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Technical Report

# Quantification of Nicotine in E-cigarette Liquid Sample Using GC-FID and Hydrogen Carrier Gas

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## Abstract:

Nicotine content is an important attribute for e-cigarette liquid and can be easily assayed using gas chromatography techniques. In this report, an e-cigarette liquid sample was analyzed by the Shimadzu GC-2030 using $H_2$ carrier gas and the concentration of nicotine was determined. This instrument setup provides an easy and robust system for routine analysis with accurate and precise results.


## 1. Introduction

E-cigarette liquid (or e-liquid) commonly contains four main components: propylene glycol, glycerin, flavoring compounds and nicotine. The concentration of nicotine determines the potency of the e-liquid, making it a key quality attribute.

The quantification of nicotine content can be easily achieved using a gas chromatograph equipped with a flame ionization detector (FID). In this report, the nicotine concentration in an e-liquid sample was determined using a Shimadzu GC-2030 with FID, coupled with the rugged AOC-20i autoinjector for easy sample introduction. Moreover, the assay was carried out using hydrogen as the carrier gas to achieve low operating cost without sacrificing the efficiency.

## 2. Experimental Methods

The nicotine standard was purchased from Restek (cat no. 34085). The E-liquid sample was obtained from a customer.

The calibration standards were prepared by diluting original Restek standard (1000 ppm in methanol) to 10, 50, 100 and 500 ppm using methanol. The e-liquid sample was prepared by taking a 100 µL aliquot of the sample material and adding 1000 µL of methanol. This yields an 11-fold dilution of the original sample.

GC-2030 FID with
AOC-20i Autoinjector

**Analytical Conditions**

| | |
|---|---|
| **Column** | SH-Rxi-624Sil MS 30m x 0.32mm x1.8µm (Shimadzu part no. 227-36077-01) |
| **GC oven temp** | 35°C hold for 1 min, ramp at 11°C /min to 250°C hold for 4 min |
| **SPL** | 250°C; Hydrogen carrier gas, constant inlet pressure at 39.7kPa, split ratio = 10 |
| **FID** | 250°C; Hydrogen flow rate 32mL/min; Air flow rate 200mL/min; Nitrogen makeup gas flow rate 24mL/min. |

Data were acquired and analyzed using LabSolutions software.

## 3. Results and Discussion

A five-point calibration curve was generated using the nicotine standards in methanol (as described in Experimental Methods) and was fitted to linear regression (Figure 1).

To address repeatability, each calibration level was run in triplicates and the *% RSD* was found to be less than 2 for all concentrations (see table listed under the calibration curve).

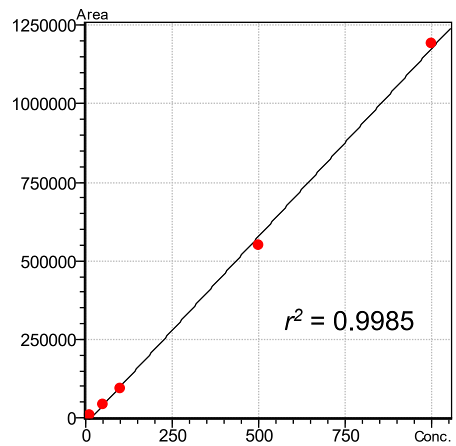


**Figure 1**: Five point calibration curve for nicotine

| Conc.(ppm) | Mean Area | %RSD |
|---|---|---|
| 10 | 8493 | 1.234312 |
| 50 | 43970 | 1.801773 |
| 100 | 92576 | 1.270758 |
| 500 | 548492 | 1.460263 |
| 1000 | 1192743 | 0.632143 |

To address potential carryover, a blank was run after the highest calibration standards. As shown in Figure 2, carryover was not observed.

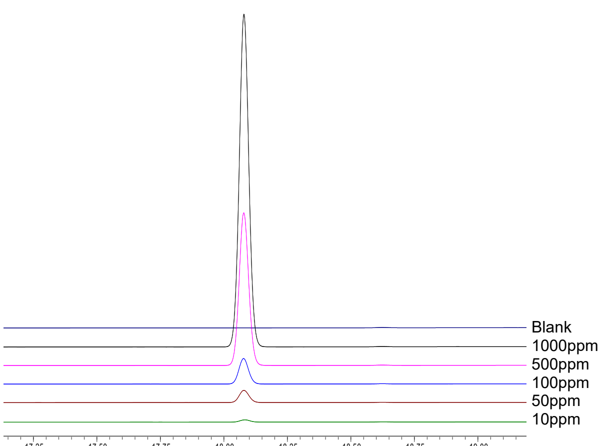


**Figure 2**: Chromatograms of nicotine standards. The blank was run after the 1000ppm triplicate runs

The e-cigarette liquid sample (prepared as described in the Experimental Methods) was run in triplicates on both columns. The concentration of nicotine was determined and reported in Table 1.

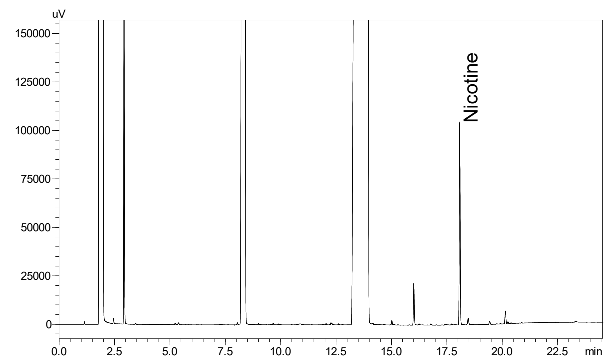


**Figure 3**: Example chromatogram of the e-liquid sample. The other major peaks are likely to be methanol (solvent), propylene glycol and glycerin

| Sample Injection | Peak Area | Concentration in diluted sample (ppm) |
|---|---|---|
| 1 | 287,711 | 257.250 |
| 2 | 279,645 | 250.516 |
| 3 | 275,962 | 247.442 |
| Average | 278587 | 251.736 |
| *%RSD* | 2.14 | 1.99 |
| Nicotine concentration in the original sample | | 2769 (±55) ppm |

**Table 1**: Measured concentrations of nicotine.

The concentration of nicotine in the original sample was calculated by multiplying the average concentration in the diluted sample by 11 (dilution factor, see Experimental Methods for detail).

## 4. Conclusions

In this report, nicotine level in an e-cigarette liquid sample was analyzed by the Shimadzu GC-2030 with FID and AOC-20i autoinjector. Calibration was carried out from 10ppm to 500ppm with good linearity. No carryover was observed with this setup and method.

As shown in the table of results above, the calculated concentration of nicotine in the e-liquid is 2769 ± 55 ppm or 2.769 ± 0.055 mg/mL, agreeing with the label-claimed concentration of 3 mg/mL. The *%RSD* is less than 2 for three injections, demonstrating both accuracy and precision.

Furthermore, this method can be adapted easily to assay other components (such as impurities and flavor) of the e-liquid. This setup can run high concentrations of the e-liquid samples with minimal carry-over, which will allow better determination of any impurities with greatly reduced instrument downtime.

For the purpose of this project, methanol was used to dilute the e-liquid sample for analysis. On the other hand, methanol could be a potential impurity in e-liquid samples. Hence, moving forward, other GC compatible solvents such as chloroform should be considered as diluent.

Hydrogen was used as the carrier gas instead of helium in this setup, providing additional advantages. Hydrogen carrier gas minimizes operating costs while providing good resolution, wide range linearity and excellent repeatability. The wide range linearity enables analysis of the more concentrated samples (less split required), making it possible to identify the less concentrated impurities within the solution in addition to nicotine. And hydrogen gas can be easily generated in the lab with a generator. Furthermore, with an integrated hydrogen sensor option for the GC-2030, the instrument can be used with hydrogen carrier gas routinely with minimal safety concerns for the operator.

Overall, the Shimadzu GC-2030 system, also featuring a modern touch screen, mobile connectivity and tool-free maintenance and column connections, provides users with an easy and reliable setup for your routine analysis needs.

First Edition: December 2018

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