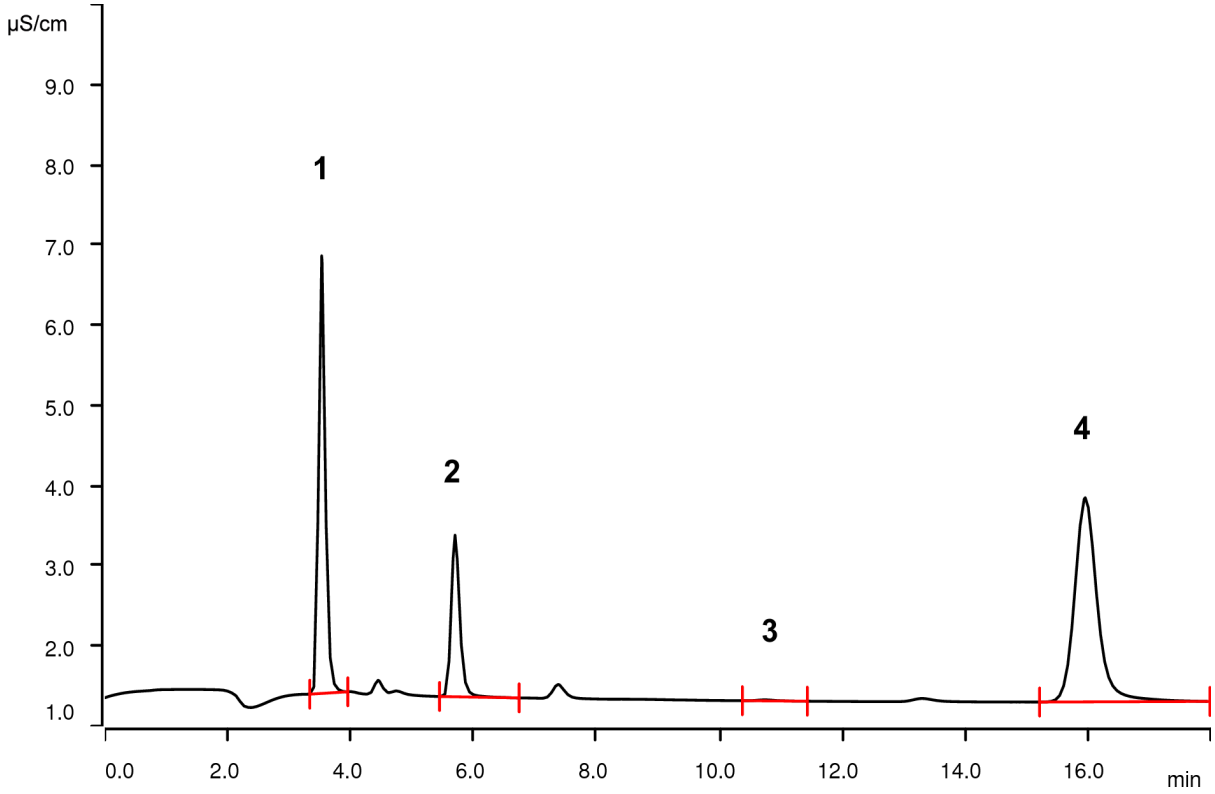
IC Application Note CIC–028

Fluorine and chlorine in iron ore by Combustion Ion Chromatography



Iron ore is an important resource for steel production. Its natural content of halogens is a quality characteristic due to the corrosiveness of the respective halogenides. Combustion IC applying the sacrificial vial technology is used for the analysis of fluorine and chlorine in ore. WO_3 usually is added to improve the release of SO_2 and therefore sulfur recovery. In this application, it also significantly improves the recovery of fluoride.

Results

	Concentration [%] (N = 3)	RSD [%] (N = 3)	Recovery CRM [%] (no WO_3)	Recovery CRM [%] (Sample : WO_3 = 1:1)
1 Fluorine	84.0	1.7	80.0	92.7
2 Chlorine	70.2	1.1	94.7	97.8

Peaks 3 and 4 correspond to bromine and sulfur (not quantified)

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Sample

Iron ore.

Sample preparation

The sample (5 to 15 mg) and in minimum the same amount of WO_3 is placed in a quartz tube with quartz wool and subsequently fixed in the vial with quartz wool. This sacrificing vial is analyzed by Combustion IC with flame sensor technology and intelligent Partial Loop Injection Technique with Inline Matrix Elimination.

Columns

Metrosep A Supp 16 - 150/4.0	6.1031.420
Metrosep A Supp 16 Guard/4.0	6.1031.500
Metrosep A PCC 2 HC/4.0	6.1006.340
Metrosep I Trap 1 - 100/4.0	6.1014.200
Metrosep A Trap 1 - 100/4.0	6.1014.000

Solutions CIC

Eluent	7.5 mmol/L sodium carbonate 0.75 mmol/L sodium hydroxide
Suppressor regenerant	100 mmol/L sulfuric acid
Rinsing solution	STREAM
Absorber solution	100 mg/L H_2O_2

Analysis

Conductivity after sequential suppression

Parameters

Flow rate	0.8 mL/min
Injection volume (IC)	200 µL (MiPT)
P_{max}	20 MPa
Recording time	18 min
Column temperature	45 °C

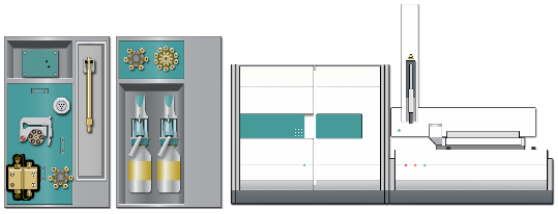
Combustion parameters

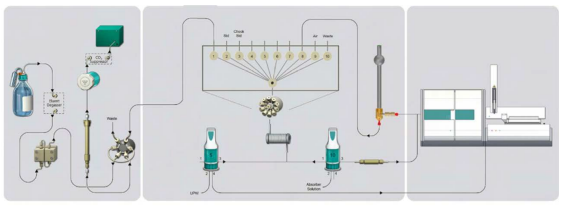
Argon	100 mL/min
Oxygen	300 mL/min
Oven temperature	1100 °C
Post-combustion time	500 s
Initial volume of absorption solution	2.0 mL
Absorber solution feed	0.2 mL/min
Water inlet	0.2 mL/min
Post-combustion rinsing volume	1.0 mL

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*
Autosampler MMS 5000	2.136.0800
Kit for solid sampling	6.7302.000

* available as 930 Metrohm Combustion IC (2.930.9010)





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