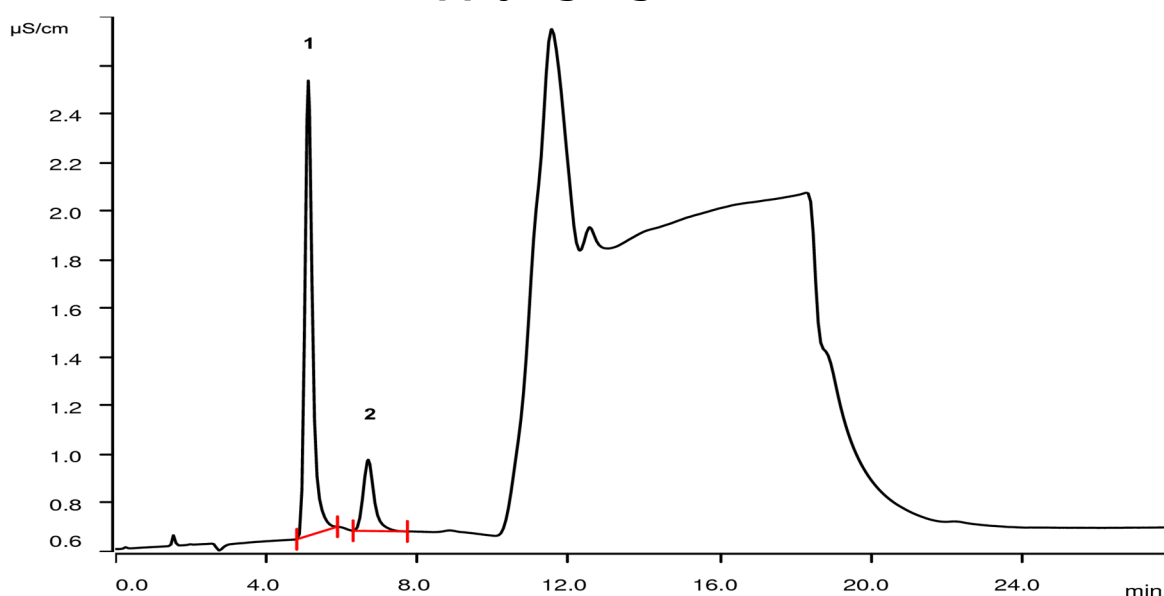


IC Application Note S-379

Fluoride in sodium fluoride tablets for pharmaceutical use

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



Chromatogram of the System Suitability Test for assay (concentrations; sodium fluoride 2 µg/mL, sodium acetate 1 µg/mL).

Sodium fluoride tablets for pharmaceutical use needs to comply with USP requirements. The actual monograph (USP 42) uses two different methods for the identification and the assay. Ion chromatography allows the determination of these two parameters in one single analysis. In the course of the USP monograph modernization, this ion chromatographic approach makes this type of analysis even easier.

Results

Anion	Sample ID	Result [%]	USP limit [%]
1 Fluoride	Assay NaF [%]	98.8	90–110
2 Acetate	Impurity	n.q.	

n.q. = not quantified. For further results, see next page.

Sample

Sodium fluoride tablets

Sample preparation

20 Tablets are homogenized, then 0.215 g of the powder is dissolved into 1000 mL ultrapure water. Final dilution to a nominal concentration of 2.0 mg/L sodium fluoride.

Columns

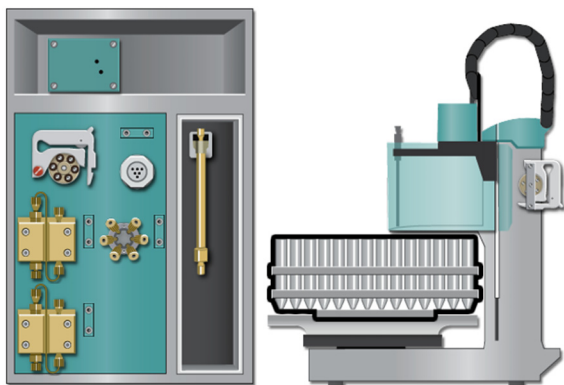
Metrosep A Supp 16 - 250/4.0	6.1031.430
Metrosep A Supp 16 Guard/4.0	6.1031.500

IC Solutions

Eluent A	100 mmol/L potassium hydroxide
Eluent B	Ultrapure water
Regenerant	100 mmol/L sulfuric acid
Rinsing	Ultrapure water

Instrumentation

940 Professional IC Vario One/SeS/PP/HPG	2.940.1540
IC Conductivity Detector	2.850.9010
858 Professional Sample Processor	2.858.0020
MSM Rotor A	6.2832.000
Adapter sleeve for Suppressor Vario	6.2842.020



Analysis

Conductivity detection after sequential suppression

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

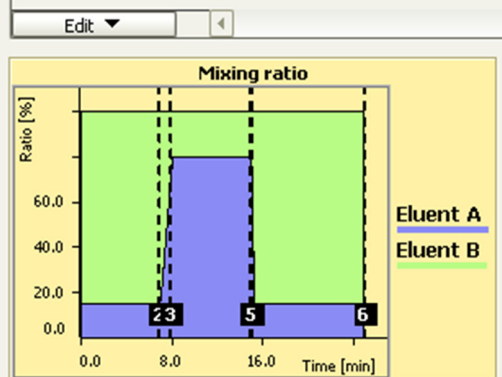
System suitability requirements for assay

USP Parameter	Result	USP required	Remarks
Resolution F ⁻ /acetate	NLT 1.5	3.7	Pass
Tailing factor F ⁻	NMT 2.0	1.4	Pass
RSD F ⁻ [%; n=6]	NMT 2.0%	0.67	Pass

Gradient

Gradient

	Time [min]	Eluent A [%]	Eluent B [%]	Curve	Flow
▶ 1	Start	15	85		1.0
2	7.0	15	85	Linear	1.0
3	8.0	80	20	Linear	1.0
4	15.0	80	20	Linear	1.0
5	15.1	15	85	Linear	1.0
6	25.0	15	85	Linear	1.0
7					



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