Combustion Ion Chromatography

Fast and reliable determination of halogens and sulfur
Combustion Ion Chromatography (CIC) extends the range of ion chromatography to all types of combustible samples. The focus here is primarily on the simultaneous determination of the various halogens and sulfur in widely differing matrices.

Metrohm’s CIC System, including sample preparation, is completely automated. CIC is superior to offline digestion methods with regard to sample throughput on the one hand and precision and correctness of the results on the other. With CIC, unlike with alternative methods, the concentrations of the different halogens can each be determined separately.

The principle
In CIC the samples are first digested under an argon atmosphere in the oven unit and then burnt with oxygen. In the 920 Absorber Module, the resulting gaseous compounds are passed into an absorption solution, which is then transferred inline to a Metrohm ion chromatography system, where it is analyzed.
The advantages of CIC at a glance

- Extended range of application of IC for all kinds of combustible samples
- Simultaneous determination of sulfur and halogens
- Quantification of the concentration for each halogen
- Ideal for checking the latest analytical standards with regard to halogen-free products (RoHS, WEEE ...)
- High sample throughput
- High precision and accuracy
- MagIC Net™ ion chromatography software for control and data management – all information in one sample table or report
- Flame sensor ensures optimum combustion time with an universal method
- FDA and GLP standards are met
- Calibration with just one standard thanks to Metrohm intelligent Partial Loop Injection Technique (MiPT)
- Completely automated sample preparation for solid and liquid samples with just one modular sample changer
Applications

CIC is ideal for routine analysis in a variety of fields, as it requires neither specific prior knowledge about the sample matrix nor complicated method development. Not only is CIC recommended for quality control of raw materials, intermediates and finished products; the method is also suitable for easy and exact monitoring of compliance with the relevant laws, standards and regulatory requirements in the environmental field (e.g. DIN EN 228, IEC 60502-1, RoHS, WEEE, ...).

Examples of areas and products where ion chromatographic analyses are possible with CIC are:

- environmentally relevant substances
  (oil, plastic waste, glass, activated carbon, ...)
- electronic components
  (printed circuit boards, resin, cables, insulation, ...)
- fuels
  (gasoline, kerosene, crude oil, heating oil, coal, catalysts, ...)
- plastics
  (polymers such as polyethylene, polypropylene, ...)
- coloring agents
  (pigments, paints, ...)
- pharmaceutical products
  (raw substances, intermediates, finished products, ...)
- foods
  (oils, spices, flavorings and fragrances, ...)

Determination of halogens and sulfur in certified polyethylene pellets ERM-EC681k: chloride: 102.4%, bromide 95.4%, sulfur 100.3%. Injection volume 20 µL

Determination of halogens and sulfur in coal reference material NIST 2682b: chloride: 103.4%, sulfur 96.8%. Injection volume 100 µL

Conditions (both analyses): Metrosep A Supp 5 - 150/4.0; eluent: 3.2 mmol/L Na$_2$CO$_3$, 1.0 mmol/L NaHCO$_3$, 0.7 mL/min; column temperature 30 °C; oven temperature 1050 °C; absorption solution: 100 mg/L H$_2$O$_2$.

Metrohm CIC complies with the following standards

**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)

**UOP991-11**  
Chloride, Fluoride, and Bromide in Liquid Organics by Combustion Ion Chromatography (CIC)

**ASTM D5987-96(2007)**  
Standard Test Method for Total Fluorine in Coal and Coke by Pyrohydrolytic Extraction and Ion Selective Electrode or Ion Chromatograph Methods
Combustion digestion controlled automatically

Metrohm's new CIC consists of a Combustion Module from Analytik Jena and an absorption and IC part from Metrohm. In the Combustion Module the sample digestion is controlled automatically. The principle is simple. An optical fiber carries the light emitted during combustion from the pyrolysis oven to an optical sensor. The optical sensor measures the intensity of the light and controls the feed rate of the sample boat into the oven in proportion to it. As a result, the duration of the combustion is optimized so that, firstly, combustion is always complete (no soot formation), and, secondly, there is no need for waiting times as a safety buffer.

Thanks to the automated control of sample digestion, method development for combustion is not needed both for different samples and different sample quantities can be treated with using the same universal «method».

Sample changer

The MMS 5000 autosampler ensures fully automatic delivery of solid and liquid samples. With the appropriate kit – for liquid or solid samples, respectively – this multi-matrix sampler can be converted in minutes without much effort. The combustion system is unaffected and can therefore be brought back into operation within a few minutes.
In the Metrohm CIC System the Combustion Module and ion chromatograph are linked together by the 920 Absorber Module. The 920 Absorber Module ensures that the gaseous compounds of the analytes are brought into solution. The professional liquid handling also includes the input of water for combustion, matrix elimination of the hydrogen peroxide (oxidizing agent), and rinsing procedures.

Furthermore, an automatic calibration of the analytical system can be obtained from a single multi-ion standard using the Metrohm intelligent Partial Loop Injection Technique (MiPT). Because MiPT allows flexible injection volumes (4 - 200 µL), a large concentration range can be covered.

The following are available for the entire liquid handling: a 10-port valve, a 6-port injection valve and two patented Metrohm 800 Dosino dosing units.

The 920 Absorber Module can also be used as a semi-online sampler for direct absorption of gas compounds (NH₃, HNO₂, HNO₃, HCl, SO₂) from the air. In this way, for example, volatile organic acids (hydrochloric acid, sulfuric acid) can be monitored in process flue gases or in the ambient air at workplaces.
Easy operation with MagIC Net™ software

The complete CIC system is controlled by MagIC Net™, the proven software for ion chromatography. MagIC Net™ offers great flexibility with regard to configuration, layout and programming. Extensive monitoring and control functions are available to the user. MagIC Net™ meets all FDA and GLP requirements and is also available in numerous languages. A modern data management system and a powerful report generator round off the package.

MagIC Net™ guarantees easy, robust and reliable handling of the system. Thanks to an intelligent operating concept, the user is able at all times to maintain an overview of all the liquid quantities that are introduced into the absorption solution. Consequently, it is possible to work without an internal standard or other aids.
Ordering information

Metrohm markets the complete system, including installation, service and training – all from the same supplier.

**Instruments**

2.881.3030 Metrohm Combustion IC

The package contains:

2.881.0030 Compact IC pro – Anion – MCS
2.850.9010 IC Conductivity Detector
2.920.0010 Absorber Module
2.136.0700 Combustion Module
6.6059.241 MagIC Net™ 2.4 Compact
6.1006.310 Metrosep A PCC 1 HC/4.0

**Sample changers**

2.136.0800 Autosampler MMS 5000 (no sampler head/rack)
6.7302.000 MMS 5000 Kit for Solid Samples
6.7303.000 MMS 5000 Kit for Liquid Samples

**Columns**

6.1031.420 Metrosep A Supp 16 - 150/4.0
6.1031.500 Metrosep A Supp 16 Guard/4.0