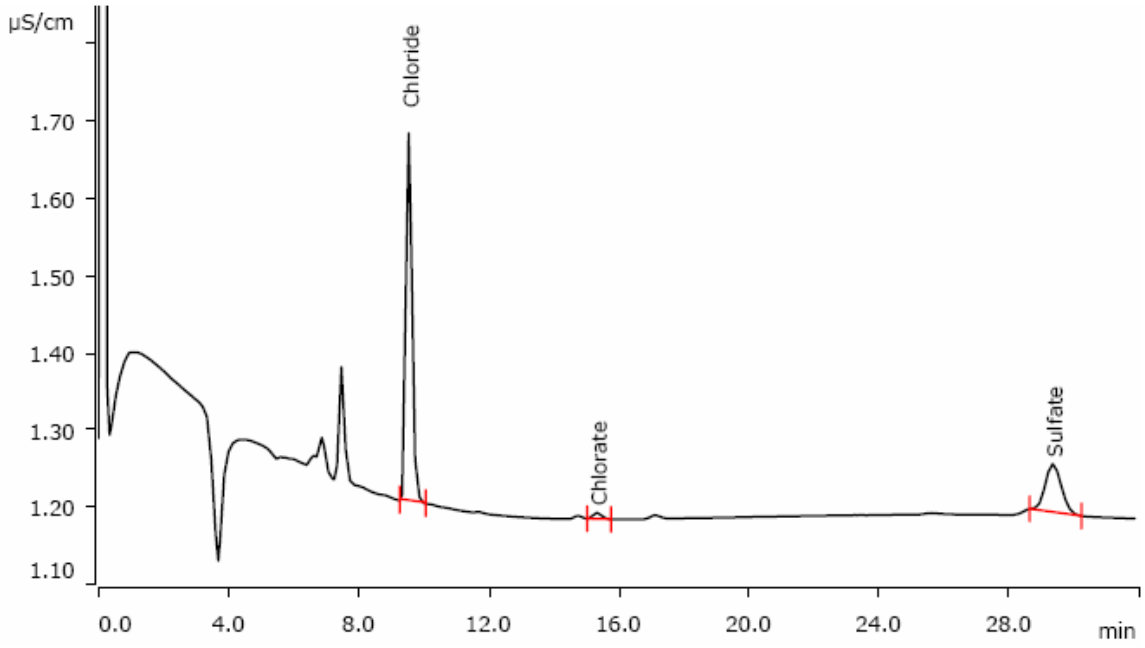
IC Application Note S–303

Anions in KOH (50%) applying Inline Neutralization and intelligent Partial Loop Injection Technique (MiPT)



Metrohm Inline Neutralization is a well-established sample preparation technique for anion determinations in hydroxide solutions. The intelligent Partial Loop Injection Technique (MiPT) allows to calibrate the system with one single standard solution and to adjust the injection volume according to the anion concentrations in the sample. This method has been successfully applied to anion analysis in potassium hydroxide (50 and 85%) and in potassium carbonate solutions (83%).

Results

	Concentration [µg/L]
Chloride	13.0
Chlorate	6.9
Sulfate	n.q.

Metrohm

Sample

Potassium hydroxide (50%)

Sample preparation

Inline Neutralization and intelligent Partial Loop injection Technique (MiPT)

Columns

Metrosep A Supp 7 - 250/4.0	6.1006.630
Metrosep RP 2 Guard/3.5	6.1011.030

Solutions

Eluent (inline eluent preparation)	3.6 mmol/L sodium carbonate
Suppressor regenerant	100 mmol/L sulfuric acid
SPM regenerant	100 mmol/L perchloric acid
Rinsing solutions	Ultrapure water

Analysis

Conductivity after sequential suppression

Parameters

Flow rate	0.8 mL/min
Injection volume	100 µL
P_{max}	15 MPa
Recording time	30 min
Column temperature	60 °C

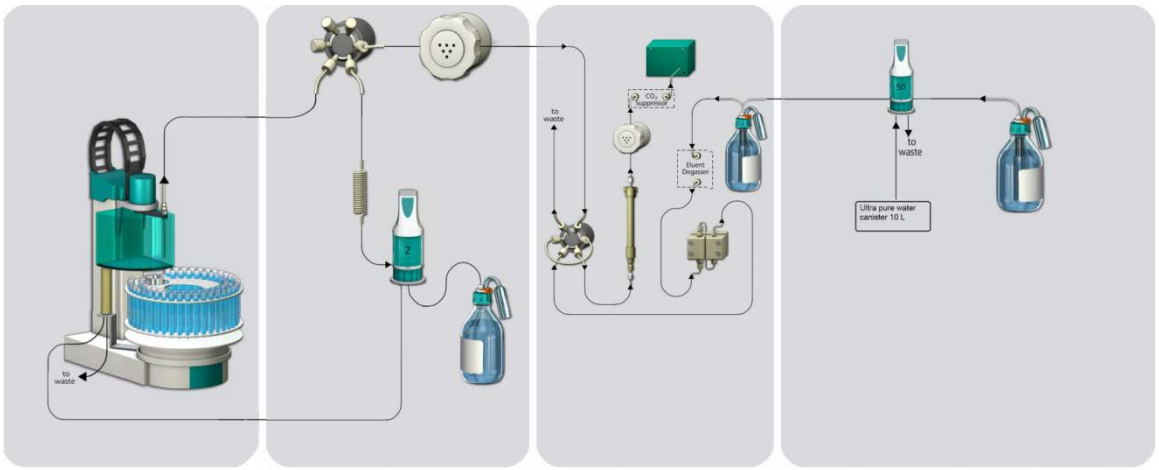
Instrumentation

850 Professional IC Anion – MCS – Prep 3	2.850.2190
IC Conductivity Detector	2.850.9010
858 Professional Sample Processor – Pump – Injector	2.858.0030
800 Dosino (liquid handling)	2.800.0010
849 Level Control for Inline Eluent Preparation	2.849.1030

Calibration MiPT

Calibration range	Factor of 10
Standard solution:	
Fluoride	10 µg/L
Chloride	500 µg/L
Chlorate	50 µg/L
Sulfate	500 µg/L
1. Level	10 µL = 50 / 5 / 1 µg/L
2. Level	20 µL = 100 / 10 / 2 µg/L
3. Level	50 µL = 250 / 25 / 5 µg/L
4. Level	100 µL = 500 / 50 / 10 µg/L

Flow chart



System setup: Sample Processor with rinsing station, diversion valve, 800 Dosino for liquid handling with transfer tubing, Sample Preparation Module (SPM), IC system with 250 µL loop for MiPT, and inline eluent preparation.

Procedure: The 800 Dosino aspirates 2 mL of sample into the transfer tubing; then 1 mL of this sample volume is used to condition the SPM and the capillaries to the injector. Subsequently, the injector is switched to the 'Fill' position and the exact volume of sample is dosed into the loop.

If sulfate has to be determined, the SPM regeneration requires perchloric acid.