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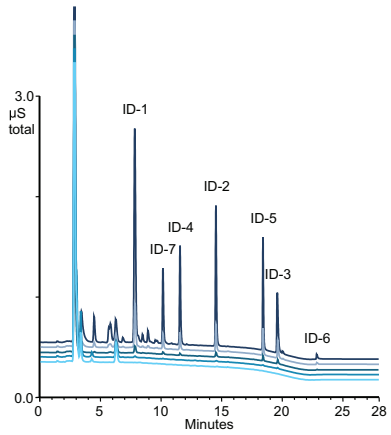
Cationic Impurities in Colesevelam Analyzed by MPIC Using IonPac NS2



Peaks: ID-1: 6-Aminohexyltrimethylammonium
ID-2: 6-Bromohexyltrimethylammonium
ID-3: Decylamine
ID-4: Aminodi(hexyltrimethylammonium)
ID-5: Decyl-6-aminohexyltrimethylammonium
ID-6: Didecylamine
ID-7: structure not given

Column:	Thermo Scientific™ Dionex™ IonPac NS2, 5 µm
System:	Thermo Scientific™ Dionex™ ICS-5000 Quaternary gradient pump w/ GM-4 mixer AS-AP sampler 400 µL sample loop
Dimensions:	4.0 × 150 mm PEEK
Mobile Phases:	A: D.I. water B: 10 mM Heptafluorobutyric acid C: Acetonitrile

Gradient:

Time	%A	%B	%C	curve
-8	76	20	4	5
0	76	20	4	5
20	0	20	80	7
28	0	20	80	5

Flow rate:	0.80 mL/min
Temperature:	Column 25 °C; detector 15 °C
Injection:	10 µL
Detection:	Suppressed conductivity, chemical mode
Suppressor:	CMMS-300 (4mm)
Regenerant:	0.05 M Tetrabutylammonium hydroxide, 3 mL/min; 4-L pressurized reservoir
Samples:	Standards 25, 5.0, 1.0, 0.20, 0.04 and 0.00 µg/mL in 50% methanol + 5 mM HCl

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Colesevelam is a hydrogel polymer used to treat high cholesterol by sequestering bile acids in the intestines. It is a strong anion exchanger, containing a list of amine impurities from the byproducts of manufacturing and degradation. These impurities have a wide range of hydrophobicity and charge thus their determination is challenging. Suppressed conductivity provides good selectivity and sensitivity for charged analytes, including ionic surfactants. Mobile-phase ion-chromatography (MPIC) is a method where an ion-pairing agent in the eluent enhances retention for charged analyte on a hydrophobic column, and then is removed by the suppressor to provide selective and sensitive detection of the analytes. For analysis of cations, heptafluorobutyric acid is one such agent that is amenable to suppression. The IonPac NS2 is a silica-based, high-efficiency, reversed-phase column designed for separation of charged analytes using MPIC with suppressed conductivity detection. For this application, it delivers good resolution and quantification limits of 0.1–0.7 µg/mL. The elution conditions are also compatible with Corona charged-aerosol detection.