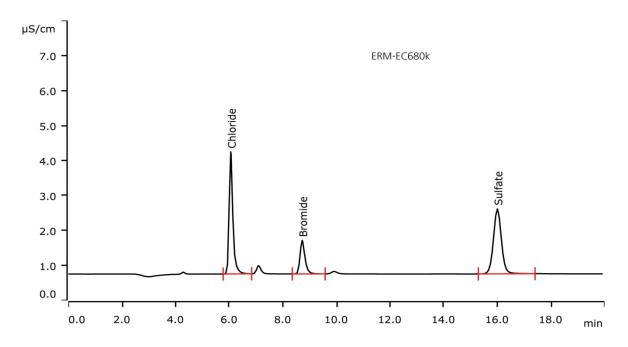
IC Application Note CIC-006

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



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 2 HC/4.0	6.1006.340

Instrumentation

930 Compact IC Flex Oven/SeS/PP/Deg	2.930.2560*
IC Conductivity Detector	2.850.9010*
MSM Rotor A	6.2832.000*
Adapter sleeve for Suppressor Vario	6.2842.020*
920 Absorber Module	2.920.0010*
Combustion Module (oven and ABD)	2.136.0700*
941 Eluent Production Module	2.941.0010
Autosampler MMS 5000	2.136.0800
Kit for solid samples	6.7302.000

^{*} available as 930 Metrohm Combustion IC (2.930.9010)

Solutions

Eluent (automatically prepared from eluent concentrates)	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

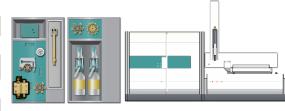
Conductivity after sequential suppression

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

Parameters

Flow rate	0.7 mL/min
Injection volume	4100 μL
P _{max}	15 MPa
Recording time	20 min
Column temperature	30 °C



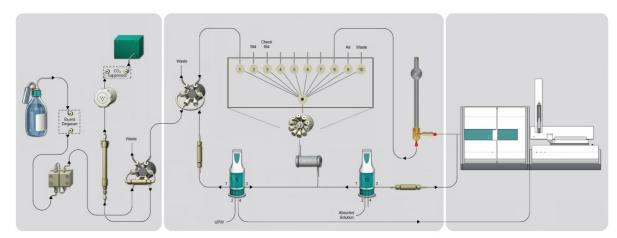
Combustion parameters

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	0.10.2 mL/min

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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 μ L. After Inline Matrix Elimination of excess H₂O₂, 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.

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