

MultiTek® Halides in Coal by Oxidative Pyrohydrolytic Combustion followed by Ion Chromatography Detection

- Fully Automated Combustion System
- Simultaneous Halogen Determination
- Sample Versatility

Keywords:

MultiTek®, Pyrohydrolysis, Halides, Coal, CIC

EXPERIMENTAL CONDITIONS

• Instrumentation

Antek MultiTek® Horizontal, Antek Model 740 boat inlet system, Antek Model 735 syringe drive, and suppressed IC system.



• Instrument Parameters

Target Sample Weight (mg)	25
IC Sample Loop (µl)	250
GFC1- Ar/He (ml/min)	130
GFC2- Pyro O2 (ml/min)	450
GFC4- Carrier O2 (ml/min)	30
Furnace (°C)	1050
Sample Burn Time (mm:ss)	04:00

**Standard IC conditions

INTRODUCTION

The combustion of coal results in the release gaseous pollutants in the atmosphere. Majority of fluorine, chlorine, and bromine in coal is converted HF, HCl and HBr. These gaseous forms can contaminate farm lands and affect the human respiratory/digestive system. Since these pollutants are developed from the combustion of coal, it is important to limit these emissions and monitor the concentrations of the halogens in fuels.

This procedure analyzes ground coal powder which undergoes pyrohydrolysis at 1050°C. Tungsten catalyst addition was initially tested. Additional reactivity caused by tungsten did not improve peak response therefore unnecessary. After combustion, gases are condensed and absorbed in a solution composed of UHP 18.2 MΩ water and phosphate internal standard. Once the absorption process is complete, the solution is transferred by the MultiTek® to an injection valve on the Ion Chromatograph (IC) for conductivity analysis.

Identification	Sample Size (mg)		Fluorine		Chlorine		Bromine	
ASTM 89-2-100	24.9	26.2	60.3	59.9	13.2	12.1	5.6	5.3
ASTM 89-5-095	25.0	25.1	164.9	194.9	47.7	60.4	8.3	8.7
ASTM 89-10-086	24.6	25.8	79.5	86.7	1306.8	1477.2	19.5	23.9
*NIST 1632b	27.8		48.6		1201.8		19.5	
*NIST 1632c	27.2		80.8		1177.5		19.7	

**NIST 1632b:

Actual concentrations Fluorine= 41.7 ± 3.2ppm / Chlorine= 1260ppm / Bromine= 17ppm

**NIST 1632c:

Actual concentrations Fluorine= 72.7 ± 6.8ppm / Chlorine= 0.1139% ± 0.0041 / Bromine= 18.7 ± 0.4ppm.

• Standards

IC calibration is performed independently of the furnace using inorganic halides dissolved in UHP 18.2 MΩ water.

Correlation

0.010ppm-1.0ppm (Fluorine)	
Correlation Coefficient	0.999959
Curve Fit	Linear
0.010ppm-1.0ppm (Bromine)	
Correlation Coefficient	0.998014
Curve Fit	Linear
0.010ppm-1.0ppm (Chlorine)	
Correlation Coefficient	0.999706
Curve Fit	Linear

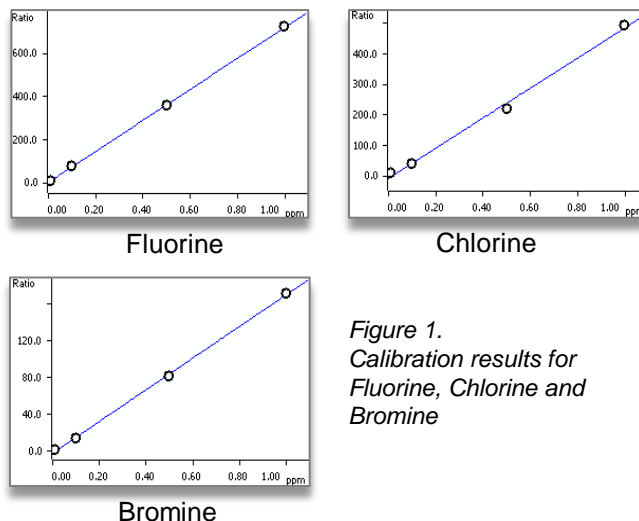


Figure 1.
Calibration results for
Fluorine, Chlorine and
Bromine

Repeatability/Stability Data (ppm)

Repeatability of ASTM coal sample (X6)
measured over a three day period:

	F	Cl	Br
Average	17.99	6038.36	121.45
%RSD	4.16	3.10	4.40

CONCLUSION

The results for fluorine, chlorine, bromine are within the certified concentration of the NIST reference material. The combustion accelerant, tungsten, did not show an increase in response for all analytes so it is not required. Depending on actual concentrations, recommended sample size is 25.0mg.

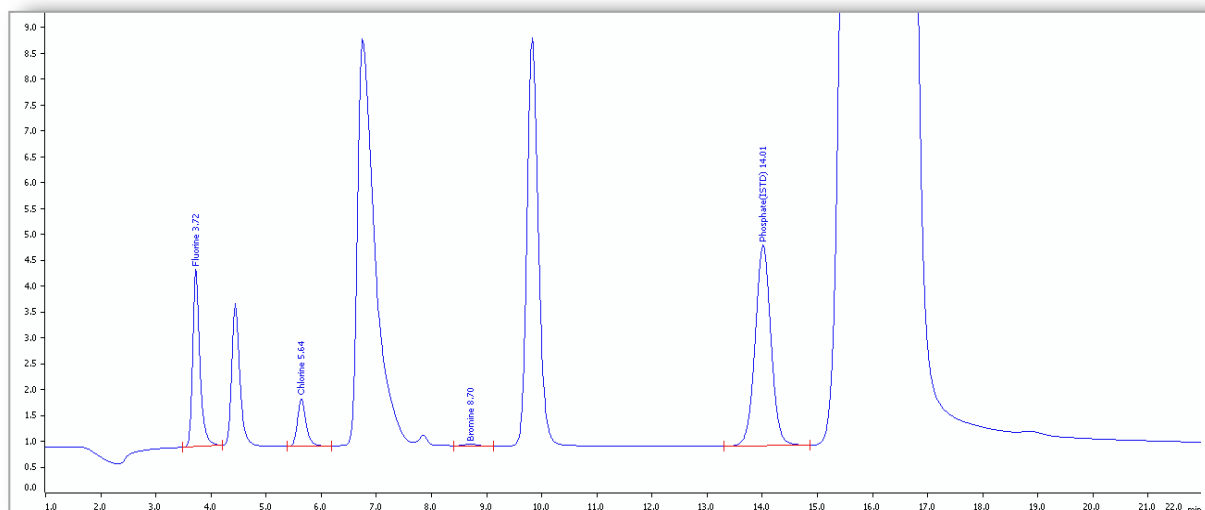


Figure 2. ASTM 90-1; Sample tested for repeatability and stability

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.