

MultiTek® Halides in Low Density Polyethylene by Oxidative Pyrohydrolytic Combustion followed by Ion Chromatography Detection

- Fully Automated Combustion System
- Halogen Determination
- Sample Versatility

Keywords:

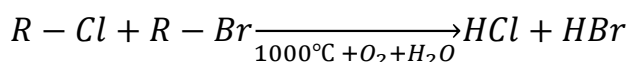
MultiTek®, pyrohydrolysis, halides, low density polyethylene, chloride, bromide, CIC, combustion IC

INTRODUCTION

Majority of governments around the world have developed legislation to monitor elements in consumer products to protect human and animal life. In Europe, legislation has been passed to limit quantities of certain elements in various products. Directives 94/62/E (plastic packaging), 2002/95/EC (electric and electronic packaging) and 2000/53/EC (old vehicles).

This procedure analyzes European Reference Materials ERM-EC680k. These low density polyethylene pellets undergo pyrohydrolysis at 1050°C. The combustion process is accelerated by the addition of tungsten oxide powder. Once combustion is completed, gases are condensed and absorbed in a solution composed of UHP 18.2 MΩ water and phosphate internal standard. The solution is then transferred by the MultiTek® to an injection system on the Ion Chromatograph (IC) for conductivity analysis.

REACTION



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)	20
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)	08:00

• Standards

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

APPLICATION NOTE



RESULTS

Sample Size (mg)	Chloride	Bromide
17.9	86.4	71.5
16.3	88.7	72.8
15.4	84.4	74.1
16.1	85.4	68.8
18.0	89.8	76.1
15.5	87.7	74.4
16.7	87.3	71.8
Average	87.1	76.0
RSD	2.1%	3.6%
**% Rec.	87%	76%

Figure 1. Low Density Polyethylene (LDPE) before combustion accelerant

Sample Size (mg)	Chloride	Bromide
16.9	89.6	80.3
19.1	89.3	81.8
16.9	89.2	85.5
18.2	90.4	81.8
17.3	93.5	83.2
17.0	91.7	80.5
Average	90.4	82.5
RSD	1.6%	1.9%
**% Rec.	91%	90%

Figure 2. Low Density Polyethylene (LDPE) after combustion accelerant addition.

** Certified values obtained from European Reference Materials EC680k. Percent recovery calculated using average sample replicates (Cl= 102 ± 3 mg/kg Br= 96 ± 4 mg/kg).

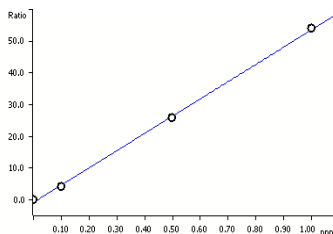


Figure 3. Chloride Calibration

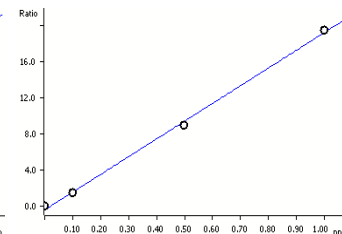


Figure 4. Bromide Calibration

• Correlation

0ppm-1.0ppm (Fluoride)	Chloride	Bromide
Correlation Coefficient	0.9996	0.9990
Curve Fit	Linear	Linear

CONCLUSION

These results demonstrate that the MultiTek® Analyzer coupled with Ion Chromatography provides a sensitive, automated and reliable analysis of halides in low density polyethylene. A combustion improver such as tungsten oxide is required to yield best recoveries. This analysis will allow the refinery and other processing to monitor halide concentration levels to satisfy governmental legislation.

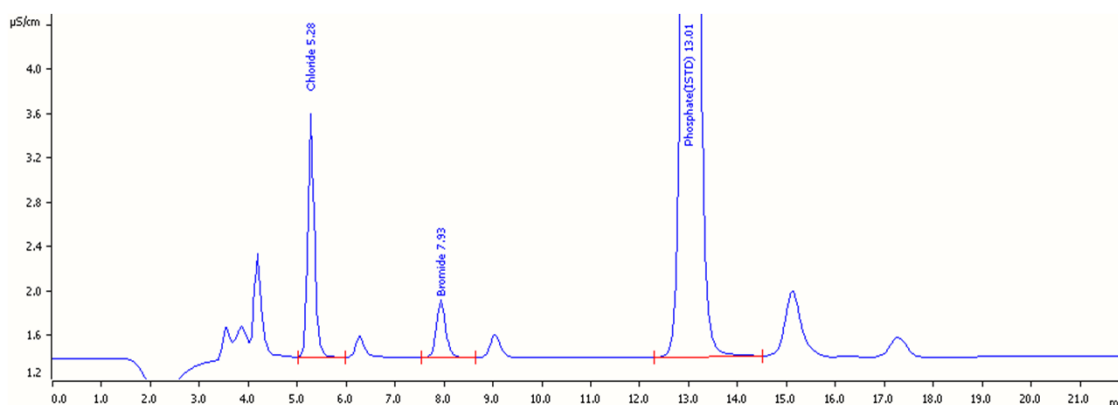


Figure 5. ERM Reference Material EC680k; Low Density Polyethylene

Antek 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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