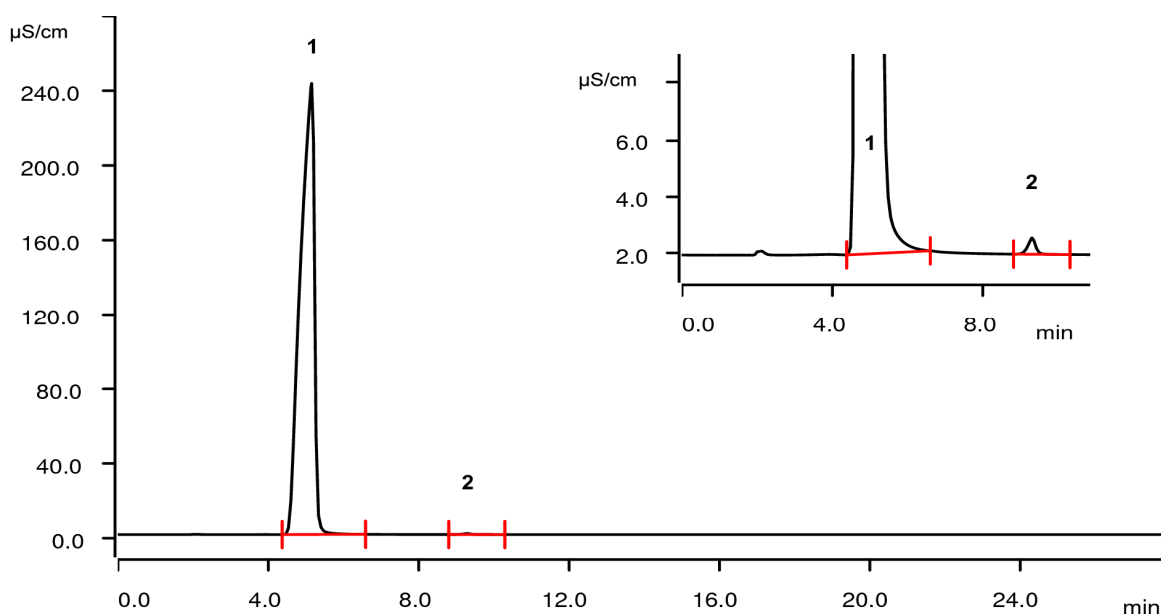


Fluoride and chloride in sodium fluoride for pharmaceutical use

USP monograph modernization initiative: ion chromatography applying a hydroxide eluent on a Metrosep A Supp 16 - 250/4.0 column (L91)



Chromatogram of the system suitability test for impurities (concentrations: sodium fluoride 1 mg/mL and sodium chloride 1 µg/mL corresponding to: fluoride 0.45 mg/mL and chloride 0.61 µg/mL).

Sodium fluoride for pharmaceutical use needs to comply with USP requirements. The actual monograph (USP 42) uses three different methods for the identification, the assay, as well as the content of chloride as an impurity. Ion chromatography allows the measurement of these three parameters in two determinations on a single system. In the course of the USP monograph modernization, this ion chromatographic approach brings an increased ease of use for this type of analysis.

Results

Anion	Sample ID	Result [%]	USP limit [%]
1 Fluoride	Assay NaF [%]	99.7	98-102
2 Chloride	Impurity chloride [%]	0.0016	≤ 0.012

For further results, see next page.

Sample

2 µg/mL sodium fluoride in ultrapure water.

Sample preparation

None

Columns

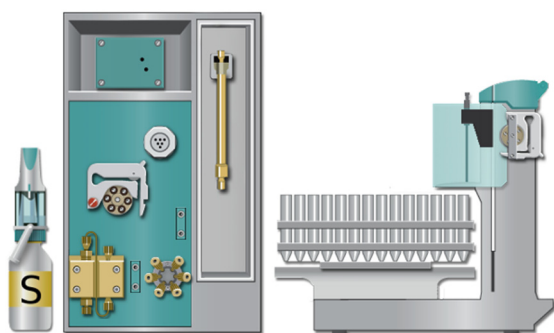
Metrosep A Supp 16 - 250/4.0	6.1031.430
Metrosep A Supp 16 Guard/4.0	6.1031.500

IC Solutions

Eluent	15 mmol/L potassium hydroxide
Regenerant Dosino	500 mmol/L sulfuric acid
Rinsing	Ultrapure water

Instrumentation

930 Compact IC Flex ChS/PP/Deg	2.930.2360
IC Conductivity Detector	2.850.9010
919 IC Autosampler plus	2.919.0020
800 Dosino for Dosino Regeneration	2.800.0010
MSM Rotor A	6.2832.000
Adapter sleeve for Suppressor Vario	6.2842.020
IC equipment: Dosino Regeneration	6.5330.190



Parameters IC

Flow rate	1.0 mL/min
Injection volume (MiPT)	20 µL
P _{max}	20 MPa
Column temperature	40 °C
Recording time	28 min

Analysis

Conductivity detection after sequential suppression

System suitability requirements for assay

USP Parameter	Result	USP required	Remarks
Resolution F ⁻ /acetate	NLT 1.5	5.9	Pass
Tailing factor F ⁻	NMT 2.0	1.1	Pass
RSD F ⁻ [%; n=5)	NMT 0.73%	0.52	Pass

System suitability requirements for impurities

USP Parameter	Result	USP required	Remarks
Resolution F ⁻ / Cl ⁻	NLT 4.0	7.7	Pass
RSD F ⁻ [%; n=5)	NMT 5.0%	4.2%	Pass
Signal/Noise for Cl ⁻	NLT 20	> 740	Pass

NLT = not less than, NMT = not more than

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