

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 Dionex Corp. (USA) can be used for automated qualitative and quantitative analysis of halogens and sulfur in petrochemicals, coal-based chemicals, and construction materials, chemicals, and polymers.

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, but these techniques can be costly and labor intensive. Automated combustion IC reduces the time and labor for determination of corrosive halogens and sulfur in difficult samples. The samples are pretreated by step combustion, absorbed in an aqueous solution, then introduced directly into the IC system for analysis. This automated

method is highly sensitive, easy-touse, saves time, and produces fewer environmental contaminants than other analytical processes. The system provides:

- Pyrolysis of complex samples
- Analysis of gaseous, solid, and viscous samples
- High precision and accuracy
- Fully compliant with ASTM D7359-08
 Standard Test Method for Total Fluorine,
 Chlorine, and Sulfur in Aromatic
 Hydrocarbons and Their Mixtures by
 Oxidative Pyrohydrolytic Combustion
 followed by Ion Chromatography
 Detection (Combustion Ion
 Chromatography-CIC)

Now sold under the Thermo Scientific brand





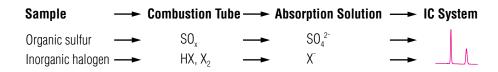
Theory of Combustion IC

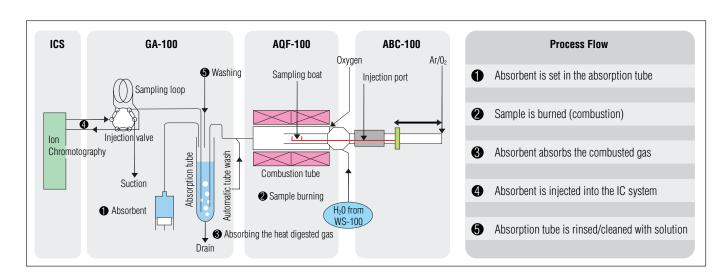
Samples are introduced into the AQF-100 using the ABC-100 Automatic Boat Controller. Pyrolysis occurs at $800-1100\,^{\circ}\text{C}$. The samples containing halogen and sulfur are oxidized by O_2 and absorbed in the absorbing solution. The samples in the absorbing solution are transferred to the IC system for analysis.

Phosphate is added into the absorbing solution as an internal standard to calibrate the analytical results. H_2O_2 is added into the absorbing solution to oxidize SO_2 , which is produced from the incomplete oxidization of S, to SO_3 , so it can be analyzed in the form of SO_4^2 .

Major Applications

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









GA-100 Absorbance System Specifications

Absorbtion Tube:

Heat-resistant glass, 10 or 20 mL

Six-Way Valve

Injection Pump:

5 mL high-pressure channel, Teflon® or PEEK $^{\mathsf{m}}$

- Absorbtion tube
- 2 Injection valve
- 3 Absorbing agent distributor
- IC connection tube
- Introduction of combustion gas
- Introduction of liquid sample



ICS-900 IC system

- High-performance, low-pulse high-pressure pump
- Thermally controlled digital conductivity cell
- MMS[™] MicroMembrane[™] suppressor
- Chromeleon® SE software



ICS-1100 IC system

- LED front panel
- High-performance dualpiston pump
- Thermally controlled digital conductivity cell
- Electrolytic suppressor
- Chromeleon software



ICS-1600 IC system

- LCD high-resolution color touch panel
- High-performance dualpiston pump
- Thermally controlled digital conductivity cell
- Electrolytic suppressor
- Column heater
- Chromeleon software



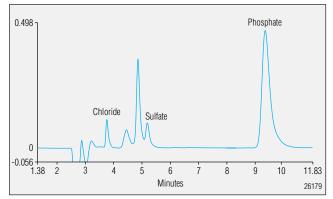
ICS 2100 IC system

- LCD high-resolution color touch panel
- Reagent-Free™ IC system
 - -- Eluent generator
 - -- Electrolytic suppressor
 - -- CR-TC
- High-performance dualpiston pump
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Simplified Analysis for Complex Samples Using Combustion Ion Chromatograpy (CIC)

Chlorine and Sulfur in Petroleum Samples

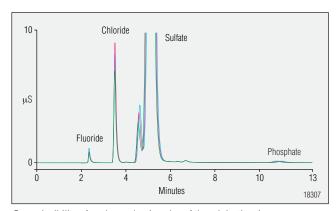
Sulfur in petroleum products causes corrosion in pipelines, poisons catalysts, affects stability during storage, and can decrease product performance. Chlorine corrodes equipment, clogs pipelines, and increases catalyst consumption during manufacturing and adversely affects product quality.



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

Chlorine and Sulfur in Coal

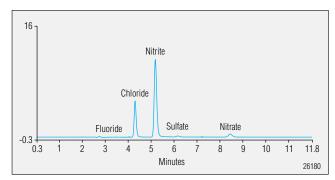
Chlorine content of coal is a key parameter to be determined for mercury emissions and control. For CIC analysis, ground coal powder is pyrolized in the combustion chamber (AQF) and the gaseous combustion products are introduced to the absorption equipment using argon gas. Sulfur dioxide (SO $_2$), hydrogen 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 $_2$ O $_2$).



Reproducibility of coal samples (overlay of three injections).

Halogens in Printing Dves and Inks

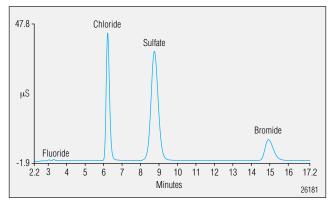
Manufacturers of printing dyes and inks need to ensure that corrosive halogens are at low concentration, to prevent high failure rates of the print cartridges due to corrosion of the product circuitry. CIC provides a simple solution in determining halogens in such difficult, high-organic matrices. The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

Halogenated materials have been used by the electronics industry for decades, raising concerns about their environmental impact. Electronics manufacturers in the European Union must monitor their products and processes to comply with the Restriction of Hazardous Substances (RoHS) and Waste from Electrical and Electronic Equipment (WEEE) Directives. CIC provides a fast and reliable method for measuring the halogen content of various consumer products and the associated waste streams.



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

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