Combustion Ion Chromatography

Fast and reliable determination of halogens and sulfur after pyrohydrolysis

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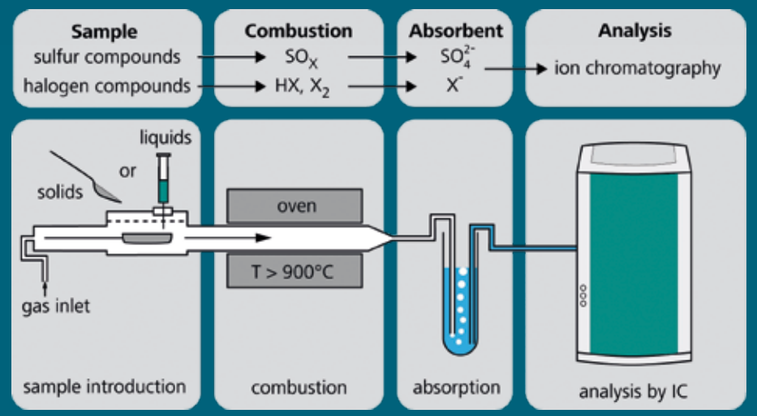
Metrohm

Combustion digestion and ion chromatography combined in one system

Combustion Ion Chromatography (CIC) extends the range of ion chromatography to all types of combustible samples. The focus is primarily on the simultaneous determination of the various halogens and sulfur in a broad range of different matrices. The Metrohm CIC System, including sample preparation, is completely automated. CIC is superior to offline digestion methods regarding sample throughput on the one hand and precision and accuracy of the results on the other. With CIC, unlike with alternative methods, the concentrations of the different halogens can each be determined separately.

How does CIC work?
In CIC the samples are pyrolized with oxygen and water (pyrohydrolysis) in an argon atmosphere. The resulting gaseous compounds are transferred into the 920 Absorber Module where they are collected in an absorption solution. This solution is injected into the IC system for subsequent analysis.

The CIC solution from Metrohm combines a combustion module (oven unit) from Trace Elemental Instruments (TEI) with an absorption module and an IC system from Metrohm. In the combustion module, standard sample matrices such as polymers or gasoline are digested by combustion in a special tube made of quartz glass. More challenging samples that are aggressive towards glass (e.g., ores and matrices with a high fluoride content) are digested in another, dedicated combustion tube made of ceramic.



THE BENEFITS OF CIC AT A GLANCE

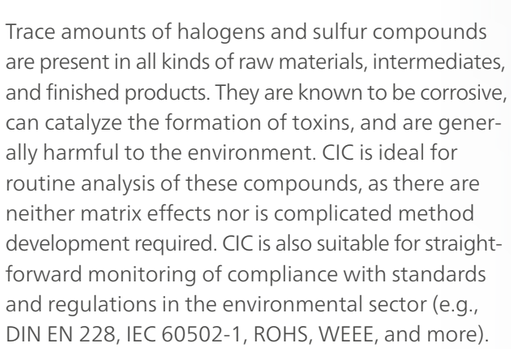
- Extends the range of IC to include all kinds of combustible samples (solid, liquid, gaseous)
- Simultaneous determination of sulfur and halogens
- Quantification of the concentration for each of the different halogens
- Ideal for checking compliance with the latest analytical standards regulating halogen concentrations (RoHS, WEEE, …)
- High sample throughput, precision, and accuracy
- Highly flexible due to dedicated sampling systems for solid, liquid, and gaseous samples
- MagIC Net ion chromatography software for control and data management
- Calibration with just one standard thanks to Metrohm intelligent Partial Loop Injection Technique (MiPT)
- Small footprint
- Choice of quartz or ceramic combustion tube depending on sample matrices

Wide scope of applications

Trace amounts of halogens and sulfur compounds are present in all kinds of raw materials, intermediates, and finished products. They are known to be corrosive, can catalyze the formation of toxins, and are generally harmful to the environment. CIC is ideal for routine analysis of these compounds, as there are neither matrix effects nor is complicated method development required. CIC is also suitable for straightforward monitoring of compliance with standards and regulations in the environmental sector (e.g., DIN EN 228, IEC 60502-1, ROHS, WEEE, and more).

CIC is applied to the following samples:

- 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, butane, propane, natural gas, catalysts, …)
- plastics (polymers such as polyethylene, …)
- coloring agents (pigments, paints, …)
- pharmaceutical 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

METROHM CIC COMPLIES WITH INTERNATIONAL NORMS AND STANDARDS LIKE FOR EXAMPLE …

- **ASTM D7359-14** 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)
- **UOP991-13** Chloride, Fluoride, and Bromide in Liquid Organics by Combustion Ion Chromatography (CIC)
- **ASTM D8247** Standard Test Method for Determination of Total Fluorine and Total Chlorine in Coal by Oxidative Pyrohydrolytic Combustion Followed by Ion Chromatography Detection
- **ASTM D7994-17** Standard Test Method for Total Fluorine, Chlorine, and Sulfur in Liquid Petroleum Gas (LPG) by Oxidative Pyrohydrolytic Combustion Followed by Ion Chromatography Detection (Combustion Ion Chromatography CIC)

CIC automation – sampling of solid, liquid, and gaseous compounds

Depending on the kind and consistency of the sample matrix, different sampling modules for fully automated sample introduction into the Combustion Oven are available. Changing from one sampler to another is fast, straightforward, and simple.

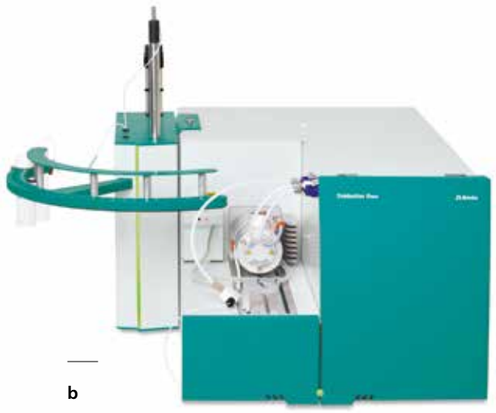
SOLID AUTOSAMPLER CIC

For solid sample matrices. In the standard version, up to 20 samples can be automatically introduced into the oven unit. Sample capacity can be extended with additional trays to up to 60 samples. The samples are introduced into the Combustion Oven in sample cups via the Boat Introduction Module. Depending on the nature of the sample matrix, either quartz cups or ceramic cups (for more aggressive matrices) are used.

LIQUID AUTOSAMPLER CIC

For liquid sample matrices. This autosampler has 30 sample positions plus an additional position for a rinsing beaker. The samples are introduced into the Combustion Oven either in dedicated sample cups with the Boat Introduction Module (a) or injected directly into the Liquid Introduction Module by means of a hollow needle (b). With this Liquid Introduction Module, samples with a boiling point of up to 420°C can be introduced into the Combustion Oven and evaporated at a steady speed. The resulting gaseous compounds are transferred into the Combustion Oven. As no sample cups are used in version (b), there is no risk of incomplete combustion (soot or coke formation) and the reproducibility of results is improved. Multiple injections from the same sample can be digested and absorbed for the same analysis improving the detection limit.

a



b

GLS SAMPLER CIC

For gaseous or LPG samples. The samples are gauged by a valve with sample loop, after which it is introduced at a steady speed into the oven unit using the Liquid Introduction Module. Separate sample flow paths for LPG and gas in the GLS Module eliminate the risk of contamination.

SAFETY FIRST

Safety is paramount when it comes to handling highly flammable commodities such as liquefied gas. The GLS provides several safety features:

- Automatic safety lock on inlet ports – this prevents the sample cylinder from being removed as long as there is pressure detected in the connection.
- Internal gas leakage sensor – all system activity is automatically aborted, and the operator alerted when any hydrocarbon spill is detected.
- Auto-shutoff inlet ports. The auto-shutoff mechanism stops any gas flow due to a dedicated valve when it is disconnected. When both the male coupling and the female plug have a shutoff valve, a double-ended shutoff connection is established.
- Gas or LPG is only consumed from the cylinders during sampling.

Wide scope of applications

COMBUSTION IC SYSTEM

STRAIGHTFORWARD OPERATION
The Metrohm CIC system is controlled by the MagIC Net software. The system is switched off or set to stand-by mode after a determination series. This feature saves costs, e.g., when analyzing expensive gases.

STRAIGHTFORWARD CALIBRATION
Thanks to the Metrohm intelligent Partial Loop Injection Technique (MiPT), the system is calibrated fully automatically using a single multi-ion standard. This saves time and helps prevent errors.

RELIABLE
CIC is superior to offline digestion methods not only regarding sample throughput but also in terms of precision and accuracy of results. Check standards for calibration and reference materials for the samples also increase reliability.

NO INTERNAL STANDARDS REQUIRED
The MagIC Net software documents all liquids that are dosed to the absorption solution. Hence, there is no need for an internal standard. This prevents any unwanted dilution effects and any interference between the internal standard and the analytes in the chromatogram.

Liquid Autosampler CIC, Solid Autosampler CIC, Combustion Oven (TEI)

920 Absorber Module

930 Compact IC Flex

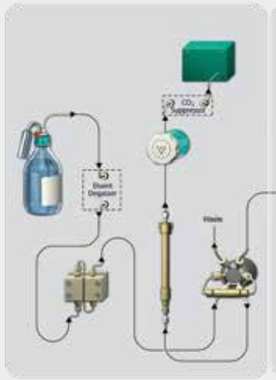
GLS Sampler CIC

Perfect liquid handling – the 920 Absorber Module

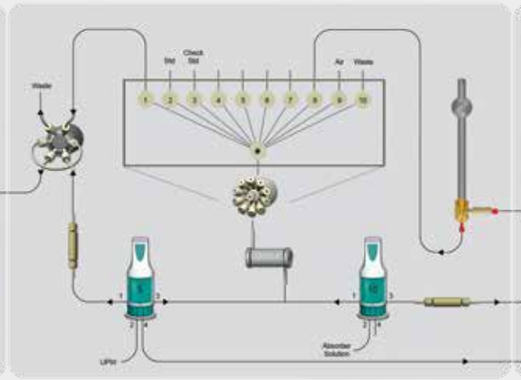
In the Metrohm CIC System the combustion module and ion chromatograph are linked together by the 920 Absorber Module. The 920 Absorber Module ensures that the gaseous compounds of the analytes are brought into solution. The professional liquid handling also includes the input of water for combustion, matrix elimination of the hydrogen peroxide (oxidizing agent), and rinsing procedures.

Moreover, a single multi-ion standard may be used for an automated calibration of the system by means of the Metrohm intelligent Partial Loop Injection Technique (MiPT). Because MiPT allows flexible injection volumes (4–200 µL), a large concentration range can be covered.

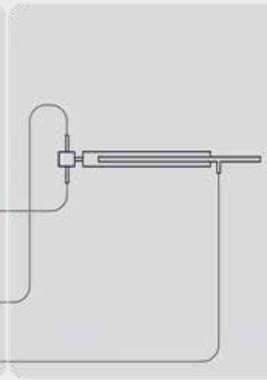
The following items are available for the entire liquid handling: a 10-port valve, a 6-port injection valve and two patented Metrohm 800 Dosinos with Dosing Units. The 920 Absorber Module can also be used as a semi-online sampler for direct absorption of gas compounds (NH_3, HNO_2, HNO_3, HCl, SO_2) 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.



930 Compact IC Flex



920 Absorber Module



Combustion Module

Straightforward control with MagIC Net software

The CIC system from Metrohm is controlled by MagIC Net software for ion chromatography while the sampling modules and the oven unit are controlled by the TEIS Software. Dedicated monitoring and control functions are available to the user. MagIC Net meets all FDA and GLP requirements and is available in numerous languages. A modern data management system and a powerful report generator round off the package.

MagIC Net guarantees simple, robust, and reliable management of the system. The user is always able to keep track of all samples that are introduced into the absorption solution. Due to this it is possible to work without an internal standard or other tools.

ORDERING INFORMATION

Instrument		
Metrohm markets the complete system, including installation, service and training – all from the same supplier.		
Combustion IC sets		
2.930.9030	**Metrohm Combustion IC Manual – Quartz**	
	2.0136.0600	Combustion Oven (TEI)
	6.07311.010	Boat Introduction Module, quartz
	6.07311.100	Quartz combustion tube
	2.930.2560	930 Compact IC Flex Oven/SeS/PP/Deg
	2.850.9010	IC Conductivity Detector
	2.920.0010	920 Absorber Module
	6.6059.401	MagIC Net 4.0 Compact CD: 1 license
	6.2832.000	MSM A Rotor
	6.2842.020	Adapter sleeve f. Suppressor
	6.1006.340	Metrosep A PCC 2 HC/4.0
2.930.9040	**Metrohm Combustion IC Manual – Ceramic**	
	2.0136.0600	Combustion Oven (TEI)
	6.07311.020	Boat Introduction Module, ceramic
	6.07311.110	Ceramic combustion tube
	2.930.2560	930 Compact IC Flex Oven/SeS/PP/Deg
	2.850.9010	IC Conductivity Detector
	2.920.0010	920 Absorber Module
	6.6059.401	MagIC Net 4.0 Compact CD: 1 license
	6.2832.000	MSM A Rotor
	6.2842.020	Adapter sleeve f. Suppressor
	6.1006.340	Metrosep A PCC 2 HC/4.0
Introduction Modules		
6.07311.010	Boat Introduction Module, quartz	
6.07311.020	Boat Introduction Module, ceramic	
6.07311.030	Liquid Introduction Module, manual	
6.07311.040	Liquid Introduction Module, auto.	
Autosamplers		
2.0136.0610	Liquid Autosampler CIC (TEI)	
2.0136.0620	Solid Autosampler CIC (TEI)	
2.0136.0630	GLS Sampler CIC (TEI)	