Analytical Method Development for the Analysis of E-Liquids

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Abstract

Electronic cigarettes are one of the most commonly used methods of nicotine delivery, especially among the adolescent population. Due to the lack of regulation in manufacturer labeling of refillable nicotine solutions (e-liquids), the actual concentration of nicotine and other additives is variable as compared to the reported values. Misreporting of nicotine content is a contributor in the development of nicotine dependency and potentially tobacco product dependency. The objective of this research is to develop reliable analytical methods to study the variations in nicotine levels in e-liquids, and to identify and quantify other potentially harmful additives in e-liquids. In this research we used gas chromatography-mass spectrometry (GCMS) and nuclear magnetic resonance (NMR) spectroscopy for identification of compounds, and we used high-performance liquid chromatography (HPLC) and GCMS for quantification of compounds.

Introduction

- Electronic nicotine delivery systems (ENDS), commonly referred to as E-Cigs, are considered an effective cessation therapy for tobacco smoking.
- E-Cigs are increasingly used among adolescents, necessitating the development of stricter regulations.
- Prior research shows that manufacturer claims of nicotine content in refillable nicotine solutions are largely discrepant to nicotine content found by direct chemical analysis.
- To quantify and qualitatively identify nicotine levels, it is necessary to have the appropriate instrumentation that can provide reliable results.
- Each analytical technique requires the optimization of several parameters and sufficient knowledge of the theory behind the method.
- While this process is time consuming, it is essential to establish which instruments will be most useful for subsequent steps in this research.

Standard & Internal Standard Preparation

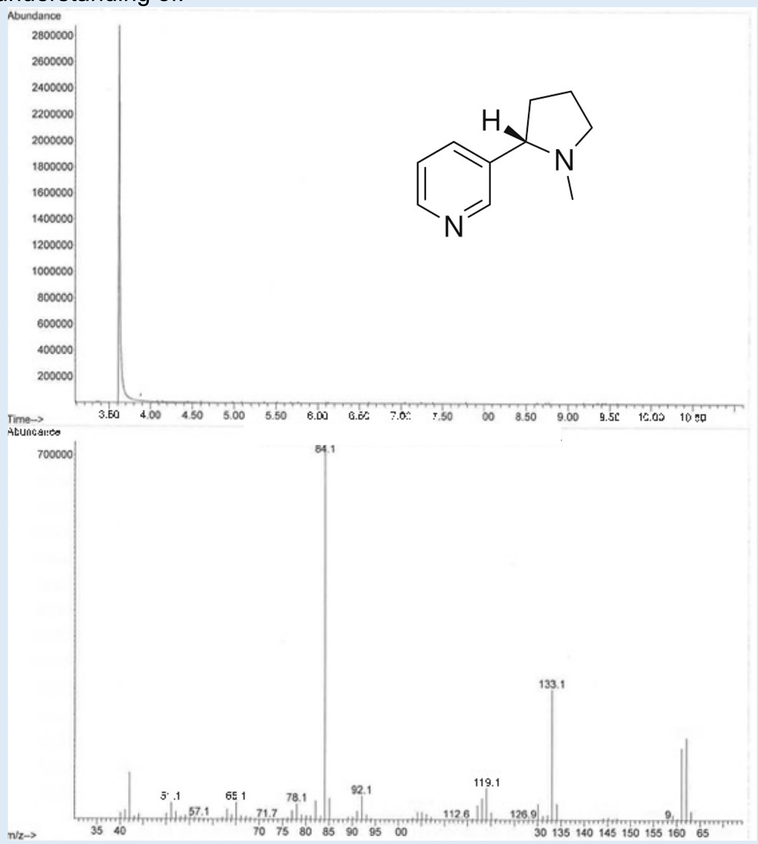
- Six standard nicotine solutions were prepared from stock standard solution (1mg/mL) with methanol and were stored at 4℃. These included: 100, 200, 400, 600, 800, and 1000 µg/mL standards.
- 15 µg of the internal standard, 99% quinoline, was added to all nicotine standards.
- Each sample was run in triplicate to prepare a nicotine calibration curve for the HPLC.

NMR

- Data was collected on an Anasazi Eft-90 NMR Spectrometer.
- Since our samples are in methanol, a solvent-suppression sequence was used to minimize the solvent signal and thus increase the resolution of the solute signals.
- The pre-programmed solvent-suppression sequence available did not adequately suppress the solvent signal, thus a better solvent-suppression sequence must be programmed before useful NMR spectra can be obtained.

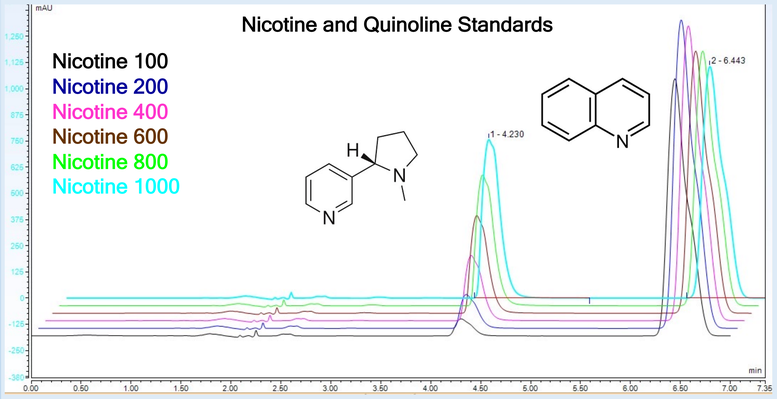
GCMS

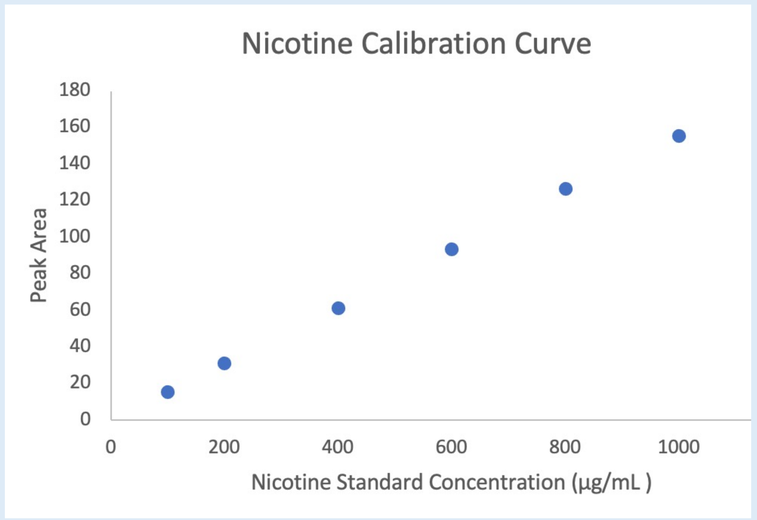
- Data was collected on an Agilent 6890N GC paired with a 5973N MS.
- The instrument is equipped with a C_{18} column and uses helium as the carrier gas.
- The initial column temperature was set to 80°C upon injection and increased at a rate of 25°C/min. to the final temperature of 245°C, which was then held for 4.5 minutes.
- The total run-time was about 11 minutes for each injection.
- Nicotine standards without quinoline were injected.
- For now, the use of the GCMS for this project is not feasible as it presents a complex system of parameters that we currently do not have thorough understanding of.



HPLC

- Data was collected on a Thermo Scientific Vanquish HPLC.
- The solvent mixture used consisted of 70% water, 30% acetonitrile, and 0.1 µg/mL of triethylamine at a flow rate of 1 mL/min. UV lamp wavelength was set at 254 nm.





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