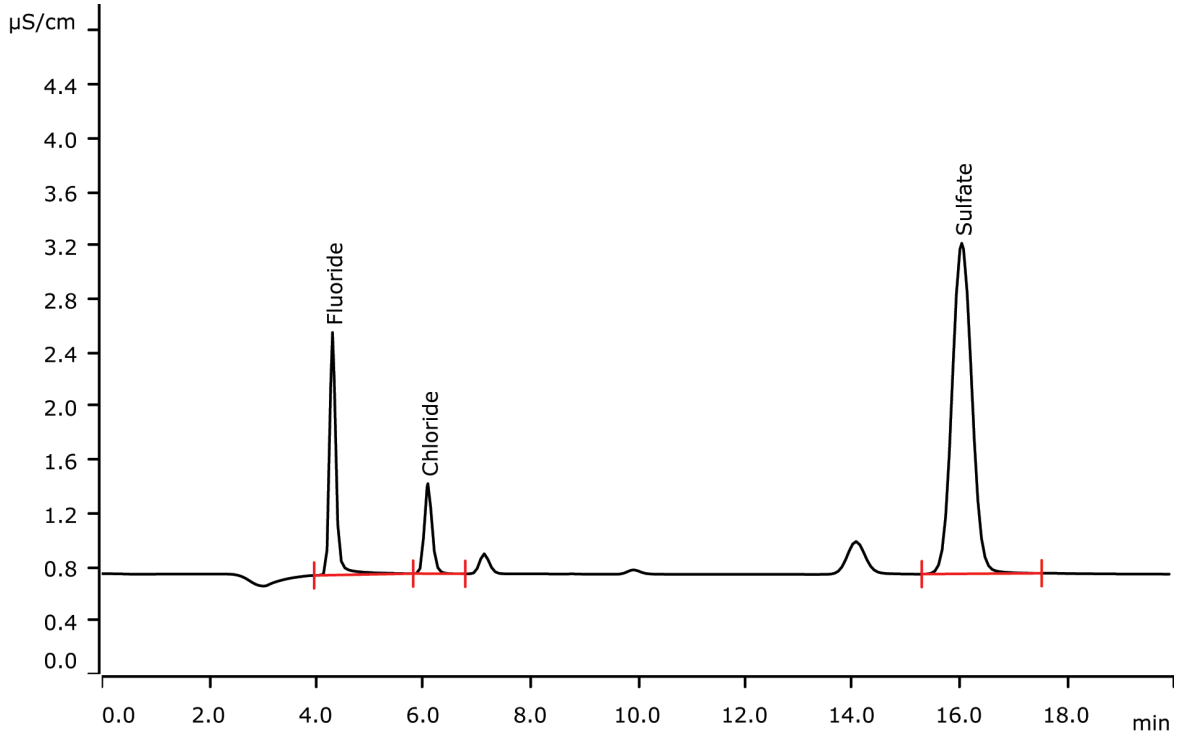
IC Application Note CIC–012

Halogens and sulfur in clay using Metrohm Combustion IC



Clay is used for tile production. Quality control issues require the determination of its halogen and sulfur content. This is ideally done by Metrohm Combustion IC, where sulfur is detected as sulfate and the halogens as their corresponding halides. As clay often has high contents of alkali and alkaline earth metals that attack the pyrolysis tube, tungsten oxide is added before combustion.

Results

	Mean [mg/kg] (n = 3)	RSD [%] (n = 3)
Fluoride	43.7	3.1
Chloride	32.7	3.1
Sulfur	145.8	4.6

Metrohm

Sample

Clay

Sample preparation

Ground sample mixed with 100 mg tungsten oxide was analyzed by Combustion IC with flame sensor technology and Metrohm intelligent Partial-Loop Injection 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	Detector outlet
Absorber solution	100 mg/L hydrogen peroxide

Parameters

Flow rate	0.7 mL/min
Injection volume	60 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	240 s
Initial volume of absorption solution	2.0 mL
Water inlet	0.1 mL/min

Analysis

Conductivity after sequential suppression

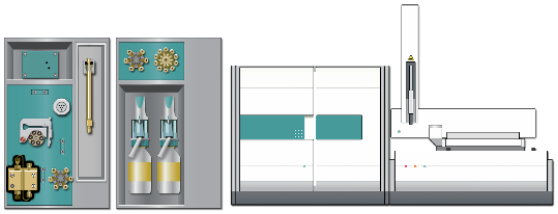
Instrumentation

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 50			
Standard solution				
Fluoride, chloride	35.0 mg/L			
Sulfate	225.0 mg/L			
1. Level	0.7 / 4.5	mg /L	=	4 µL
2. Level	1.4 / 9.0	mg /L	=	8 µL
3. Level	3.5 / 22.5	mg /L	=	20 µL
4. Level	7.0 / 45.0	mg /L	=	40 µL
5. Level	17.5 / 112.5	mg /L	=	100 µL
6. Level	35.0 / 225.0	mg /L	=	200 µL



www.metrohm.com

Metrohm