

Combustion Ion Chromatography



Fast and reliable determination of halogens and sulfur

Combustion digestion and ion chromatography combined in one system

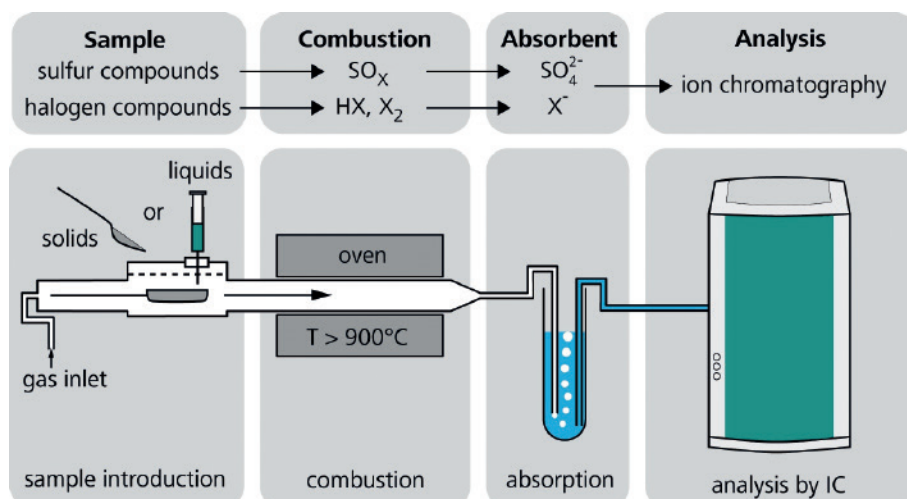
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Combustion Ion Chromatography (CIC) extends the range of ion chromatography to all types of combustible samples. The focus here is primarily on the simultaneous determination of the various halogens and sulfur in widely differing matrices.

The Metrohm 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.



Schematic representation of a complete Combustion IC system

Configuration with an 930 Compact IC Flex Oven/SeS/PP/Deg, a 920 Absorber Module and a Combustion Module from Analytik Jena equipped with solid autosampler



The advantages of CIC at a glance

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- Extended range of application of IC for all kinds of combustible samples (solid, liquid, gaseous)
- 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, 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
- Compliance with FDA and GLP standards
- Calibration with just one standard thanks to Metrohm intelligent Partial Loop Injection Technique (MiPT)
- Fully automated sample preparation for solid and liquid samples with just one modular sample changer

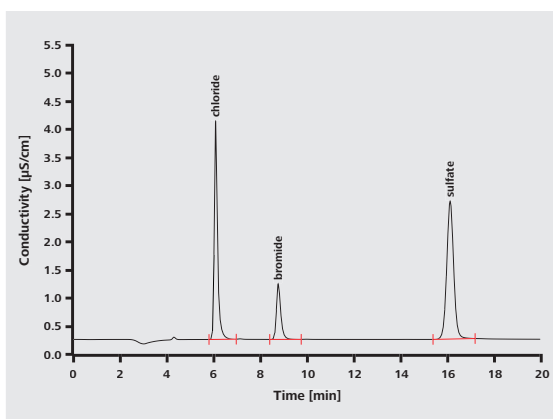


Applications

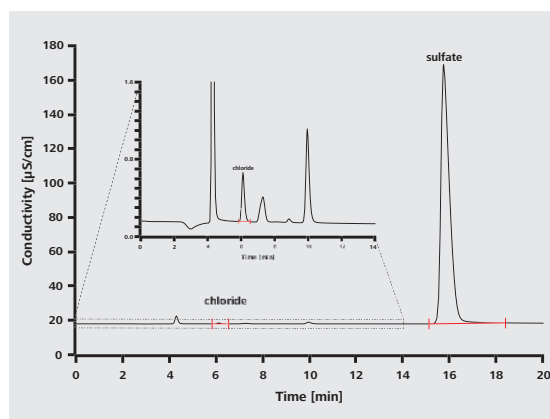
CIC is ideal for routine analysis in a variety of fields, as neither details about the nature of the sample matrix need to be known nor complicated method development is necessary. Not only is CIC recommended for quality control of raw materials, intermediates and finished products; the method is also suitable for straightforward 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 analyses are possible with CIC:

- 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, 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

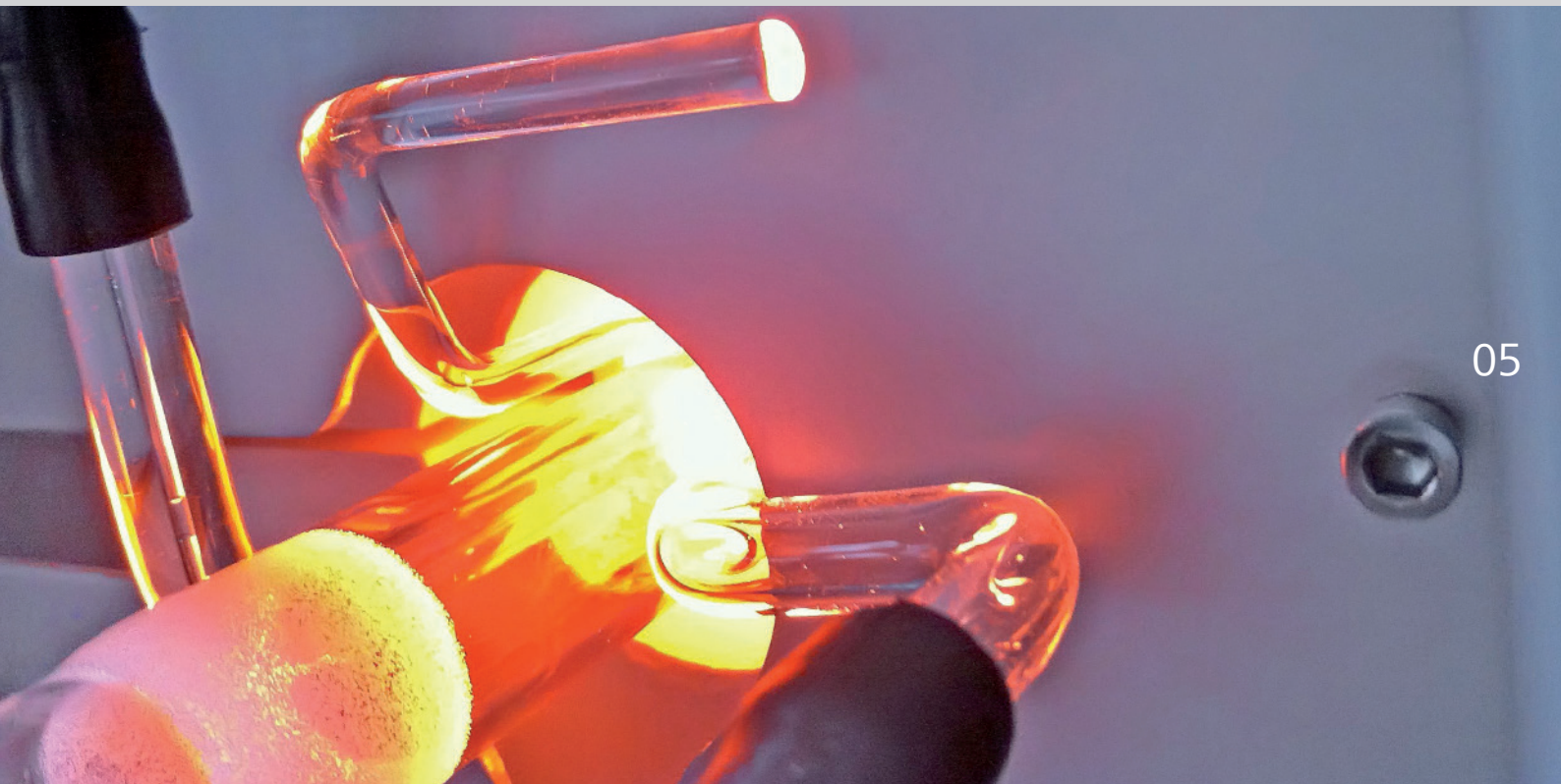


Determination of halogens and sulfur in coal reference material NIST 2682b: chloride: 103.4%, sulfur 96.8%. Injection volume 100 µL

Conditions (both analyses): Metrosep A Supp 5 - 150/4.0; eluent: 3.2 mmol/L Na_2CO_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_2O_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
ASTM WK 24757	Work item for F, Cl, S in LPG



A flame sensor measures the intensity of light in the Combustion Module and controls the combustion digestion automatically.

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 com-

bustion 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. Different samples as well as different sample quantities can be subjected to one and the same universal «method».

Sample changer

The MMS 5000 autosampler ensures fully automatic introduction of solid and liquid samples. With the appropriate kit for liquid or solid samples, respectively this multi-matrix sampler can be converted in minutes with-

out much effort. The combustion system is unaffected and can therefore be brought back into operation within a few minutes.



CIC automation. Liquid or solid samples – the MMS 5000 autosampler can work with either.

Reliable operation under high pressure: LPG/GSS Gas Module

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The LPG/GSS Gas Module makes it possible to analyze both liquefied gases (LPG) and gaseous samples (GSS) – such as natural gas – using Combustion IC. The dosing volume can be selected freely, something which even allows calibration of a wide concentration range without the need for an additional calibration gas standard. Moreover the separate flow paths for LPG and GSS also eliminate any risk of contamination.

MagIC Net controls the Gas Module fully automatically. A special dosing valve that uses Peltier cooling is used for dosing liquefied gases, which prevents the sample from being released prematurely. Following the sampling process, the liquefied gas is vaporized in a heated chamber. This enables even components that are less easily volatilized to be brought into a gaseous state. The sample is fully transferred to the combustion system, a process that is also supported by a permanent flow of argon rinsing gas. This prevents memory effects.

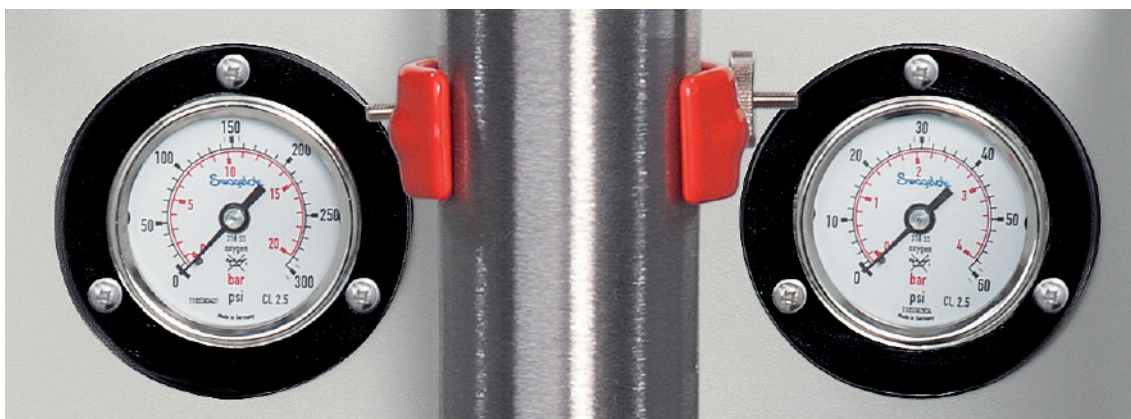


LPG/GSS Gas Module for analyzing gases in liquefied and gaseous states.



Key parameters in gas analysis using Combustion IC

	LPG	GSS
Sample cylinder pressure	Max. 18 bar	Max. 250 bar
Pressure at sample valve	Determined by sample cylinder	0.7–1.7 bar, typically at 1 bar
Sample volume	1–50 μ L in 1- μ L steps	1–20 mL in 250- μ L steps
Temperature of flash chamber	85 $^{\circ}$ C	–



CIC in the overview

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Simple operation

Once the sample table has been created, the Combustion IC system operates entirely automatically. All parameters are controlled by MagIC Net software. The system is automatically switched off after a determination series, which saves costs in cases where expensive gases are being used, for example.

Highly flexible

The MMS 5000 sample changer makes the system highly flexible. The sample changer can be refitted to handle liquid samples instead of solid ones within two minutes – meaning that just one sample changer is required. What's more, there is no need to readjust the hardware such as the gas line when changing between different types of sample, saving valuable time.



Straightforward calibration

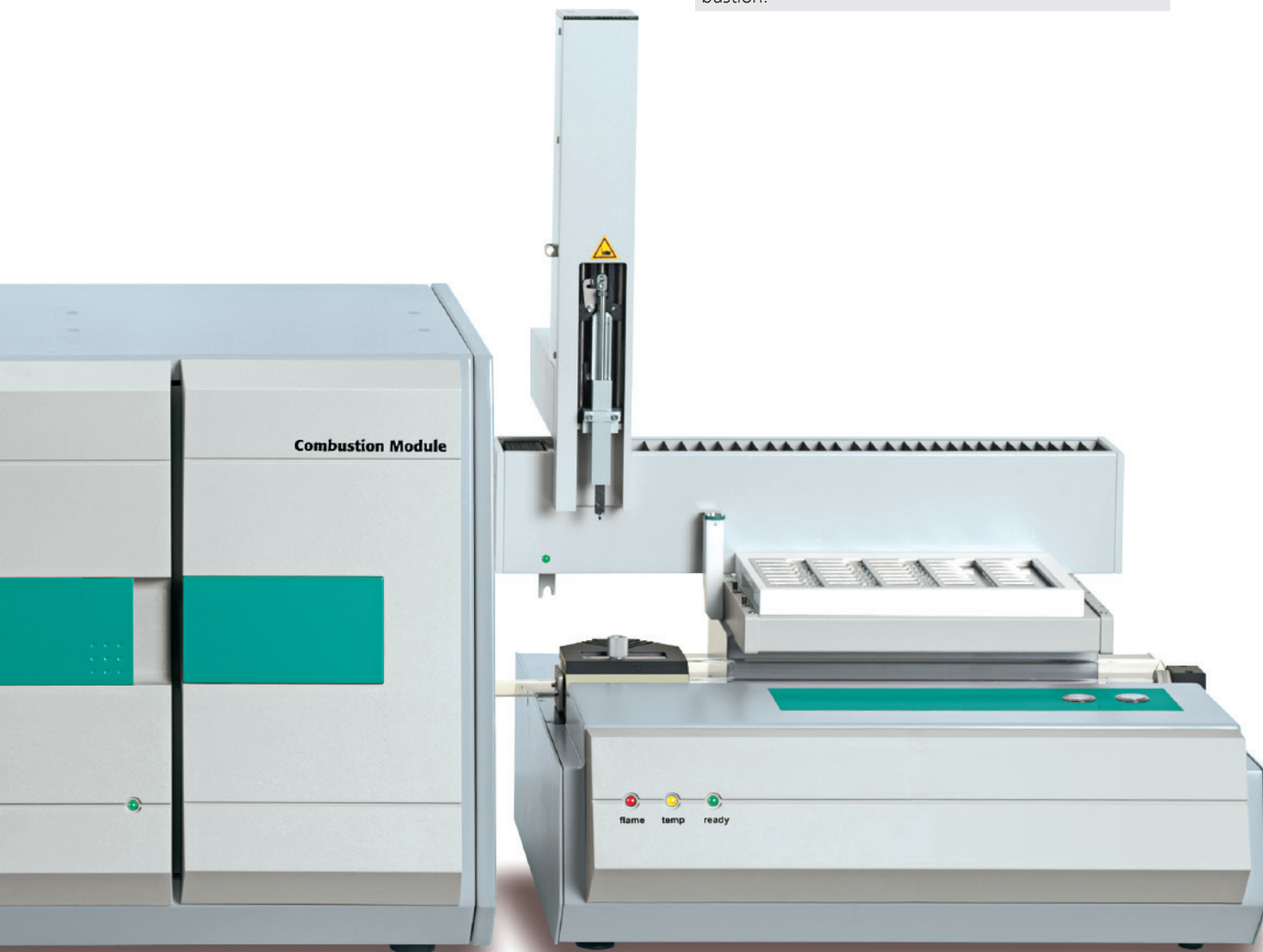
Thanks to the Metrohm intelligent Partial Loop Injection Technique (MiPT), calibration can be performed rapidly, reliably, and – above all – automatically, using a single multi-ion standard. This saves time and is less prone to error.

Reliable

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

Flame sensor technology

Whether solid or liquid samples are analyzed, or the amount of sample varies, the flame sensor optimizes the combustion process. This ensures fast, soot-free combustion.

**No internal standard required**

MagIC Net documents all liquids that are dosed to the absorption solution. As a result, there is no need for an internal standard in the absorption solution. This prevents any undesirable dilution effects and also any interference between the internal standard and the analytes in the chromatogram.

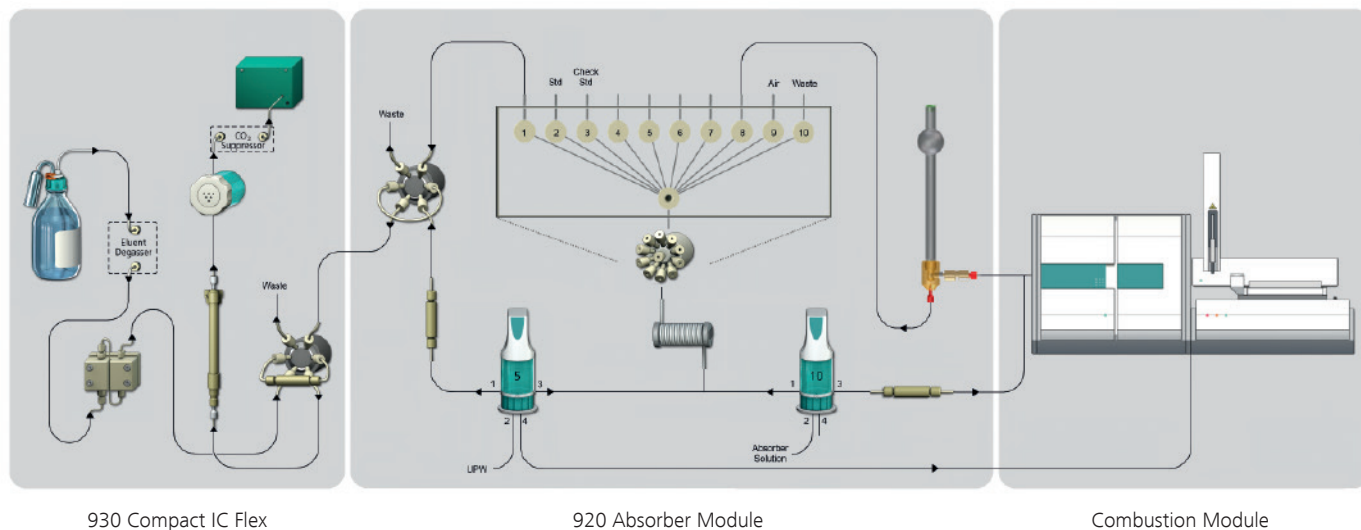
Perfect liquid handling – the 920 Absorber Module

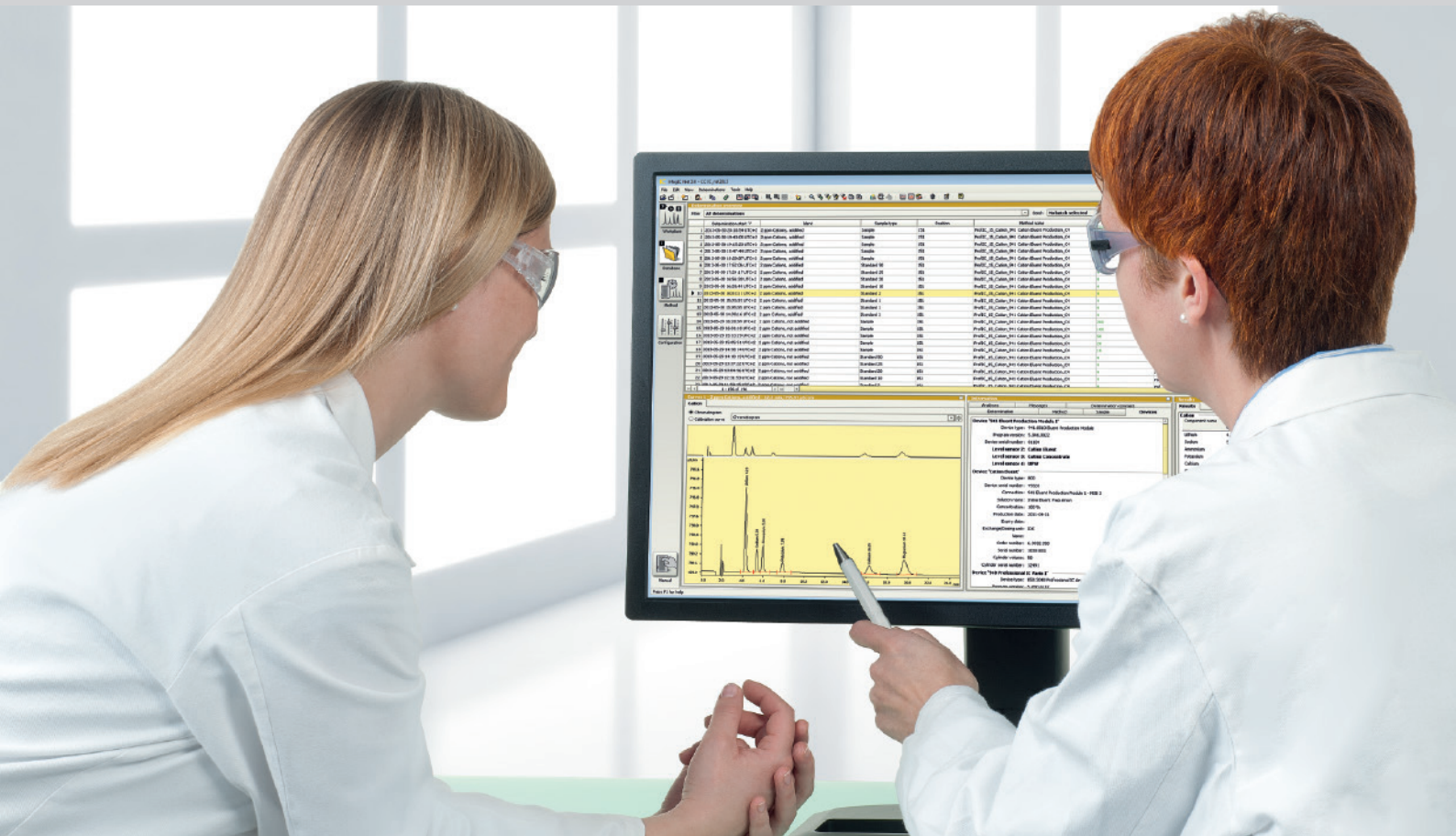
In the Metrohm CIC System the Combustion Module and ion chromatograph are linked together by the 920 Absorber Module. The 920 Absorber Module ensures that the gaseous compounds of the analytes are brought into solution. The professional liquid handling also includes the input of water for combustion, 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 Dosino 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.





Straightforward control 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 method programming. Several 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. The user is able at all times to keep 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 tools.

Ordering information

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

Instruments

2.930.9010 Metrohm Combustion IC

The package contains

2.930.2560 Compact IC Flex Oven/SeS/PP/Deg
2.850.9010 IC Conductivity Detector
2.920.0010 Absorber Module
2.136.0700 Combustion Module (Oven and ABD)
6.6059.301 MagIC Net 3.0 Compact
6.1006.310 Metrosep A PCC 1 HC/4.0

2.136.0700 Combustion Module (Oven and ABD)
2.136.0730 Combustion Module (Oven and LPG/GSS, including direct coupling 6.7304.030 to oven)
2.136.0710 Auto Boat Drive (ABD)
2.136.0720 LPG/GSS Module (including coupling to ABD)
6.7304.030 Direct coupling of LPG/GSS Module to oven

Sample changers

2.136.0800 Autosampler MMS 5000 (sample rack not included)
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



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