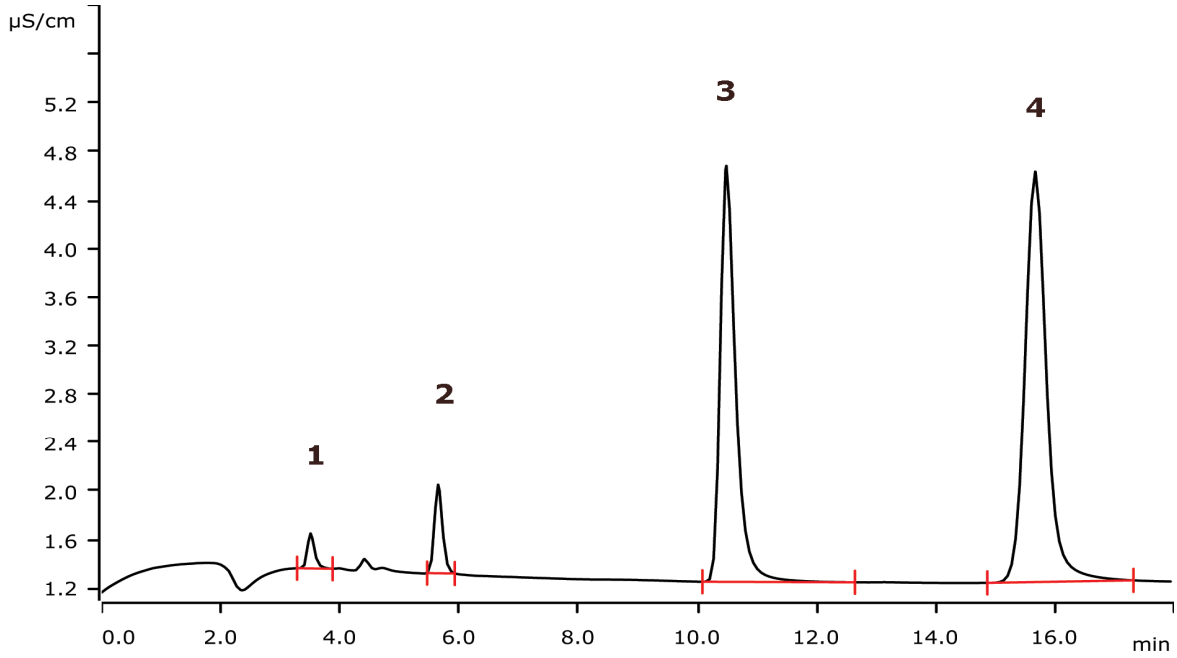
IC Application Note CIC–21

Halogen and sulfur in chlorinated and brominated halobutyl rubber applying Combustion IC



Halobutyl rubber is frequently used in the production of pharmaceutical stoppers. It is ideal for this application due to its low permeability to gases and its chemical resistance. Chlorinated and brominated butyl rubber stoppers are analyzed for their halogen and sulfur content. Halogen and sulfur compounds are released by pyrohydrolysis and analyzed by subsequent ion chromatography (IC).

Results

	Chlorinated butyl rubber		Brominated butyl rubber*)	
	Conc. [mg/kg]	RSD [%]	Conc. [mg/kg]	RSD [%]
1 Fluorine	36	1.2	< 100	-
2 Chlorine	6582	2.6	290	23
3 Bromine	n.d.	-	8595	1.9
4 Sulfur	62	2.9	2509	3.6

*) Chromatogram shown above

Metrohm

Sample

Halogenated butyl rubber

Sample preparation

The sample 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 1 HC/4.0	6.1006.310

Solutions

Eluent	7.5 mmol/L sodium carbonate 0.75 mmol/L sodium hydroxide
Suppressor regenerant	250 mmol/L sulfuric acid
Rinsing solution	STREAM
Absorber solution	100 mg/L hydrogen peroxide

Parameters

Flow rate	0.8 mL/min
Injection volume (IC)	20 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	4.0 mL
Water inlet	0.2 mL/min

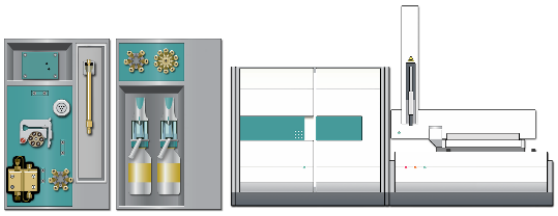
Analysis

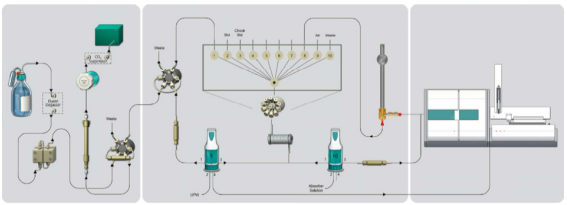
Conductivity after sequential suppression

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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