Fully Automated Analysis of E-Cigarette Juice

Chiew Mei Chong[1], Hans-Joachim Huebschmann[1], Nick Zhang[2]

[1]CTC Analytics Asia Ple. Ltd., 117610 Singapore

[2]Guangzhou Ingenious Laboratory Technology Co. Ltd., Guangzhou, 510000 China

Contact: CCHONG@CTC.CH

PAL SYSTEM Ingenious sample handling

Overview

Purpose: Provide an automated method for comprehensive analysis for compounds of a very wide concentration range.

Methods: Static headspace and HS-SPME Arrow are applied.

Results: Static headspace for the main volatiles and HS-SPME extraction are combined in one workflow for comprehensive analysis of main and trace components.

Introduction

E-Cigarette, which is termed as Electronic Nicotine Delivery Systems (ENDS) by the World Health Organization (WHO), can be available in multiple forms either as a cigarette like design or a cartridge like handheld device. The device is filled with nicotine-containing liquid, which is commonly known as E-Juice. [1,4] Since the first E-Cigarette was developed in 2003[1], the market of E-Cigarette has been expending exponentially. By January 2014, according to S.H.Zhu, there were a total of 466 brands of E-Cigarette available for online purchase.[2] The IMARC group reported that the global E-cigarette market has reached US$ 11.5 billion in 2018 and is expected to continue growing to reach US$ 24.2 billion by 2024.

The E-Cigarette has gained popularity because some manufacturers marketed it as a healthier option than the tobacco smoking. The manufacturers claimed that the E-Cigarette does not emit by-products of the incomplete combustion and nitrosamines which are produced in tobacco. The E-Cigarette juice main ingredients are mostly nicotine, propylene glycol, glycerin, flavor and water. The E-Cigarette usage is the most popular among the adolescents and young adults due to the big variety of flavors available in E-Cigarette.[6] A total of 7764 unique E-Cigarette flavors were available from the online platforms alone in 2014.

Analytical Strategy

Sample Description

Analytical quality control is one of the important ways to determine the E-Cigarette juice quality. Although the major components and flavors of E-Cigarette juice can be analyzed by Gas Chromatography, the E-Cigarette juice samples could not be directly injected into the GC-MS. The sample is too viscous and concentrated, which can easily contaminate the GC injector and require constant maintenance in the GC-MS system. The highly viscous samples could also cause the formation of air bubble in the injection syringes. Therefore, the E-Cigarette juice samples need to be diluted with a compatible solvent prior to the analyses.

Figure 1: The highly viscous, yellow E-Cigarette juice

An unknown E-Cigarette juice, or commonly called as "E-Juice" was received for analysis as shown in Figure 1 below. The E-Cigarette juice was a highly viscous yellow fluid with strong aroma.

The full analysis of E-Cigarette juice was separated into two parts:

First Part

was using the PAL RTC System to automatically dilute the E-Cigarette juice with isopropanol and inject into the GC-MS to determine the major components in the juice.

Second Part

on focusing on the determination of the E-Cigarette flavor profile. To extract the volatile flavoring components released from the E-Cigarette juice, SPME Arrow, which is a more robust form of SPME, was used. The SPME Arrow, consists of a larger sorption surface than the conventional SPME fibers. The SPME Arrow can extract with 10 times higher sensitivity than conventional SPME. Therefore, even the flavor compounds with very low aroma thresholds can be included into the detection. The SPME Arrow with DVB/PDMS adsorption phase was used to extract the E-Cigarette flavor because B. Savareear et. al. found that the SPME with DVP/PDMS phase could extract most of the components from tobacco aerosol.

Instrument Configuration

A total solution of E-Cigarette juice analysis which includes the major components analysis and profiling of the low-level volatile flavor compounds, can be prepared automatically by using the CTC PAL RTC System.

With only one setup, as illustrated in Figure 2, the PAL RTC System can perform the dilution, sample clean up, SPME Arrow extraction and GC injection.

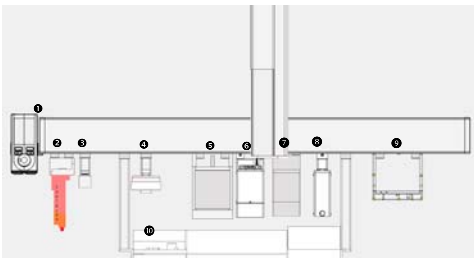


Figure 2. PAL RTC System setup with modules for the fully automated analyses of major components and flavor profile:

PAL RTC System			
1.	PAL RTC 120	11.	Rack VT54
2.	Park Station	12.	Rack VT15
3.	Standard Wash Module	13.	Liquid Tool, D7
4.	Solvent Module	14.	Liquid Tool, D8
5.	Agitator	15.	Arrow Tool Holder
6.	Heatex Stirrer	16.	DVB/PDMS Arrow
7.	Vortex Mixer	17.	10uL Liquid syringe
8.	Arrow Conditioning Module	18.	1000uL liquid syringe
9.	Tray Holder		
10.	Arrow Injector		
GC-MS System used:			
1.	Agilent GC7890-MS7000	2.	Rxi-624 (30m x 0.25mm x 1.40µm)

Upon completing sample preparation, the samples were directly injected by the PAL System into the GC-MS for qualitative and quantitative analyses. This allows the analysis of E-Cigarette juice to be continuously running and producing data daily without human intervention.

Methods

Part A: Determination of Major Components in E-Cigarette Juice - Sample Preparation

The determination of the E-Cigarette juice major components was based on the "dilute and inject" principle. The E-Cigarette juice was diluted with isopropanol to the required concentration, then injected into the GC-MS system for analysis.

Manually weighed 0.100g (±0.015g) of the E-Cigarette juice into a 2mL vials. Then, screwed the vial with magnetic cap and placed the vial to the sample rack VT54 on the PAL RTC System.

The sample vial containing the E-Cigarette juice underwent automatic dilution with the workflow in Fig. 3, generated by the PAL Method Composer software to dilute the E-Cigarette juice and subsequently inject into the GC-MS for analysis.

Figure 3. Automated workflow generated by PAL Method Composer software .

PAL System Parameter

The critical parameters to dilute the E-Cigarette juice and inject into GC for analysis is shown in Table 2 below.

Table 2. PAL RTC System and GC-MS parameter of the E-Cigarette juice dilution and injection

PAL RTC System	
Dilution	
Tool	D8/57 Liquid Tool
Source	Isopropanol
Volume	Depends on user setting
Vortexing Speed	1500rpm
Vortexing Time	60s
Liquid Injection	
Tool	D7/57 Liquid Tool
Sample Volume	1µL
Bottom Sense	On
Height From Bottom of Sample Vial	1mm
Pre and Post Wash	3 times with isopropanol
GC	
Inlet Temperature	250°C
Inlet Mode	Split
Split Ratio	200:1
Flow	2mL/min
Pressure	120.66kPa
Oven Temperature	50°C (1min) → 35°C/min → 260°C (1min)
MSD Transfer Line Temperature	250°C
MS	
Scan m/z	29 - 350
Scan Time	300ms
Source Temperature	230°C

Figure 4. TIC of the major components from the 10 times diluted E-Cigarette juice

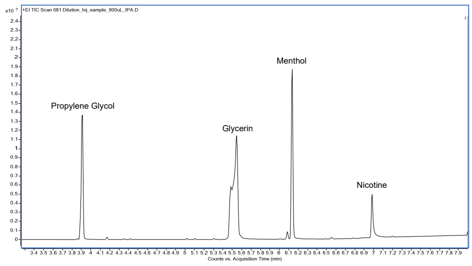


Figure 5. TIC from the SPME Arrow extraction of the volatile components, peaks of the automatically identified

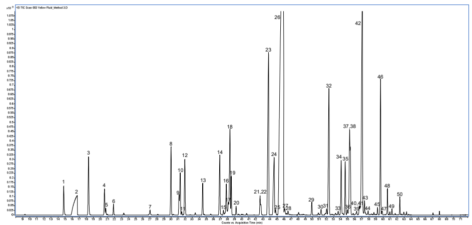
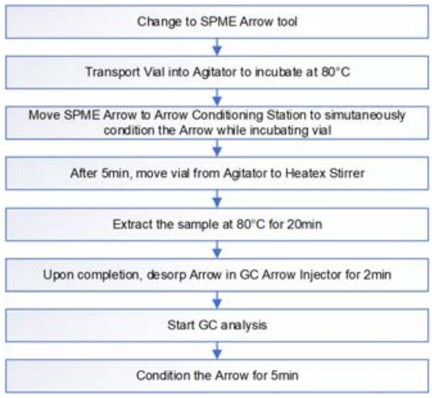


Figure 6. Workflow of the automated headspace SPME Arrow extraction



Results

Part A: The 0.1g E-Cigarette juice was diluted 10 times to the final volume of 1mL and injected into GC-MS. The solvent was transferred from the Solvent Module into the sample vial and followed by vortex mixing before injection of the final diluted sample into the GC-MS, as shown in the Figure 4. Major compounds of the E-Cigarette such as propylene glycol and glycerin, nicotine and flavor can be detected

Part B: With the SPME Arrow extraction workflow shown in Figure 6, the volatile compounds of E-Cigaratte juice were extracted from the headspace and analyzed by GC-MS. The chromatogram of the volatile profile from the E-Cigarette juice is displayed in Figure 5.

A total of 50 peaks were automatically detected with peak area more than 10,000,000 counts, even more compounds elute with smaller intensity. The peaks were identified with the NIST14 mass spectral library Almost all the peaks can be identified based on the library score of more than 80%. The full list of the detected compounds is available from the authors.

Most of the detected compounds belong to the family of esters which give the fruity aroma. Three of the commonly added tobacco flavour compounds were detected, e.g. eucalyptol, menthol and pulegone.

The compound quantitation achieved a precise calibration with an R^2 of better than 0.992 for the measured analytes

Conclusion

- 2 Methods for 1 sample on 1 PAL System.
- Samples are diluted automatically by the PAL System.
- SPME Arrow extracts automatically all flavor compounds.
- True walkaway automation, just put the sample on the rack.

For the full report please mail to CCHONG@CTC.CH.