Table of Contents

Combustion IC System

Halogen and sulfur determinations-pyrolysis of difficult samples

thermo scientific

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, 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 D7994 (F, Cl and S in LPG) and other international methods for the analysis of many types of materials.

thermo scientific

Table of Contents

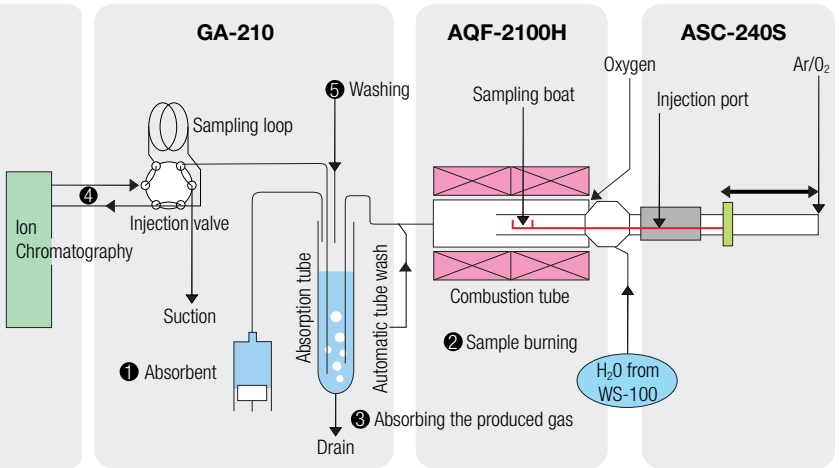
Theory of Combustion IC

Solid or semisolid samples are introduced into the AQF-2100H using the ASC-240S Automatic Boat Controller. Pyrolysis occurs at 800–1100 ˚C. The samples are oxidized by O_2 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_2O_2 is added in the absorbing solution to oxidize SO_2, which results from the incomplete oxidization of S, to SO_3, which is then quantitated as SO_4^{2-}.

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
- F, Cl, Br, and I in 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

Sample	→ Combustion Tube	→ Absorption Solution	→ IC System
Organic sulfur	→ SO_x	→ SO_4^{2-}	→
Inorganic halogen	→ HX, X_2	→ X^-	→



Process Flow

1. Absorbent is set in the absorption tube
2. Sample is burned (combustion)
3. Absorbent absorbs the combusted gas
4. Absorbent is injected into the IC system
5. Absorption tube is rinsed/cleaned with solution

thermo scientific

Table of Contents

Thermo Scientific™ Dionex™ Integrion™ HPIC™ System

Mitsubishi GA-210 Gas Absorption Unit

Mitsubishi HF-210 Horizontal Furnace

Mitsubishi ASC-270S Automatic Sample Changer

PC

thermo scientific

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. Absorbtion tube
2. Injection valve
3. Absorbing agent distributor
4. IC connection tube
5. Introduction of combustion gas
6. Introduction of liquid sample

thermo scientific

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™ Integrion™ 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

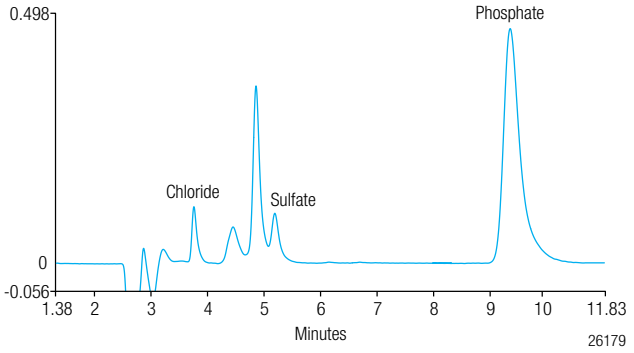
Thermo Scientific™ Dionex™ ICS-6000 HPIC™ System

- Modular system
- Single or dual channels system
- Reagent-Free™ IC system
- Eluent generator (analytical and capillary)
- Dionex DRS 600 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

Simplified Analysis for Complex Samples Using Combustion Ion Chromatograpy (CIC)

Chlorine and Sulfur in Petroleum Samples

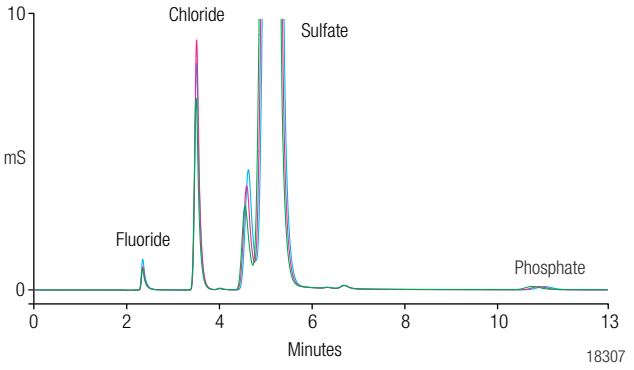
Sulfur in petroleum products causes corrosion in pipelines, poisons catalysts, affects stability during storage, and can decrease product performance. Chlorine corrodes equipment, clogs pipelines, and increases catalyst consumption during manufacturing and limits product quality.



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 pyrolized in the combustion chamber (AQF) and the gaseous combustion products are introduced to the absorption equipment using argon gas. Sulfur dioxide (SO_2), 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_2O_2).

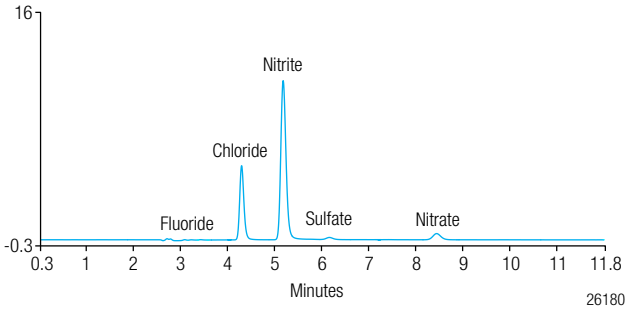


Reproducibility of coal samples (overlay of three injections).

thermo scientific

Halogens in Printing Dyes and Inks

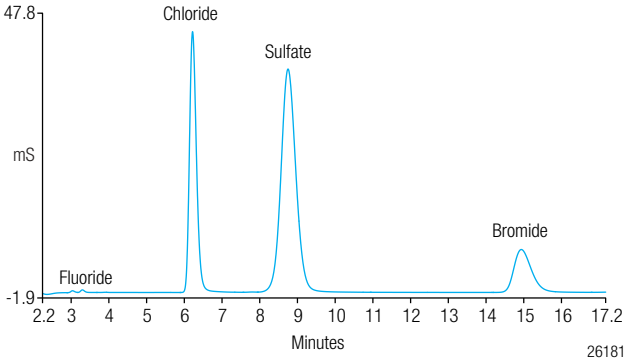
Manufacturers of printing dyes and inks need to ensure that corrosive halogens are to low concentrations, to prevent high failure rates of print cartridges due to corrosion of the product circuitry. CIC provides a simple solution in determining halogens in such difficult, high-organic matrices. The organic 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 and sulfur in black dye, determined in their ionic forms.

Halogens from Electronic Components

Brominated flame retardants have been used as flame retardants in printed circuit boards. This is 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.



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

thermo scientific

Table of Contents

For More Information

Download Application Note — Determination of Halogens in Polymers and Electronics Using a Combustion Ion Chromatography System

Download Application Note — Determination of Chlorine, Bromine, and Sulfur in Polyethylene Materials Using Combustion Ion Chromatography

Download Application Note — Determination of Adsorbable Organic Halogen in Wastewater Using a Combustion Ion Chromatography System

Download Application Note — Determination of Halogens in Coal Using Combustion Ion Chromatography

thermofisher.com/combustionIC

BR71114-EN 0418S

ThermoFisher SCIENTIFIC