

A MODIFIED AND IMPROVED IC METHOD FOR THE DETERMINATION OF AMMONIA IN MAINSTREAM CIGARETTE SMOKE

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ABSTRACT

Ammonia is included in the draft list of Harmful and Potentially Harmful Constituents in tobacco products and tobacco smoke under Section 904(a) (3) of the Federal Food, Drug and Cosmetic Act.

Eurofins | Lancaster Laboratories has developed an analytical method that can effectively quantitate ammonia in mainstream smoke. Our modified and improved method is specific, sensitive, simple and robust. The newly developed and validated method is an improvement over the historical method. A Cerulean SM450 smoke machine equipped with a Hoffman capture system is used to collect mainstream smoke onto a Cambridge filter pad and one impinger containing 40 mL of 0.006M HCl to effectively trap ammonia. The sample is analyzed on a Dionex ICS 2100 ion chromatograph, which is equipped with eluent generation and which utilizes a lower flow rate. An IonPac CS12 column is used to provide better separation of ammonia from any interference. The average spike recoveries obtained were within $\pm 25\%$ of the theoretical value. This method is linear from approximately 0.39 $\mu\text{g/mL}$ to 3.54 $\mu\text{g/mL}$ ammonia. The results obtained by this method were within the range of results from the CORESTA Special Analytes Task Force.

EQUIPMENT

- Cerulean SM450 smoke machine, fitted with Hoffman capture system
- Dionex ISC-2100 Ion Chromatography system equipped with a DS6 heated-conductivity cell and a Dionex AS or AS_AP autosampler
- Dionex EGIII MSA eluent generator cartridge
- Dionex IonPac CS12 2 mm x 250 mm column and CG12 2 mm x 50 mm guard column
- Dionex CSRS-300 cation self-regenerating suppressor

METHOD OPTIMIZATION

Experiments were conducted to optimize the extraction solution, the # of impingers and the vol. of extraction solution in an impinger, extraction time and IC column for a robust method and good chromatography. CS12A and CS12 columns were explored. The CS12 column gave better peak shape and separation of ammonia from its interfering peaks.

SAMPLE PREPARATION

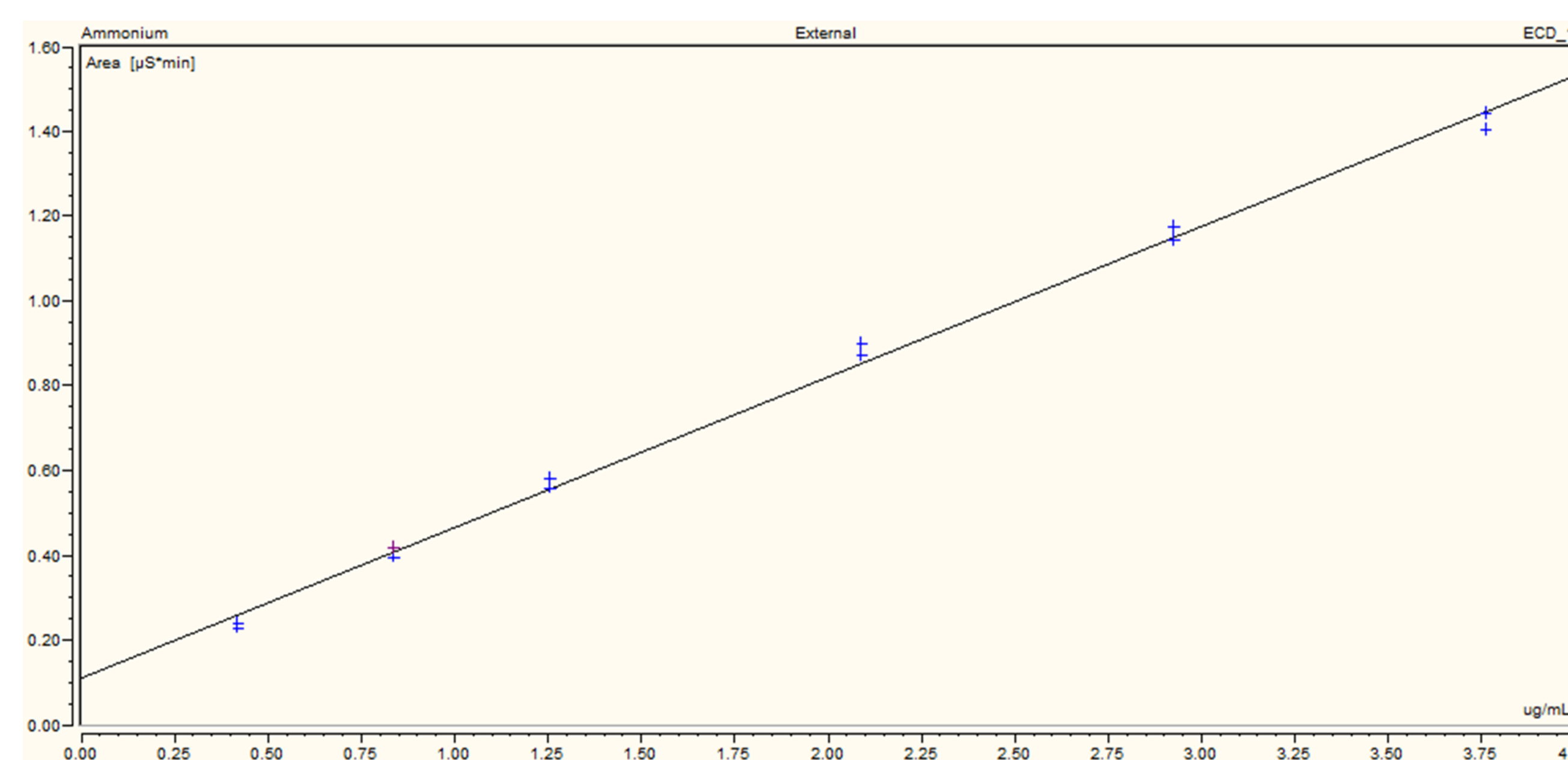
The smoke machine is set up with one Cambridge filter pad and one impinger containing 40 mL of 0.006 M HCl per port. After smoking, the filter and extraction solution are combined and shaken for 1 hour at 300 rpm. After shaking the samples are filtered through an 0.45 μm PVDF filter into an LC vial.

ION CHROMATOGRAPH PARAMETERS

Flow Rate: 0.30 mL/min
Injection Volume: 20 μL
Column Temperature: 30 $^{\circ}\text{C}$
Detector Temperature: 35 $^{\circ}\text{C}$
Autosampler Temperature: 5 $^{\circ}\text{C}$
Continuously Regenerated Trap Column: ON
Suppressor Current: 36 mA
MSA Concentration: 8 mM from 0 to 9 minutes, 40 mM from 9 to 13 minutes, 8 mM from 13 to 20 minutes
Total Run Time: 20 minutes

LINEARITY AND LOD/LOQ

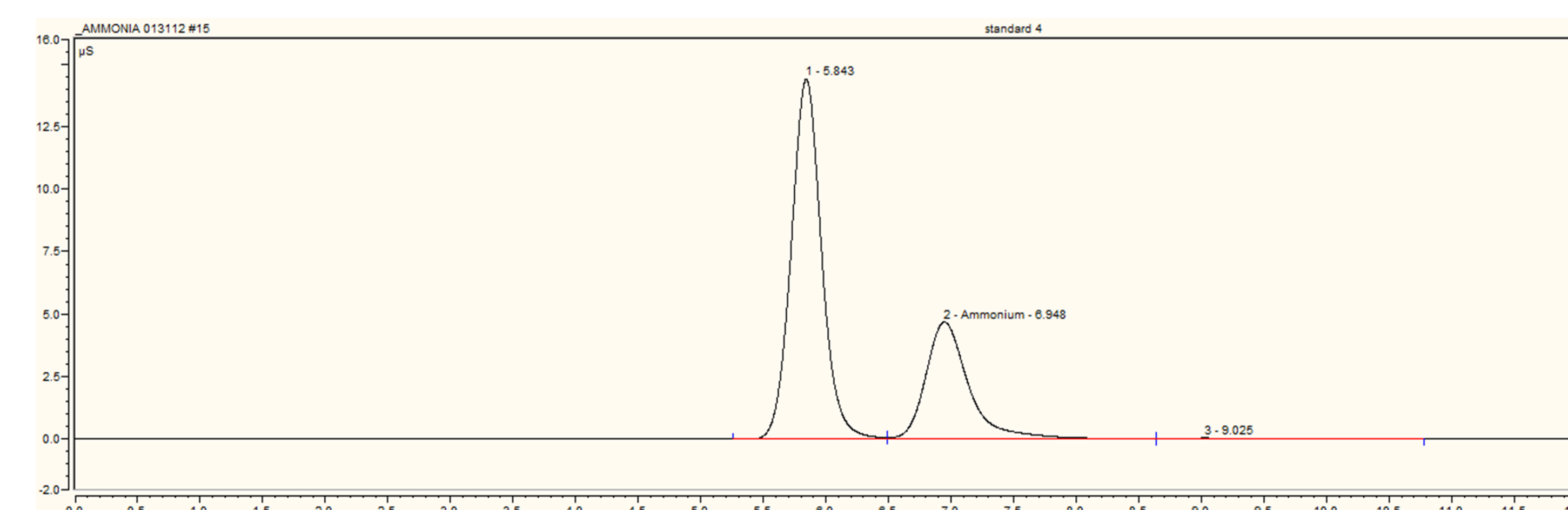
This method is linear through the calibration range of approximately 0.39 $\mu\text{g/mL}$ to 3.54 $\mu\text{g/mL}$ ammonia. A representative calibration curve shown below has a coefficient of determination of 0.9966. The theoretical limit of quantitation (LOQ) for ammonia is 0.1378 $\mu\text{g/mL}$ and the theoretical limit of Detection (LOD) is 0.0414 $\mu\text{g/mL}$.



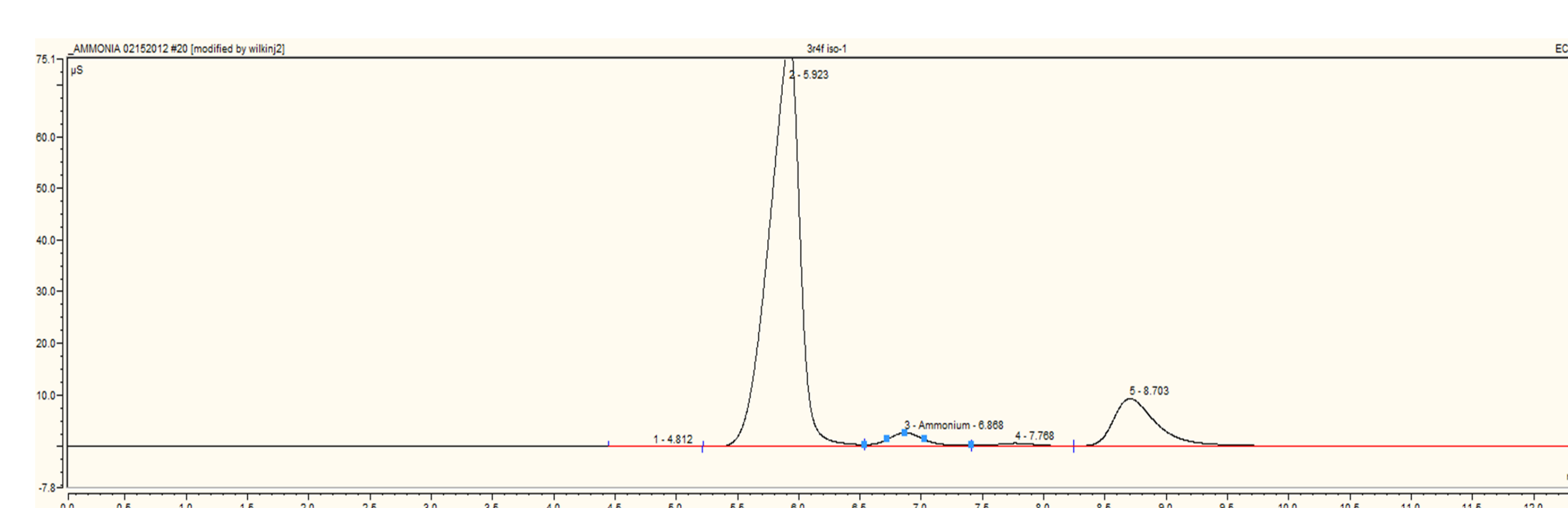
ACCURACY

A standard prepared from a second source was run on seven separate days and results were within 10% of the expected value with a %RSD of < 4 . 3R4F cigarettes were analyzed using both Health Canada and ISO smoke regimes to establish recovery. The ISO recovery results ranged from 88.5% to 122.5%. The Health Canada smoke regime recovery results ranged from 92.3% to 98.1%. For comparison with other CORESTA Special Analytes Task Force (SATF) data, three replicates of 2R4F and three replicates of 1R5F cigarettes were smoked under the ISO smoke regime. This work gave an average result of 7.74 $\mu\text{g/cig}$. For 2R4F cigarettes and 1.7 $\mu\text{g/cig}$ for 1R5F cigarettes. These results were within the range of results from the SATF.

STANDARD CHROMATOGRAM



SAMPLE CHROMATOGRAM



CONCLUSIONS

Eurofins | Lancaster Laboratories has developed an analytical method that can effectively quantitate ammonia in mainstream smoke. This method uses a Cerulean SM450 smoke machine with Hoffman capture system and an ICS-2100 ion chromatograph with eluent generation. Our modified and improved method is specific, sensitive, simple and robust.