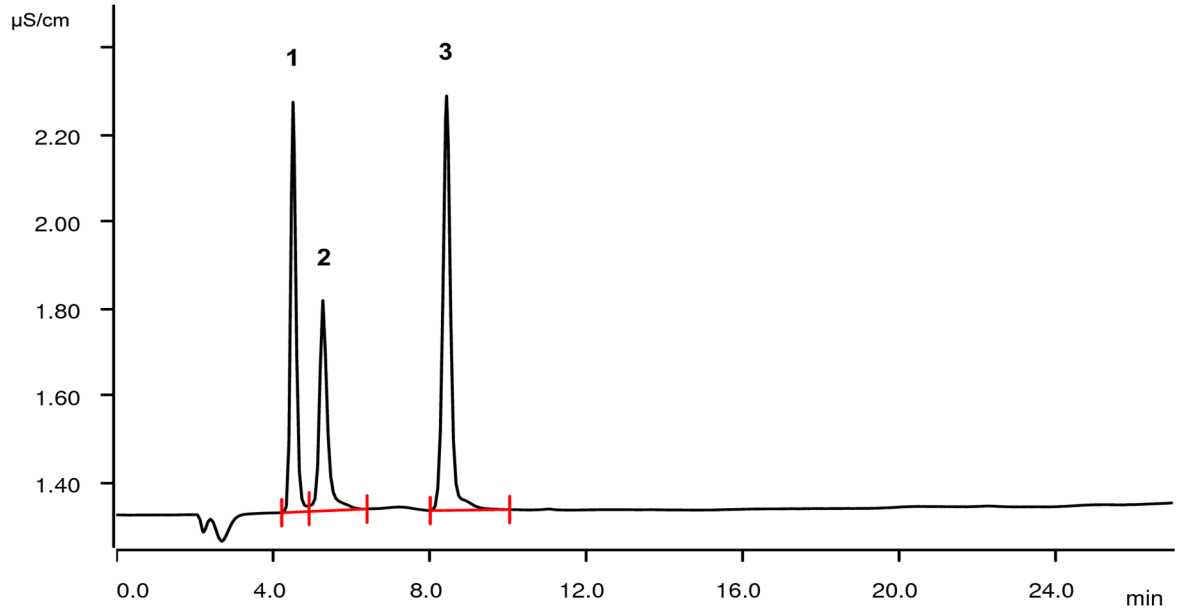
IC Application Note S–385

Fluoride in stannous fluoride gel for pharmaceutical use

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



Chromatogram of the assay (concentration; fluoride 1 µg/mL). The separation of fluoride and the unknown component (2) still fulfills the USP requirements.

Stannous fluoride gel for pharmaceutical use needs to comply with USP requirements. The actual monograph (USP 42) utilizes two different methods for the identification and the assay. Ion chromatography allows the analysis of these two parameters within a single determination. 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 [%]	100.1	90–110
2	unknown	Impurity	n.q.	-
3	Chloride	Impurity	n.q.	-

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

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Sample

Dental gel based on stannous fluoride.

Sample preparation

Dilution with ultrapure water, sonication for 20 min, filtration through a 0.2 µm filter. The final concentration is 1 µg/mL.

Columns

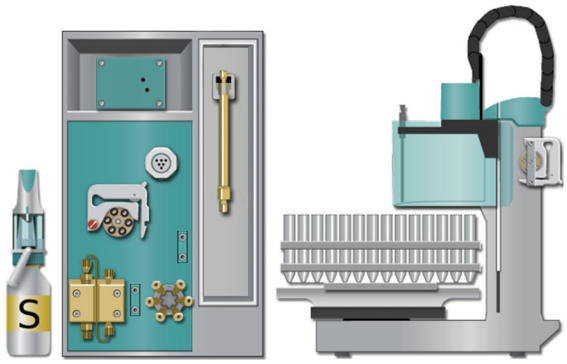
Metrosep A Supp 16 - 250/4.0	6.1031.430
Metrosep A Supp 16 Guard/4.0	6.1031.500

IC Solutions

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

Instrumentation

930 Compact IC Flex Oven/ChS/PP/Deg	2.930.2360
IC Conductivity Detector	2.850.9010
858 IC Autosampler plus	2.858.0020
800 Dosino for DR	2.800.0010
MSM-HC Rotor A	6.2832.000
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	27 min

Analysis

Conductivity detection after chemical suppression

System suitability requirements for assay

USP Parameter	Result	USP required	Remarks
Resolution F^-/acetate	NLT 1.5	5.4	Pass
Tailing factor F^-	NMT 2.0	1.1	Pass
RSD F^- [%, n=5]	NMT 2%	0.24	Pass
Resolution F^-/unknown	(NLT 1.5)*)	2.6	Pass

NLT = not less than, NMT = not more than
*) resolution requirement for F-/acetate

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