THE DETERMINATION AND COMPARISON OF AMMONIA CONTENT IN MAINSTREAM TOBACCO SMOKE BY ION CHROMATOGRAPHY AND SPECTROPHOTOMETRIC METHODS

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Purpose

The FDA Center for Tobacco Products (CTP) has identified ammonia as a harmful or potentially harmful constituent (HPHC) in tobacco and tobacco products. However, there is currently not a generally accepted method for the determination of ammonia in mainstream tobacco smoke. Several analytical techniques are commonly used to measure ammonia, including ion chromatography (IC) with a conductivity detector, LC-MS/MS and spectrophotometric detection. For this study, two methods were used, ion chromatography and spectrophotometric detection.

Methods

In this study, the ammonia amounts in mainstream smoke from 3R4F, CM7 and 1R5F reference cigarettes under both the ISO and the Canadian Intense smoking regimes were determined by two different analytical methods.

Sample Preparation-Smoke collection

All samples were collected on a Cerulean SM450 smoke machine and were smoked under both the ISO and Intense regimes.

Sample Preparation-IC

- Samples were collected on a Cambridge filter pad followed by a impinger containing 40mLs of 0.025 M H_2SO_4 solution for the IC analysis
- The Cambridge filter pad was extracted in 40mLs of 0.025 M H_2SO_4 solution from the impinger for 30min on orbital shaker at 200 rpm.
- Samples were syringe filtered using a 0.45 µm filter prior to the analysis on the IC system.

Sample Preparation-UV-Vis

- Samples were collected by two impingers containing 50mLs of saturated boric acid solution. Following smoking the samples had 20 minute hold to allow the smoke to absorb in the solution
- Samples were treated with charcoal and filtered
- Samples were then treated with a chlorine solution and held for 4 minutes
- Samples then had a catalyst and a phenol solution added and allow the reaction to occur for exactly 7 minutes
- Finally a 3 M NaOH solution was added to stop the reaction.
- The indophenol blue color is stable for 1 hour.

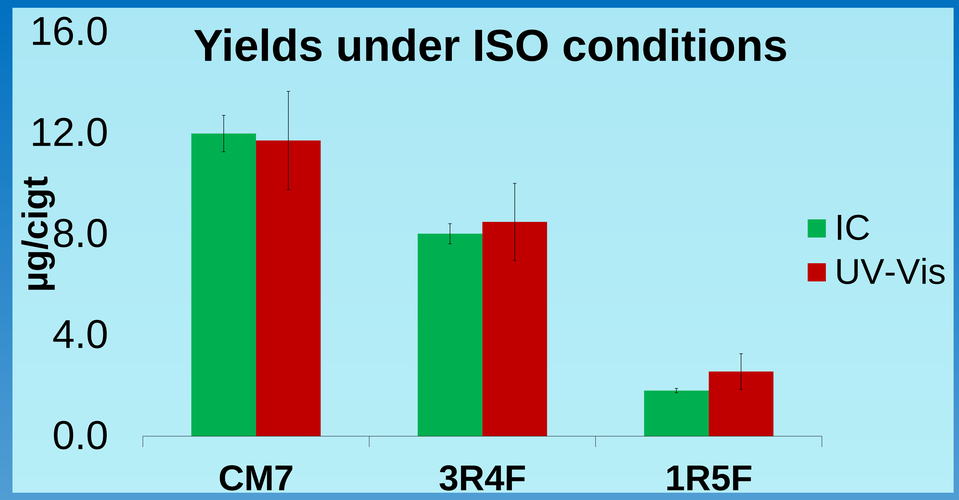
Instrumental Conditions

- **UV-Vis- Method***
  Method = chlorine/phenol
  Wavelength = 625mm
  Run time = ~2 samples per hour
  Calibration curve range: 0.10 – 4.0 µg/mL ammonia
- **IC with a Electrochemical detection**
  Method = Conductivity
  Run time = ~3 samples per hour
  Calibration curve range: 0.094- 9.4 µg/mL ammonia

Results

Method Precision 3R4F	UV-Vis	IC
AVG (µg/cigt.)	8.48	8.02
Std. Dev.	1.53	0.399
%RSD	18.0%	4.97%

Cigarette	1R5F-ISO		1R5F-Intense	
Method	UV-Vis	IC	UV-Vis	IC
AVG (µg/cigt.)	2.56	1.81	14.6	22.9
Std. Dev.	0.71	0.09	2.21	1.89
%RSD	27.6%	4.72%	15.2%	8.27%



Cigarette	CM7-ISO		3R4F-ISO	
Method	UV-Vis	IC	UV-Vis	IC
AVG (µg/cigt.)	11.7	12.0	8.48	8.02
Std. Dev.	1.94	0.721	1.53	0.399
%RSD	16.6%	6.02%	18.0%	4.97%

Method LOD	UV-Vis	IC
LOD (µg/cigt.)	0.103	0.201
LOQ (µg/cigt.)	0.342	0.669

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Conclusion

The concentrations of ammonia are comparable across the two different analytical methods (UV-Vis and IC) used on this study under ISO conditions. There is more variability in the data when cigarettes are smoked under Intense conditions.
For both methods, the LOD and LOQ are comparable.
The method precisions for the IC were excellent with % RSD of less than 10%.

* Harrell, K.L "Colorimetric Method for the Determination of Ammonia in Tobacco Smoke" Tobacco Science 147; 1975, pages 145-147