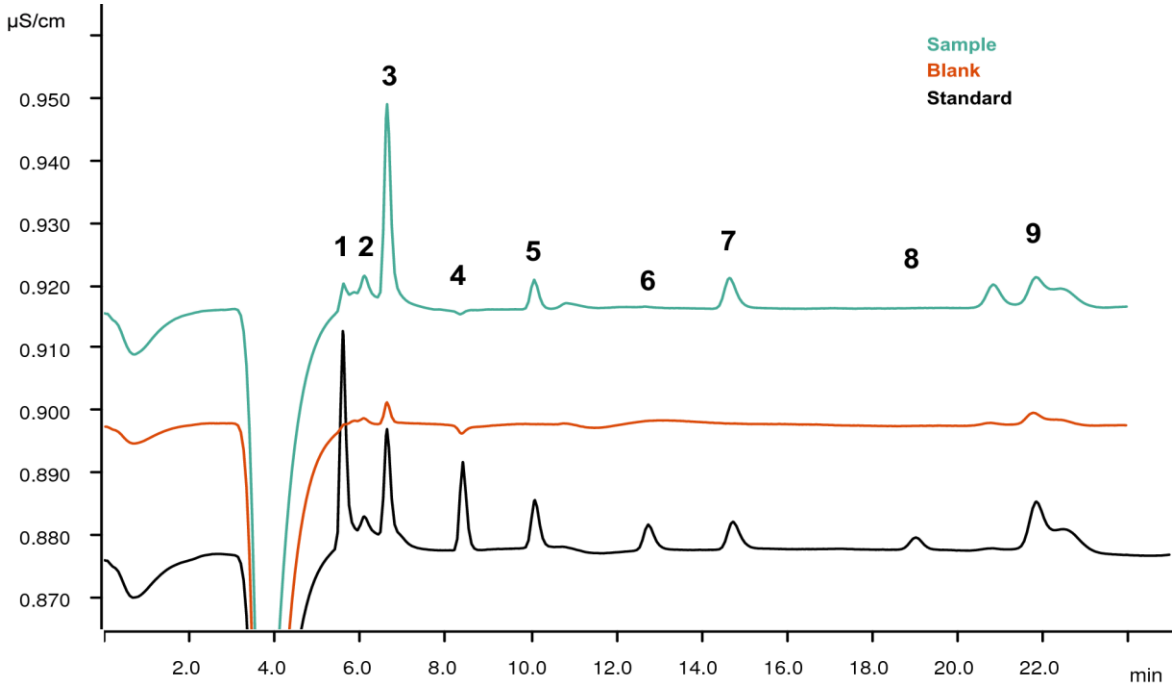
IC Application Note S–365

Anionic impurities in concentrated semiconductor grade ammonium hydroxide



Ultrapure chemicals are required to be applied in semiconductor industry. Ionic impurities may lead to compromised products. This application describes the determination of anionic impurities in semiconductor grade 28% ammonium hydroxide solution. To avoid matrix disturbances Inline Neutralization and Inline Preconcentration with Matrix Elimination needs to be applied.

Results

Anion	Conc. [µg/L]	RSD [%, n = 6]	Anion	Conc. [µg/L]	RSD [%, n = 6]
1 Fluoride	1.8	2.1	6 Bromide	n.d.	-
2 Acetate	11.1	13.7	7 Nitrate	20.8	2.6
3 Formate	56.7	2.9	8 Phosphate	n.d.	-
4 Chloride	n.d.	-	9 Sulfate	7.9	5.5
5 Nitrite	11.7	0.6			

Standard in graph: acetate, formate 8 µg/L each, all others 4 µg/L each

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Sample

Semiconductor grade ammonium hydroxide (28%)

Sample preparation

Dilution 1:5 with ultrapure water, Metrohm Inline Neutralization with subsequent Metrohm Inline Preconcentration technique with Matrix Elimination (MiPCT-ME).

Columns

Metrosep A Supp 5 - 250/4.0	6.1006.530
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	6.1017.200

Solutions

Eluent concentrate (1:40)	128 mmol/L sodium carbonate 40 mmol/L sodium hydrogen carbonate
Eluent	3.2 mmol/L sodium carbonate 1.0 mmol/L sodium hydrogen carbonate
Suppressor regenerant	100 mmol/L sulfuric acid
Rinsing solution	STREAM
SPM regenerant	100 mmol/L perchloric acid
SPM rinsing	Ultrapure water

Parameters

Flow rate	0.8 mL/min
Preconcentration volume	100 µL
P_{max}	15 MPa
Recording time	25 min
Column temperature	30 °C

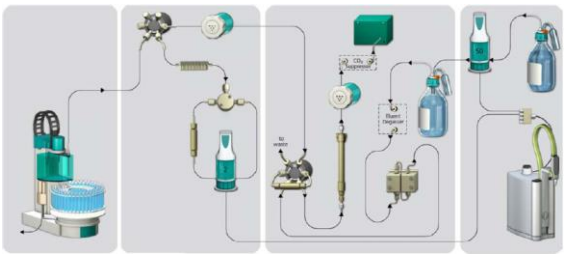
Analysis

Conductivity detection after sequential suppression

Instrumentation

940 Professional IC Vario ONE/SeS/PP/Prep 3	2.940.1530
IC Conductivity Detector	2.850.9010
858 Professional Sample Processor	2.858.0030
941 Eluent Production Module	2.941.0010
800 Dosino	2.800.0010
ELGA PURELAB flex 5/6	-
IC equipment: MiPT	6.5330.180
Sample rack 3 x 60.6 mm and 35 x 41 mm	6.9920.191
MSM Rotor A	6.2832.000
Adapter sleeve for Suppressor Vario	6.2842.020
SPM Rotor A	6.2835.000
20 x PE bottle, 50 mL	6.1608.100
20 x Septum screw cap	6.1627.100
Sample needle MF 1/16 in.	6.2624.200
Needle/tube holder 1/16 in.	6.2833.010

Setup scheme



Remarks

The use of the ELGA PURELAB flex 5/6 with its direct ultrapure water supply is a prerequisite to achieve the required very low blank values. The preconcentration of 20 µL of the undiluted sample instead of 100 µL of the 1:5 dilution shows the same results.

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