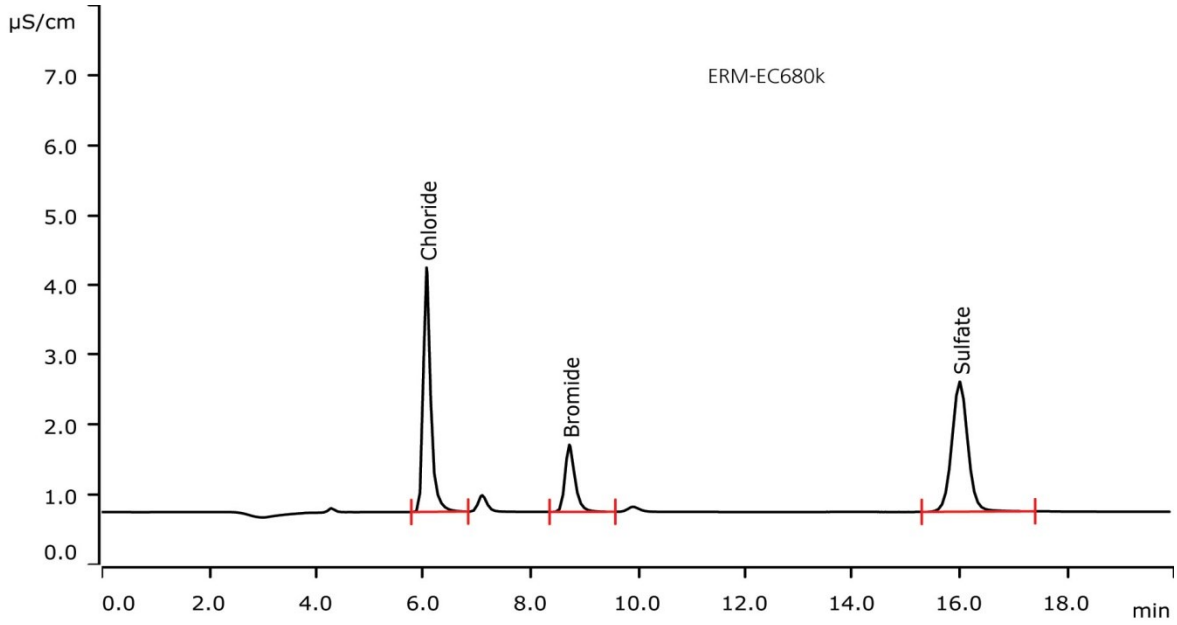
IC Application Note CIC–6

Recovery of chloride, bromide, and sulfate in certified reference materials using Metrohm Combustion IC



Combustion IC combines pyrohydrolytic sample combustion and absorption of the evolving combustion gases in an oxidizing aqueous solution that is then fed into an ion chromatograph for analysis of halides and sulfur (as sulfate). The combustion and analysis of certified reference materials (CRMs) proves the reliability of the Metrohm Combustion IC.

Results

	Mean [mg/kg] ERM-EC680k	RSD [%]	Recovery [%]	Mean [mg/kg] ERM-EC681k	RSD [%]	Recovery [%]
Chloride	103.2	2.1	101.0	783.0	1.0	97.9
Bromide	98.9	1.6	103.0	763.3	1.8	99.1
Sulfate	77.9	2.2	102.5	636.6	1.5	101.1

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Sample

ERM-EC681k, ERM-EC680k (polyethylene pellets)

Sample preparation

Combustion with flame sensor technology, intelligent Partial-Loop Injection (MiPT) with Inline Matrix Elimination

Columns

Metrosep A Supp 5 - 150/4.0	6.1006.520
Metrosep A Supp 4/5 Guard/4.0	6.1006.500
Metrosep A PCC 1 HC/4.0	6.1006.310

Solutions

Eluent	3.2 mmol/L sodium carbonate 1.0 mmol/L sodium hydrogen carbonate
Suppressor regenerant	100 mmol/L sulfuric acid
Rinsing solution	Ultrapure water
Absorption solution	100 mg/L hydrogen peroxide

Analyses

Conductivity after sequential suppression

Parameters

Flow rate	0.7 mL/min
Injection volume	4…100 µL
P_{max}	15 MPa
Recording time	20 min
Column temperature	30 °C

Combustion parameters

Argon	100 mL/min
Oxygen	300 mL/min
Oven Temperature	1050 °C
Post-combustion time	120 s
Initial volume of absorption solution	2.0 mL
Water inlet	0.1…0.2 mL/min

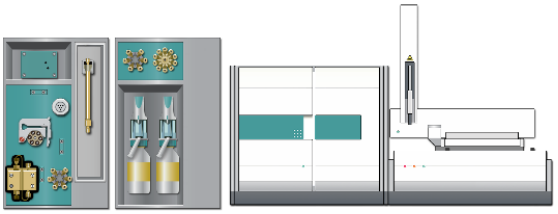
Instrumentation

881 Compact IC pro – Anion – MCS	2.881.0030*
IC Conductivity Detector	2.850.9010*
920 Absorber Module	2.920.0010*
Combustion Module	2.136.0700*
Autosampler MMS 5000	2.136.0800
Kit for solid samples	6.7302.000

* available as 881 Metrohm Combustion IC (2.881.3030)

Calibration MiPT

Calibration range	Factor of 10
Standard solution	
Chloride, bromide	20 mg/L
Sulfate	40 mg/L
1. Level	2/ 4 mg/L = 4 µL
2. Level	6/12 mg/L = 12 µL
3. Level	10/20 mg/L = 20 µL
4. Level	20/40 mg/L = 40 µL



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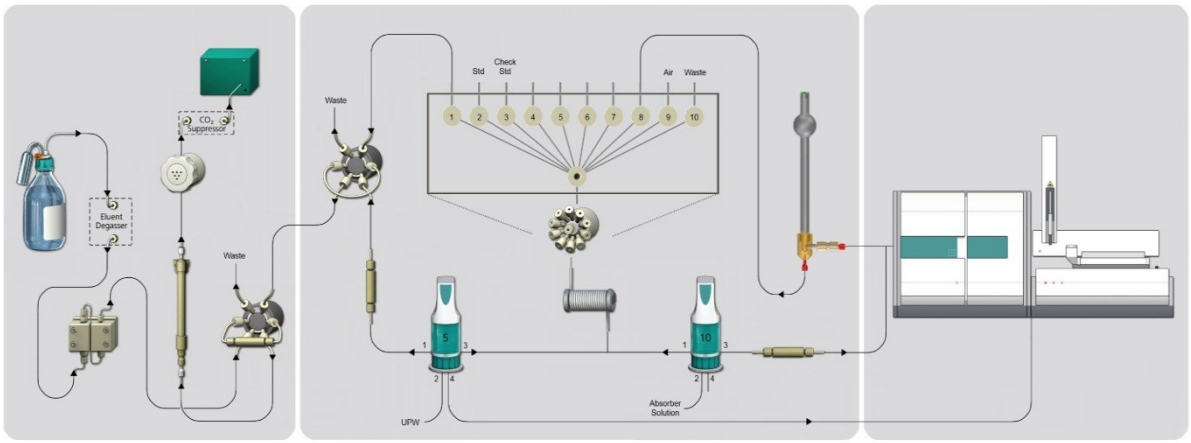
Flow chart



The transfer of samples and standards into the loop of the 920 Absorber Module is performed by the 5 mL Dosino using Metrohm intelligent Partial Loop Injection Technique (MiPT). The injection volumes are variable and range between 4 and 200 µL. After Inline Matrix Elimination of excess H_2O_2, the sample is injected. No internal standard is required, as MagIC Net™ considers all volumes that have been dosed into the combustion and absorption part.

The Metrohm Combustion IC system also copes with liquid samples. To this end, only a few parts (6.7303.000 Kit for liquid samples) have to be exchanged.