

MultiTek[®] Trace Halides and Sulfur in Aviation Turbine (Jet) Fuel by Oxidative Pyrohydrolytic Combustion followed by Ion Chromatography Conductivity Detection

- Fully Automated Combustion System
- Halogen & Sulfur Determination
- Sample Versatility

Keywords:

MultiTek[®], pyrohydrolysis, petrochemical, trace halides, Aviation Turbine (Jet) Fuel, fluoride, chloride, bromide, sulfur, sulfate, combustion ion chromatography (CIC)

INTRODUCTION

Tighter specifications on commercial aviation turbine (jet) fuels and more efficient petrochemical plant operations have introduced the need for an instrument capable of detecting trace amounts of corrosive components. Fluoride, chloride, bromide and sulfur can have the biggest influences on corrosion and damage to processing equipment of refineries, pipelines, and storage facilities.

This procedure analyzes halides and sulfur in commercial grade aviation turbine (jet) fuel. Samples undergo pyrohydrolysis at 1050°C where halogen containing compounds are converted into an acid gas state. Sulfur compounds are oxidized into sulfur dioxide (SO₂) and at higher concentrations, sulfite (SO₃) & sulfate (SO₄). Testing has demonstrated that the complete oxidation to SO₄ is repeatable at trace concentrations less than 1mg/kg sulfur. When combustion is completed, gases are condensed and absorbed in a UHP 18.2 M Ω water. The solution is then transferred to a preconcentration column on the lon Chromatograph (IC) for conductivity analysis.

REACTIONS

Halogen Reaction $R - F + R - Cl + R - Br \xrightarrow[1050^{\circ}C + O_2 + H_2O]{} HF + HCl + HBr$ Sulfur Reaction $R - S \xrightarrow[1050^{\circ}C + O_2 + H_2O]{} SO_x \xrightarrow[scrubber solution]{} SO_4$

To ensure the highest oxidation conversion from sulfite to sulfate, optima grade hydrogen peroxide can be added to the scrubber solution. This experiment did not include hydrogen peroxide due to complete oxidation to sulfate (SO₄) at lower concentration levels and the unavailability of a reagent without any contamination of trace level halides.

EXPERIMENTAL CONDITIONS

Instrumentation

Antek MultiTek Horizontal IC, Trace Halides Kit, Antek Model 740 boat inlet system, Antek Model 735 syringe drive, autosampler and suppressed IC system.



Instrument Parameters

748 Injection Volume (µL)	25
Preconcentration Volume (mL)	2.75
GFC1- Ar/He (mL/min)	130
GFC2- Pyro O2 (mL/min)	450
GFC3- Steam O2 (mL/min)	5
GFC4- Carrier O2 (mL/min)	30
Furnace (°C)	1050
Sample Burn Time (mm:ss)	5:00

Standards

CIC calibration is performed through entire sample flow path using certified standard materials.



APPLICATION NOTE





A four level calibration is made by diluting stock solutions of chlorocyclohexane, bromobenzene, 1,4-difluorobenzene, and dibenzothiophene in isooctane. Bromide calibration curve produced three points due to the first level (blank) producing no peak response.

Limit of Detection

	Fluoride	Chloride	Bromide	Sulfur
LOD (µg/kg)	100	75	25	75

Highest quality (+99.9%) reagents, gases and 18.2 M Ω water are required to achieve these LODs.

RESULTS

Injection	Chloride (µg/kg)	Sulfur (µg/kg)
1	64	140
2	67	140
3	64	137
4	66	144
5	67	145
6	67	144
Average	66	142
RSD	2.2%	2.2%

Repeatability of ASTM Aviation Turbine (Jet) Fuel. This application was calibrated for bromide and fluoride but none was detected.

CONCLUSION

These results demonstrate that the MultiTek Analyzer equipped with a trace halides kit and Ion Chromatography provides a sensitive, automated and reliable analysis of halides and sulfur in aviation turbine (jet) fuel. This analysis will allow the refinery and other processing to monitor sub ppm concentration levels. Finding these contaminants early prevents costly repairs in the future.

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Antek's MultiTek is the only instrument on the market that combines sulfur, nitrogen, and halides testing all in one. Compact, powerful, automated, and multi-configurable, it's the perfect solution for today's increasing demand for fast and accurate detection 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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