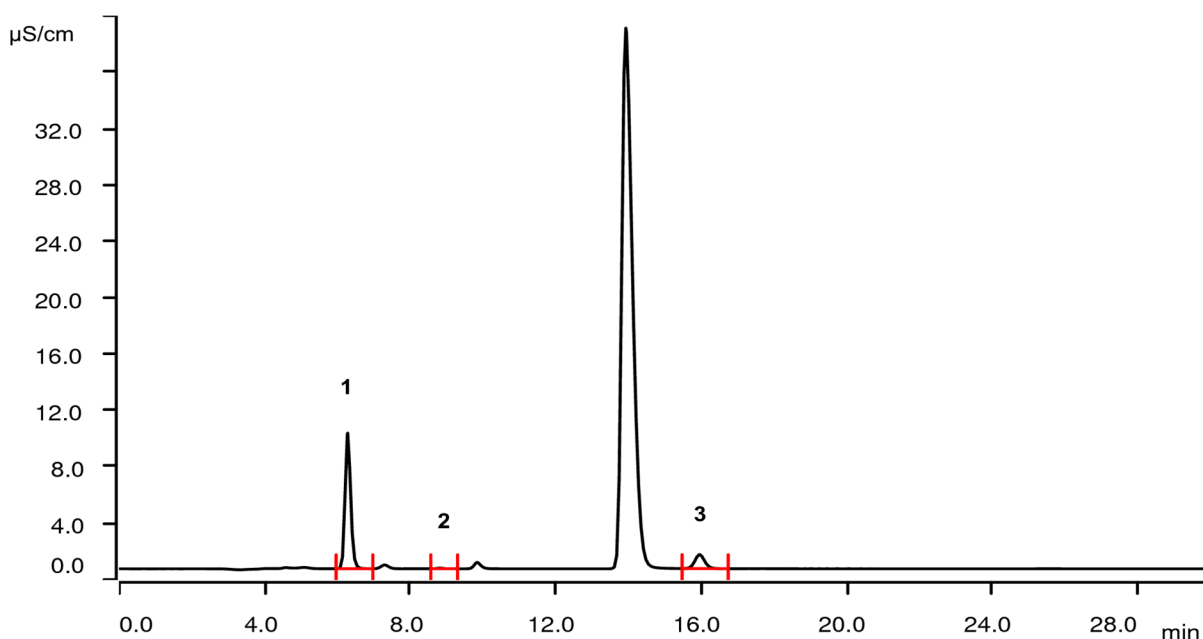


IC Application Note CIC-032

Chloride as an indicator of residual solvent presence in cellulose ester-based foil

Chloride released from cellulose ester foil after pyrohydrolysis is analyzed by Combustion IC, indicating residual solvent levels.



Chromatogram of the absorber solution after pyrohydrolysis of a cellulose ester-based foil.

Cellulose ester foils are produced using chlorinated solvents. The residual amount of the solvent used in production evaporates within a few days in ambient conditions. The residual solvent is determined by combustion IC, through the conversion of organically bound chlorine to chloride by pyrohydrolysis. The final product needs to be free of all chlorinated solvents. Therefore, critical contents of such compounds can be detected in quality control analysis. Application of MiPT in this study has enabled an automated and precise calibration out of a single standard.

Results

Residual solvent over time		Concentration [mg/kg]	
		Day 1	Day 8
1	Chloride	230.4	58.2
2	Bromide	n.q.	n.q.
3	Sulfate	n.q.	n.q.

n.q. = not quantified; the peak at 13.8 min corresponds to phosphate. Under the actual conditions, phosphorus is not completely converted to phosphate and therefore cannot be quantified.

Sample

Cellulose ester-based foil.

Sample preparation

A 30–40 mg sample of the foil 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
Regenerant	100 mmol/L sulfuric acid
Rinsing solution	STREAM
Absorber solution	0.1 g/L hydrogen peroxide

Analysis

Conductivity after sequential suppression

Parameters

Flow rate	0.7 mL/min
Injection volume (IC)	25–200 µL (MiPT)
P _{max}	15 MPa
Recording time	30 min
Column temperature	35 °C

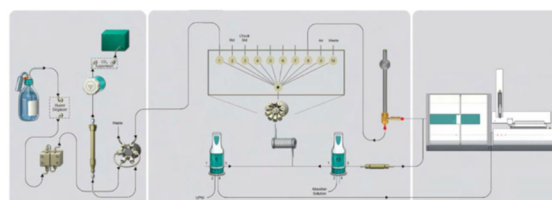
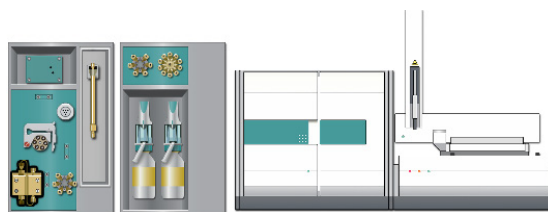
Combustion parameters

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

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
Ceramic boats 40 x 9 mm	6.7302.130

* available as 930 Metrohm Combustion IC (2.930.9010)



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