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# Combustion IC System

Halogen and sulfur determinations-pyrolysis of difficult samples

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# Fully Automated Analysis of Halogens and Sulfur in Petrochemicals, Industrial Chemicals, and Consumer Products

Fluorine, chlorine, bromine, and sulfur can cause corrosion in many industrial processes, decrease the lifetime of many catalysts, and cause environmental pollution. The combustion IC system developed by Mitsubishi (Japan) and Thermo Fisher Scientific (USA) can be used for automated qualitative and quantitative analysis of halogens and sulfur in samples comprised of difficult matrices - intractable solids, semisolids, liquids and even gasses. Samples can be as diverse as petrochemicals, coal-based chemicals, construction materials, chemicals, polymers, and pharmaceutical intermediates and finished formulations.

## **Analysis of Corrosive Species in Complex Matrices**

Petrochemicals, gaseous samples, solid samples, and complex chemicals are very difficult to analyze with conventional ion chromatography (IC). Sample preparation is required to extract analytes or remove interfering matrices, and these techniques are costly and labor intensive. Automated combustion IC reduces the time and labor for determination of corrosive halogens and sulfur in difficult samples, by eliminating complex sample preparation steps. The samples are pyrolyzed in an oxidizing atmosphere, the resultant vapors are absorbed in an aqueous solution, then introduced directly into the IC system for analysis. This automated method is highly sensitive, easy-to-use, saves time and produces fewer environmental contaminants than other sample preparation procedures such as acid digestions, or back extractions from organic solutions.

The system provides:

- Pyrolysis of complex samples
- Analysis of gaseous, solid, and viscous samples
- High precision and accuracy
- Full compliance with ASTM D7359-08 and other international methods the analysis of many types of materials.

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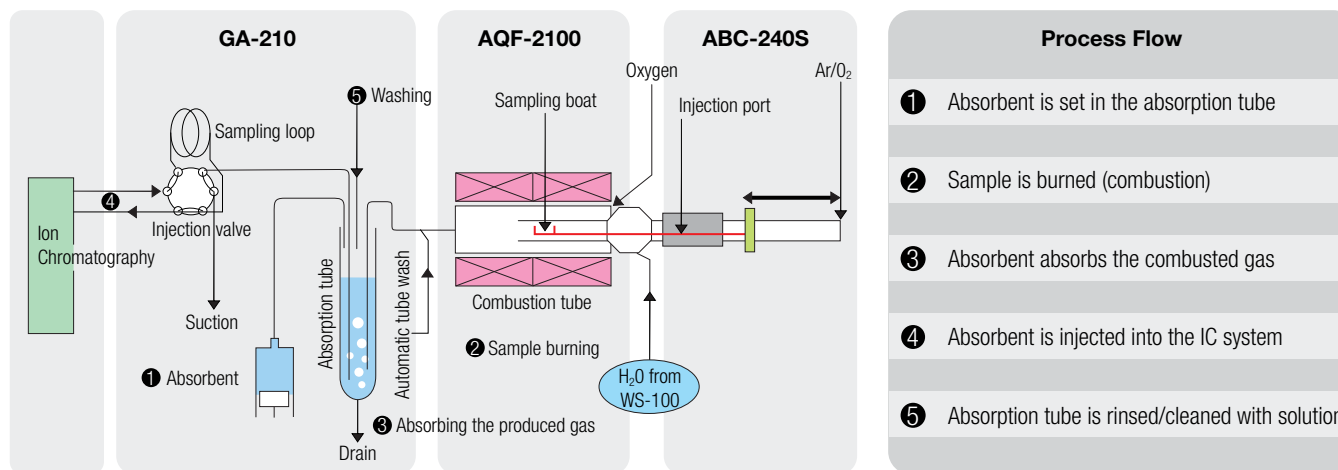
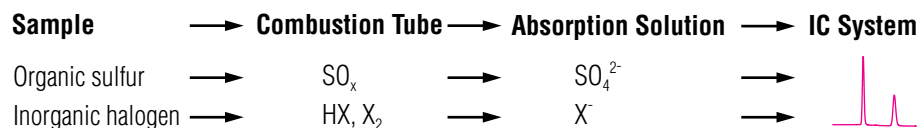
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## Theory of Combustion IC

Solid or semisolid samples are introduced into the AQF-2100 using the ABC-240S Automatic Boat Controller. Pyrolysis occurs at 800–1100 °C. The samples are oxidized by O<sub>2</sub> at high temperature and the vapors are sparged through the absorbing solution. The samples in the absorbing solution are transferred to the IC system for analysis. Phosphate may be added to the absorbing solution as an internal standard to calibrate the analytical results. H<sub>2</sub>O<sub>2</sub> is added in the absorbing solution to oxidize SO<sub>2</sub>, which results from the incomplete oxidization of S, to SO<sub>3</sub>, which is then quantitated as SO<sub>4</sub><sup>2-</sup>.

## Major Applications

- S, Cl, F, and Br in plastics
- Cl, F, Br in pharmaceutical raw materials and finished ingredients
- Cl in epoxy resins
- S, Cl, and Br in industrial lumber plates
- Br in disposable (one-time use) polymers
- Cl in lubricating oil
- F, Br, and I in soil samples
- F in ores
- Cl and F in aluminum powder
- F in polishing agents



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**Thermo Scientific™  
Dionex™ Integrion™  
HPIC™ System**

**Mitsubishi  
GA-210 Gas  
Absorption Unit**

**Mitsubishi  
AQP-2100**

**Mitsubishi  
ABC-240S  
Automatic Boat  
Controller**

**PC**

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## GA-210 Absorbance System Specifications

### Absorption Tube:

Heat-resistant glass, 10 or 20 mL

### Six-Way Valve

### Injection Pump:

5 mL high-pressure channel, Teflon® or PEEK

- 1 Absorbance tube
- 2 Injection valve
- 3 Absorbing agent distributor
- 4 IC connection tube
- 5 Introduction of combustion gas
- 6 Introduction of liquid sample



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### Thermo Scientific™ Dionex™ Aquion™ IC System

- Integrated system
- Single channel system
- LED front panel
- High-performance dual-piston pump
- Thermally controlled digital conductivity cell
- Thermo Scientific™ Dionex™ ERS™ 500 Electrolytically Regenerated Suppressor
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software



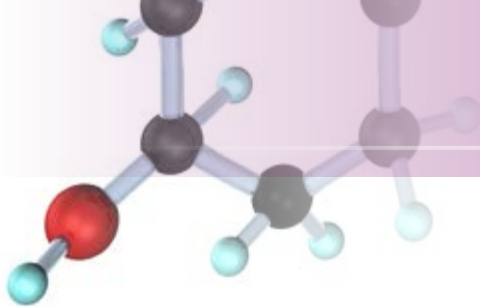
### Thermo Scientific™ Dionex™ Integriion™ HPIC™ System

- Integrated system
- Single channel system
- Reagent-Free™ IC system
- Eluent generator (analytical only)
- Dionex ERS 500 suppressor
- Thermo Scientific™ Dionex™ CR-TC Continuously Regenerated Trap Column
- High-performance dual-piston pump
- Supports HPIC chemistries
- Thermally controlled digital conductivity cell
- Dual temperature zone control
- Column heater
- Compartment heater/ cooler
- Chromeleon CDS software



### Thermo Scientific™ Dionex™ ICS-5000+™ HPIC™ System

- Modular system
- Single or dual channels system
- Reagent-Free™ IC system
- Eluent generator (analytical and capillary)
- Dionex ERS 500 suppressor
- Dionex CR-TC Trap Column
- High-performance dual-piston pump
- Supports HPIC chemistries
- Isocratic/ quaternary gradient analytical pump options
- Isocratic capillary pump option
- Thermally controlled digital conductivity cell
- Dual temperature zone control
- Column heater/ cooler
- Compartment heater/ cooler
- Automation manager
- Chromeleon CDS software



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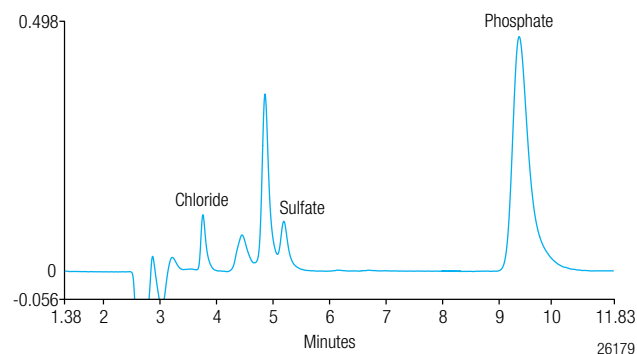
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## Simplified Analysis for Complex Samples Using Combustion Ion Chromatography (CIC)

### Chlorine and Sulfur in Petroleum Samples

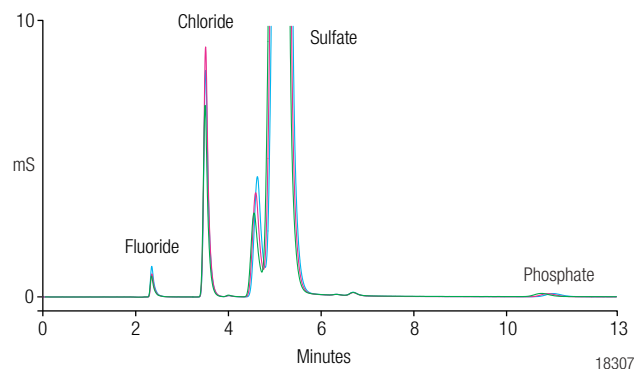
Sulfur in petroleum products causes corrosion in pipelines, poisons catalysts, affects stability during storage, and can decrease product performance. Chlorine corrodes equipment, clogs pipelines, and increases catalyst consumption during manufacturing and limits product quality.



Chlorine and sulfur in a refinery intermediate, detected as chloride and sulfate.

### Chlorine and Sulfur in Coal

Chlorine content of coal is a key parameter to be determined for mercury emissions and control. For CIC analysis, ground coal powder is pyrolyzed in the combustion chamber (AQF) and the gaseous combustion products are introduced to the absorption equipment using argon gas. Sulfur dioxide (SO<sub>2</sub>), hydrogen fluoride (HF), and hydrogen chloride (HCl) are trapped by an aqueous absorption solution consisting of a known amount of phosphate (internal standard) and a small percentage of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>).



Reproducibility of coal samples (overlay of three injections).

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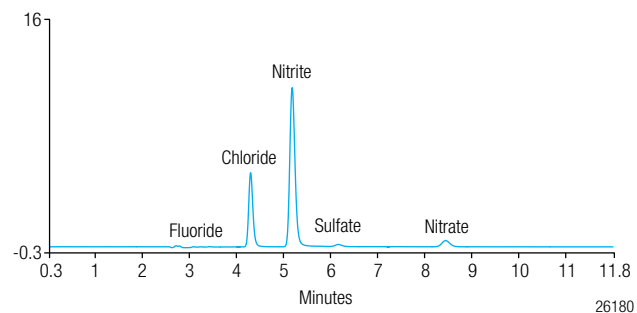
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### Halogens in Printing Dyes and Inks

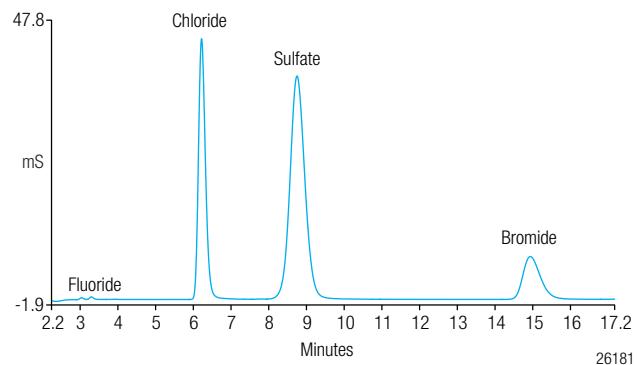
Manufacturers of printing dyes and inks need to ensure that corrosive halogens are to low concentrations, to prevent high failure rates of print cartridges due to corrosion of the product circuitry. CIC provides a simple solution in determining halogens in such difficult, high-organic matrices. The organic matrix is eliminated by pyrolysis and the halides produced are absorbed in a solution which is directly injected into an IC system for rapid analysis.

### Halogens from Electronic Components

Brominated flame retardants have been used as flame retardants in printed circuit boards. This is now prohibited by Restriction of Hazardous Substances (RoHS) and WEEE (Waste from Electrical and Electronic Equipment) regulations. CIC provides a fast and reliable method for measuring the halogen content of various consumer products and the associated waste streams. Lack of bromide in the sample is evidence that prohibited brominated compounds are not present.



Halogens and sulfur in black dye, determined in their ionic forms.



Fluorine, chlorine, sulfur, and bromine from electronic components.



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## For More Information

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On-Demand  
Webcast

Webcast:

**Combustion Ion Chromatography - Solving Difficult to Process Analytical Samples**

Download  
Poster  
Note

Poster Note:

**Combustion Ion Chromatography - Enhancing Halogen Detection Using Preconcentration Methods**

Download  
AN  
Note

Application Note

**Determination of Halogens in Coal Using Combustion Ion Chromatograph**

Download  
Poster  
Note

Poster Note:

**Accurate and Reproducible Determination of Halogens in Coal Using Combustion Ion Chromatography**

[www.thermofisher.com](http://www.thermofisher.com)

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