APPLICATION NOTE

MultiTek® Fluoride in Aluminum Oxide by Oxidative Pyrohydrolytic Combustion followed by Ion Chromatography Detection

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

Keywords:
MultiTek®, Pyrohydrolysis, Halides, Alumina, Bauxite, alumina trihydrate, fluoride, CIC, combustion IC

INTRODUCTION

The manufacture of aluminum oxide begins with the extraction of alumina from bauxite. Bauxite is an aluminum ore and is our main source of aluminum. Fluoride is naturally found in bauxite, and follows in trace amounts with alumina during extraction and in the formation of alumina trihydrate. During calcination of trihydrate, fluoride can cause abnormal grain growth which results in difficulty in grinding further in process. These grain abnormalities push the need to monitor fluoride throughout the manufacturing process.

This procedure analyzes aluminum oxide powder which undergoes pyrohydrolysis at 1050ºC. 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 system on the Ion Chromatograph (IC) for conductivity analysis.

Dilution factors derived from known volume of scrubber solution divided by actual sample mass which undergoes pyrohydrolysis and absorption.

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 (ºC)	1050
Sample Burn Time (mm:ss)	04:00

Sample Size (mg)	Concentration ppm (Before Dilution Factor)	Dilution Factor	Final Concentration
26.4	0.260	212	55
26.6	0.268	211	56
26.8	0.242	209	51
25.4	0.247	220	54
26.5	0.268	211	56
26.7	0.258	210	54
26.3	0.247	213	53
		AVG	54
		RSD	3.27%

- **Standards**

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

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- **Repeatability/Stability Data (ppm)**

Repeatability of AlO3 sample (X7) measured	
Average	54
RSD	3.27%

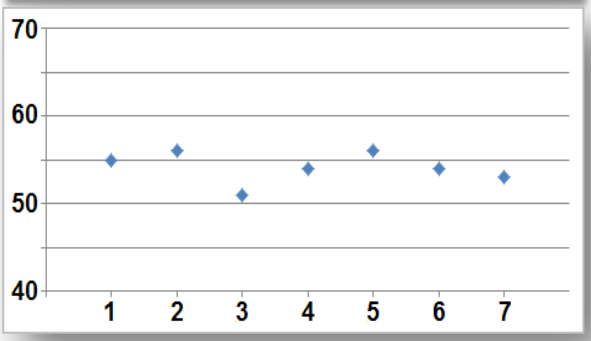

Figure 1. Repeatability results for Fluoride

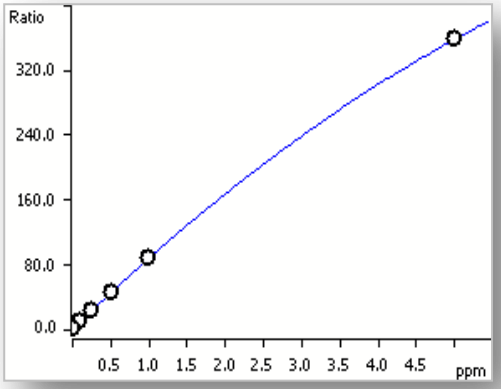

Figure 2. Calibration results for Fluoride; 0.01ppm-5ppm

- **Correlation**

0.010ppm-1.0ppm (Fluoride)	
Correlation Coefficient	0.99999
Curve Fit	Quadratic

- **Limit of Blank & Limit of Detection**

  Data used for these calculations produced a signal to noise ratio greater than 3:1

LOB= AVG (Blank) + 1.645 (SD_{Blank}) LOD= LOB + 1.645 ($SD_{low\ conc.\ Sample}$)	
Limit of Blank (LOB)	18.9 ppb
Limit of Detection	1.29ppm

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CONCLUSION

These results demonstrate that the MultiTek® Analyzer coupled with Ion Chromatography provides a sensitive, automated and reliable analysis of fluoride in Al2O3. This analysis will allow the refinery and other processing to monitor process concentration levels to prevent grain abnormalities.

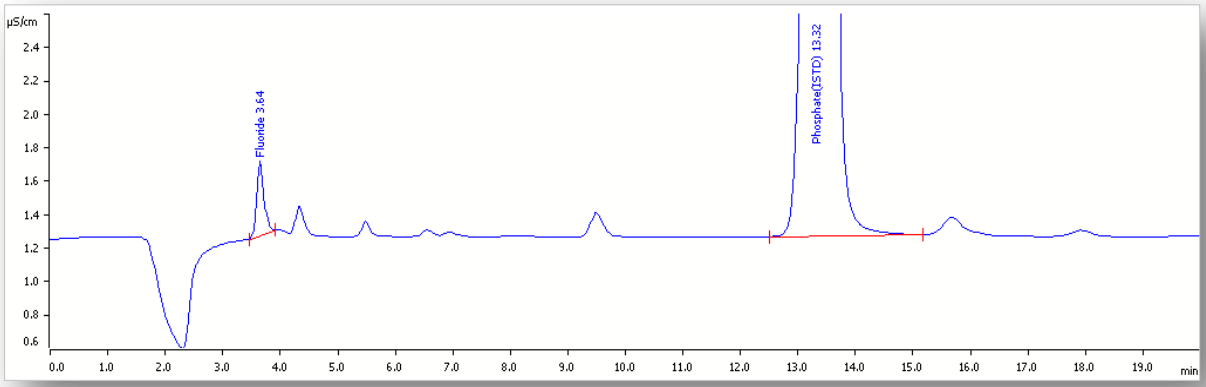

Figure 3. Reference Standard Chromatography; Sample tested for LOB and LOD

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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