid, 2,4,5-Trichlorophenol and dibenzothiophene in xylene for F, Cl, and S respectively.

RESULTS

	Fluoride	Chloride	Sulfur
Certified P-Xylene 0.50ppm	0.46	0.48	0.45
Certified P-Xylene 1.00ppm	1.10	0.95	0.94
1.00ppm P-Xylene QC	1.06	1.05	1.17
ASTM PTP (x3 AVG)	ND	0.10	0.03
	% RSD	3.03	8.8

*ND; results lower then quantification limits allow.

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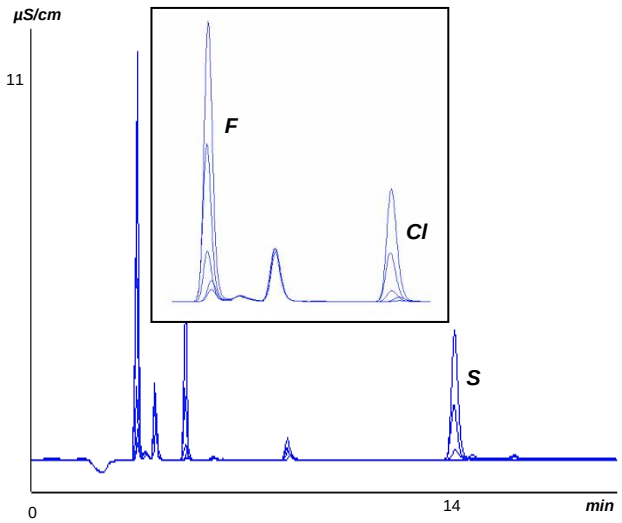
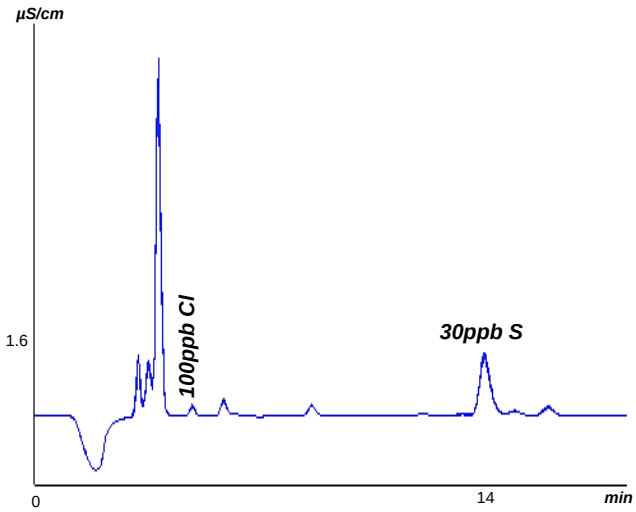
CONCLUSION

The MultiTek HS-IC is the only instrument on the market that has the ability to determine total fluoride, chloride and total sulfur in aromatic samples all in a single instrument. The results demonstrate that the MultiTek Analyzer coupled with Ion Chromatography provides a sensitive, automated and reliable elemental analysis of aromatic hydrocarbon compounds. This analysis will help optimize refinery processes by monitoring these components.

CHROMATOGRAPHY



Calibration Chromatography (0 / 0.5 / 1 / 5 / 10ppm)



*3 replicates ASTM PTP. Fluoride *ND*

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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