

A QUICKER METHOD FOR THE ANALYSIS OF AMMONIA IN E-CIGARETTE AEROSOLS AND E-LIQUIDS BY ION CHROMATOGRAPHY

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Abstract

Ammonia has been routinely tested for cigarette smoke for a number of years. Common methods employed are based on ion chromatography using cation exchange columns such as Dionex IonPac CS12 and CS16 columns. These two columns provide good separation of the ammonium peak from the preceding sodium peak. However, the run times including equilibrating time typically exceed well over 20 minutes because of the high retention of ammonium in these columns. Herein we wish to present a quicker and robust method utilizing Dionex CS18 column with run time of 13 minutes (including equilibrating time) that still provides good separation of ammonium peak from sodium peak. The method LOQ is 0.16 µg/puff, with no quantifiable ammonia determined in the e-cigarette sample matrices we have analyzed. A Dionex ICS 5000 ion chromatography system has been utilized for the method. The new method has a calibration range of 0.1 to 2 µg/mL for ammonia and has been validated with good recoveries on e-cigarette aerosols and e-liquids.

Calibration Standards and LOQ

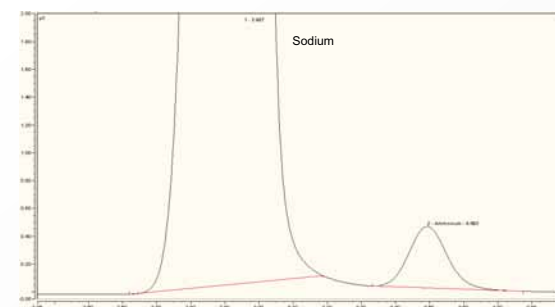
The calibration range is 0.1 to 2 µg/mL, with the calibration curve having quadratic regression, intercept not forced to zero, and 1/x weighting. The LOQ is set at the lowest spiking level (0.2 µg/mL) during the method validation, rather than the lowest standard level. The method LOQ is 0.16 µg/puff for aerosol and 26.7 µg/g for e-liquid.

Results

Recoveries

E-Cigarette	Spike Level	Aerosol	E-Liquid
E-Cigarette A	Low Spike	101%	106%
	Mid Spike	84%	97%
	High Spike	82%	94%
E-Cigarette B	Low Spike	108%	120%
	Mid Spike	79%	103%
	High Spike	99%	93%

Aerosol LOQ Spike Sample Chromatogram from E-Cigarette A



Note: Large sodium peak is due to glass impinger being used for aerosol collection. Ammonium peak only available in spiked samples.

Method

Aerosol from e-cigarettes is generated under conditions specified by the collection regime, passed through a 44-mm glass fiber filter and collected in one impinger containing 40 mL of 6 mM aqueous HCl solution. After aerosol collection, the filter is combined with the contents in the impinger and extracted. E-liquids are extracted in 20 mL of extraction solution instead. The extracts are filtered and subsequently analyzed by Ion Chromatography.

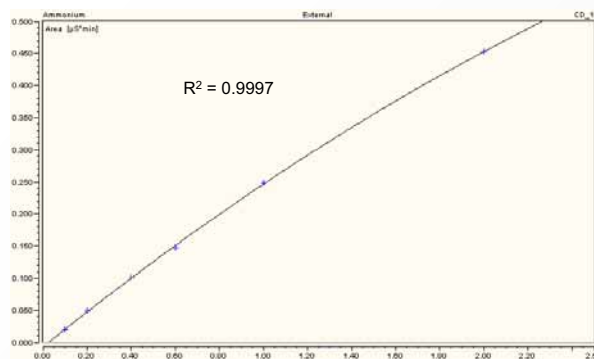
IC Conditions

Column: Dionex IonPac CS18 Analytical 2 x 250 mm
Column temperature: 30 °C
Mobile phase: methanesulfonic acid (MSA) in DI water
Flow rate: 0.35 mL/min
Gradient: 0-4 min, 3 mM MSA; 4.1 min, 15 mM MSA (hold for 3.9 min)
8.1 min, 3 mM MSA (hold for 4.9 min)
Injection volume: 10 µL

Detector Parameters

Suppressor: CERS 500, 2 mm
Detector cell temperature: 30 °C
Compartment temperature: 23 °C
Suppressor current: 4 mA

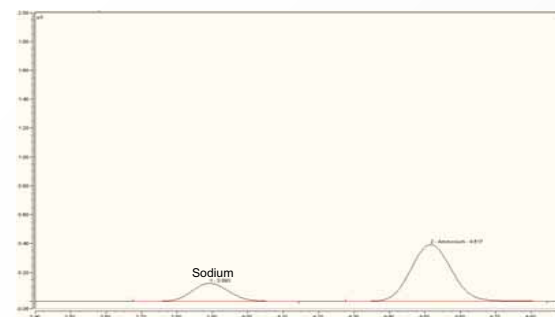
Calibration Curve



S/N at Low Standard levels

	Ammonium
Std 1 (0.1 µg/mL)	128
Std 2 (0.2 µg/mL)	325

E-Liquid LOQ Spike Sample Chromatogram from E-Cigarette A



Note: Ammonium peak only available in spiked samples.

Conclusions/Future Work

ITG Brands has developed a quicker and robust method for the analysis of ammonia in e-cigarette aerosols and e-liquids utilizing Dionex CS18 column by ion chromatography with good accuracy and sensitivity. The method may be used for cigarette smoke samples analysis in the future if non-glass impingers can be utilized to avoid interferences from sodium peak.