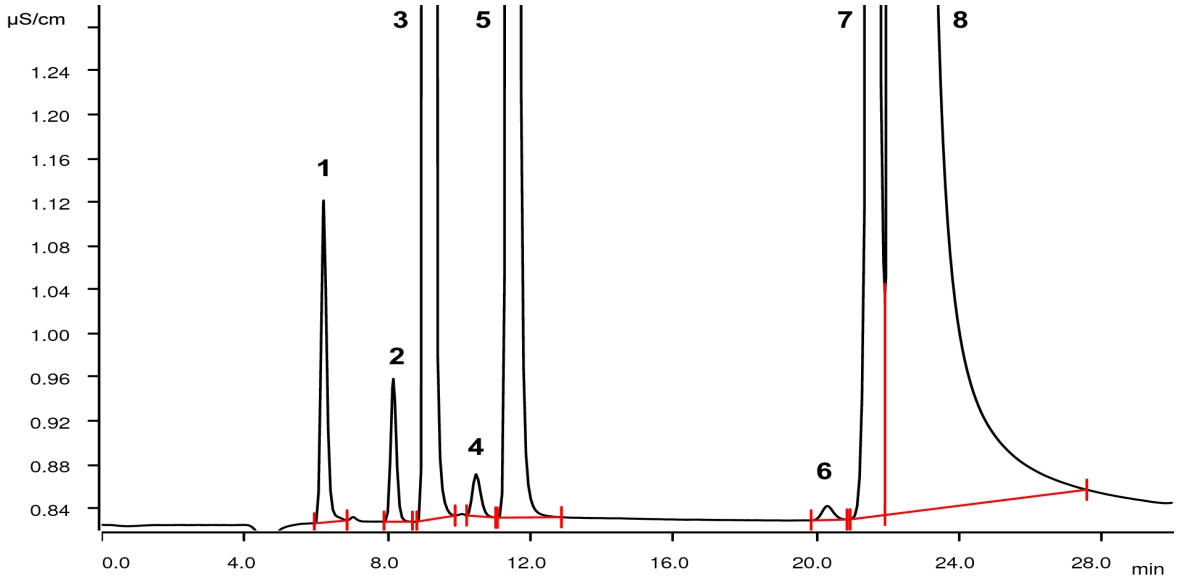
IC Application Note CIC–025

AOF, AOCl, AOBr, AOI, and AOX by Combustion Ion Chromatography



Adsorbable organically bound halogens (AOX) is the sum parameter for organically bound halogens in water. The organic compounds are adsorbed to active carbon and subsequently combusted. Determination of the sum of chloride, bromide, and iodide is traditionally performed by titration.

With Combustion IC, it is possible to determine the individual halogens (AOCl, AOBr, AOI) as well as fluorine (AOF) which cannot be determined by the argentometric titration method.

Results

	Concentration [µg/L] (N = 3)	RSD [%] (N = 3)	Recovery [%]	Blank [µg/L]
1 Fluorine	7.2	3.2	143	2.8
2 Chlorine	5.8	1.0	117	1.2
4 Bromine	5.4	4.9	108	< 1
6 Iodine	4.1	1.4	81.3	< 1

The samples contain 5.0 µg/L of the halogens each, as the respective halobenzoic acid.

Peaks number 3, 5, 7, and 8 correspond to nitrite, nitrate, unknown, and sulfate

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Sample

Water spiked with organic example components baring fluorine, chlorine, bromine, and iodine.

Sample preparation

Sample preparation according to DIN EN ISO 9562 (AOX). 50 mL of the nitrate stock solution is added to 950 mL of sample. 100 mL of this mixture is percolated over two columns (quartz) packed with activated carbon. Automatic absorption is performed on a APU sim (Analytic Jena).

The loaded active carbon is analyzed by Combustion IC with flame sensor technology and intelligent Partial Loop Injection Technique.

Columns

Metrosep A Supp 5 - 250/4.0	6.1006.530
Metrosep A Supp 5 Guard/4.0	6.1006.500

Solutions CIC

Eluent	2.5 mmol/L sodium carbonate 4.0 mmol/L sodium hydroxide
Suppressor regenerant	100 mmol/L sulfuric acid
Rinsing solution	STREAM
Absorber solution	100 mg/L H_2O_2

Sample preparation solutions

Nitrate stock solution	17 g/L sodium nitrate 25 mL nitric acid (65%)
Nitrate rinsing solution	50 mL/L nitrate stock solution

Parameters

Flow rate	0.7 mL/min
Injection volume (IC)	20 µL (MiPT)
P_{max}	15 MPa
Recording time	30 min
Column temperature	55 °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	0.2 mL/min
Post-combustion rinsing volume	1.0 mL

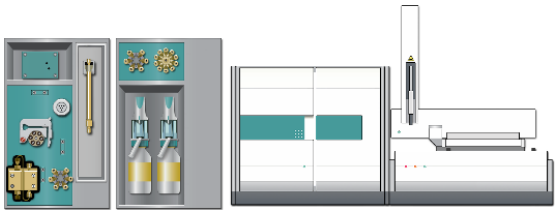
Analysis

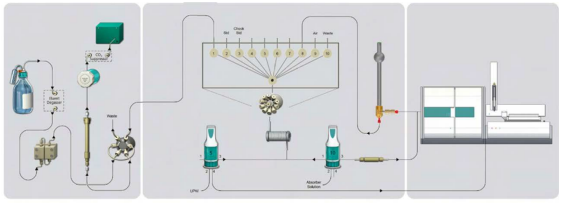
Conductivity after sequential suppression

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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