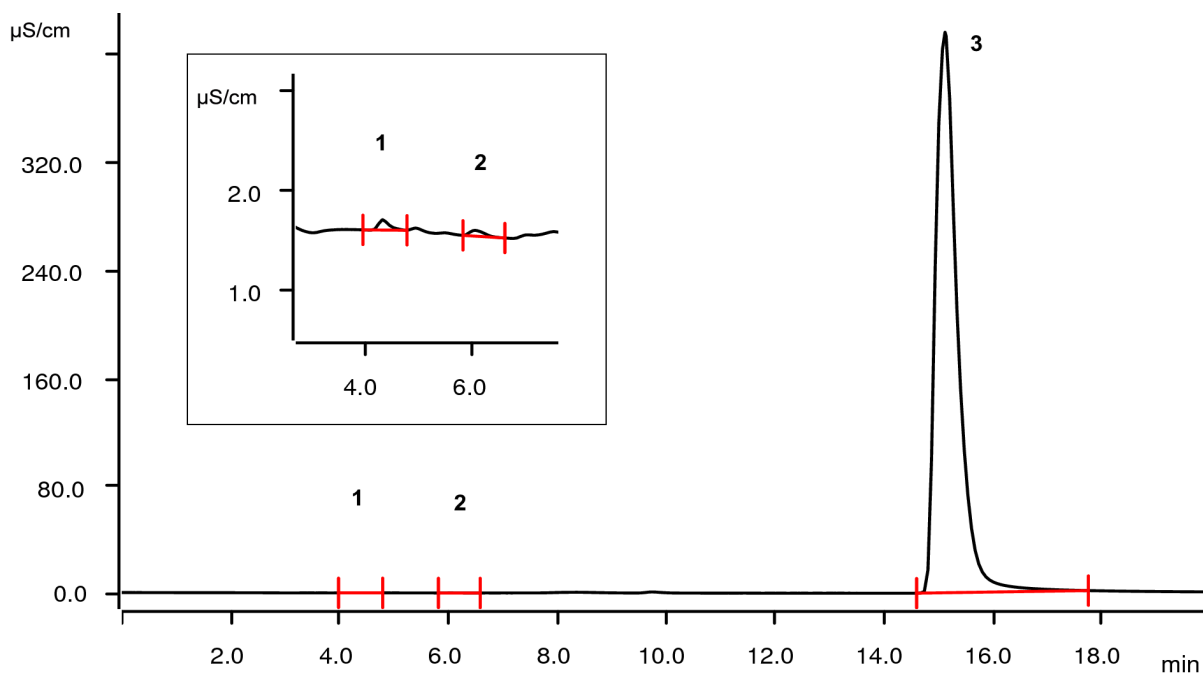


IC Application Note CIC-29

Organic halides in crude oil

Organically bound halogens are released in their acid form by pyrohydrolysis and analyzed by Combustion IC



Crude oil typically contains no organic halides. These are introduced at production sites, in pipelines, or in storage tanks. These components produce HF, HCl, and other acids in reforming and hydro-treating processes, leading to corrosion and catalyst poisoning. Speciation of the halides is an important parameter to measure in order to trace the contamination source. The current specifications expect to find less than 2 mg/kg organic chlorine in crude oil. Sulfur in crude oil could be quantified on the fly. Due to the specific request in this application, only the halogens are determined.

Results

	Concentration [mg/kg] (N = 5)	RSD [%] (N = 5)
1 Fluorine	n.q.	-
2 Chlorine	0.6	7.9
3 Sulfur	n.q.	-

n.q. = not quantified

Sample

Crude oil

Sample preparation

A 100 µL aliquot of crude oil is analyzed by Combustion IC with flame sensor technology and intelligent Partial Loop Injection Technique with Inline Matrix Elimination.

Columns

Metrosep A Supp 5 - 150/4.0	6.1006.520
Metrosep A Supp 5 Guard/4.0	6.1006.500
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	3.2 mmol/L sodium carbonate 1.0 mmol/L sodium hydrogen carbonate
Suppressor regenerant	100 mmol/L sulfuric acid
Rinsing solution	STREAM
Absorber solution	300 mg/L H ₂ O ₂

Analysis

Conductivity after sequential suppression

Parameters

Flow rate	0.7 mL/min
Injection volume (IC)	200 µL (MiPT)
P _{max}	15 MPa
Recording time	20 min
Column temperature	30 °C

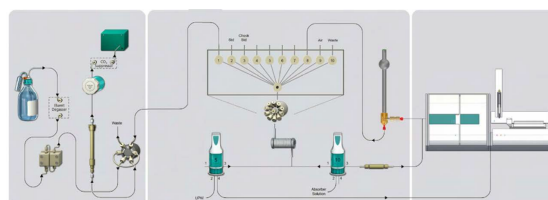
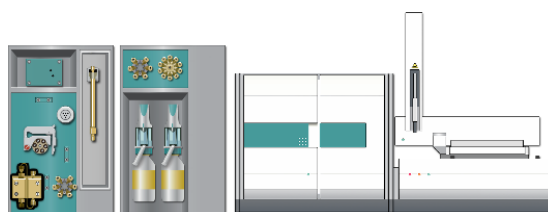
Combustion parameters

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
Absorber solution feed	0.2 mL/min
Water inlet	0.1 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 liquid sampling	6.7303.000

* available as 930 Metrohm Combustion IC (2.930.9010)



www.metrohm.com

 **Metrohm**