The Determination of Flavors in E-Liquids Using Purge and Trap Concentration

Application Note

Food and Flavor

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Abstract

In the past five years, Vaping has become more and more prevalent. People are trying to quit smoking and vaping provides a somewhat healthier alternative. E-cigarettes are composed of a heating element and a small compartment for the vape solution. The vape liquid is heated and inhaled. E-liquid is composed of water, glycerin and propylene glycol. The liquid is then flavored with a large assortment of flavors and can be procured with or without Nicotine. This application note will examine the flavor components in the vape solution and how nicotine can affect the how the vape liquid tastes.

Introduction:

E-liquid can be composed of propylene glycol or vegetable glycerin alone or can be a combination of the two. Propylene glycol is less viscous and carries better flavor. It also acts as a preservative. However, it does produce less vapor and some people are allergic to it. Vegetable glycerin, on the other hand, is much denser, is super hypoallergenic and produces more vapor. The drawback to the vegetable glycerin is that it is not a preservative, so the vape liquid will not last as long.

Both glycerin and propylene glycol readily dissolve in water, so preparing the samples to test by purge and trap was relatively easy. Conversely, as the E-liquid emitted a strong odor of the added flavors, only a small amount of the solution could be used for analysis. The volatile flavor components of the vape liquid were examined by dissolving the solution in water and sampling the water using purge and trap.

Experimental:

The Evolution purge and trap concentrator was configured with a Vocarb 3000 trap. The trap was conditioned at 260°C for one hour. The Centurion WS syringe autosampler was set in water mode in order to run 5 ml samples. Next, samples were prepared using 10ul of vape solution dissolved in 100mls of de-ionized water. The parameters for the autosampler/purge and trap concentrator and the Gas Chromatograph/Mass Spectrometer (GC/MS) are outlined in Tables 1 and 2.

Purge and Trap Concentrator	EST Analytical Evolution
Trap Type	Vocarb 3000
Valve Oven Temp.	130ºC
Transfer Line Temp.	130ºC
Trap Temp.	35ºC
Moisture Reduction Trap (MoRT) Temp.	39ºC
Purge Time	11 min
Purge Flow	40mL/min
Dry Purge Temp.	Off
Dry Purge Flow	40mL/min
Dry Purge Time	1.0 min
Desorb Pressure Control	On
Desorb Pressure	5psi
Desorb Time	0.5 min
Desorb Preheat Delay	5 sec
Desorb Temp.	250ºC
Moisture Reduction Trap (MoRT) Bake Temp.	210ºC
Bake Temp	260ºC
Sparge Vessel Bake Temp.	110ºC
Bake Time	6 min
Bake Flow	85mL/min
Purge and Trap Auto-Sampler	**EST Analytical Centurion WS**
Sample Type	Water
Sample Fill Mode	Syringe
Sample Volume	5mL
Sample Prime Time	7 sec
Loop Equilibration Time	5 sec
Sample Transfer Time	5 sec
Syringe Rinse	On/20mL
Number of Syringe Rinses	2
Sample Loop Rinse	On/10 sec
Sample Loop Sweep Time	5 sec
Number of Sparge Rinses	Syringe/2
Rinse Volume	5mL
Rinse Transfer Time	10 sec
Rinse Drain Time	15 sec
Number of Foam Rinse Cycles	3
Water Heater Temp.	85°C
Internal Standard Vol.	0µl

Table 1: Evolution and Centurion WS Experimental Parameters

GC/MS	Parameters
Inlet	Split/Splitless
Inlet Temp.	220ºC
Inlet Head Pressure	7.774 psi
Mode	Split
Split Ratio	40:1
Column	Rxi-624Sil MS 30m x 0.25mm I.D. 1.4µm film thickness
Oven Temp. Program	45ºC hold for 1 min, ramp 15ºC/min to 220ºC, hold for 1.33 min, 14 min run time
Column Flow Rate	1mL/min
Gas	Helium
Total Flow	44mL/min
Source Temp.	230ºC
Quad Temp.	150ºC
MS Transfer Line Temp.	180ºC
Scan Range	m/z 35-300
Scans	5.2 scans/sec
Solvent Delay	0.7 min

Table 2: GC/MS Parameters

Assorted flavored E-liquids were procured for testing and analysis. Samples included five different flavors and one flavor with three levels of nicotine. Each flavor was tested four times in order to determine flavor compound composition of the sample and reproducibility (%RSD) of the results. Figures 1 through 5 display the chromatograms of each flavor of vape solution. Tables 3 through 6 are data summaries of the compound compositions of each sample and the resulting %RSD of each compound. Finally, Table 7 is a comparison of the flavor compound composition when the nicotine level is different while Figure 6 is a graph of those flavors.

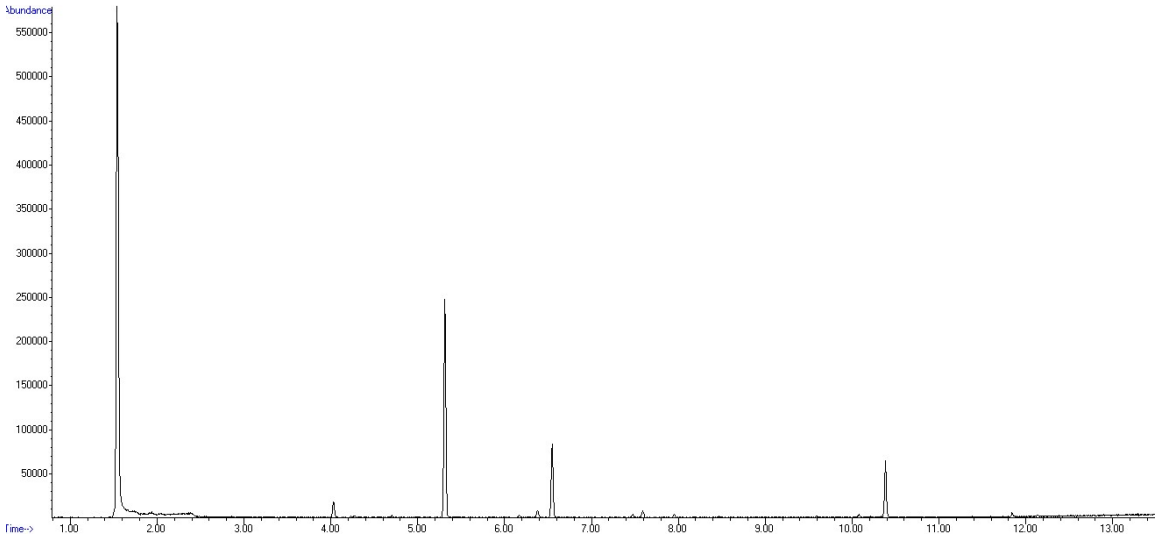

Figure 1: Cookie Vape Chromatogram

Cookie Flavor			
RT	**Compound**	**%RSD**	**% of Total**
4.031	ethyl acetate	5.83	4.49
5.311	propanoic acid ethyl ester	5.76	59.46
6.546	butanoic acid ethyl ester	5.62	19.88
10.385	butanoic acid, 3-methyl, 3-methylbutyl ester	5.35	16.17

Table 3: Cookie Flavor Composition Summary

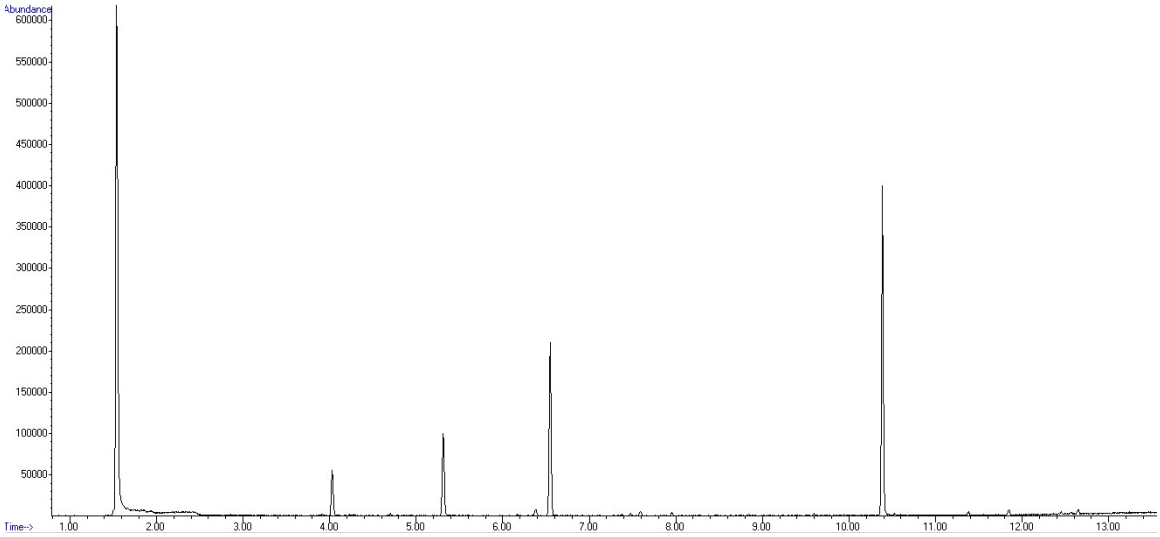

Figure 2: Vanilla Custard Vape Chromatogram

Vanilla Custard Tobacco Flavor			
RT	**Compound**	**%RSD**	**% of Total**
4.031	ethyl acetate	10.29	7.08
5.314	propanoic acid ethyl ester	10.01	12.86
6.549	butanoic acid ethyl ester	8.14	27.10
10.385	butanoic acid, 3-methyl, 3-methylbutyl ester	4.04	50.99
11.378	2-methoxy-5-methylphenol	4.48	0.62

Table 4: Vanilla Custard Flavor Composition Summary

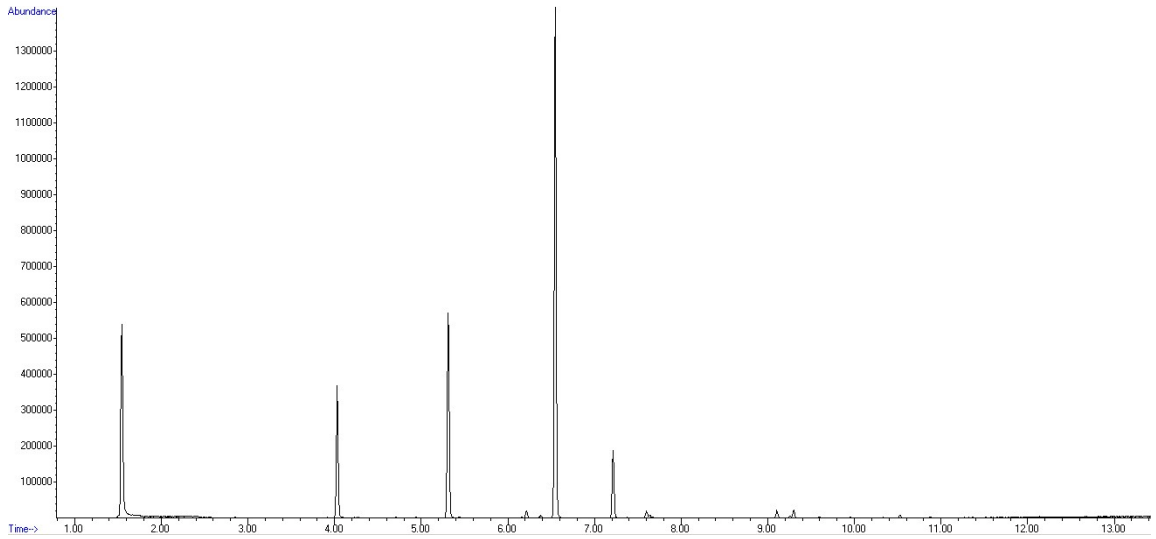

Figure 3: Strawberry Cereal Vape Chromatogram

Strawberry Cereal Flavor			
RT	**Compound**	**%RSD**	**% of Total**
4.031	ethyl acetate	6.34	14.08
5.311	propanoic acid ethyl ester	5.73	21.81
6.215	acetic acid, 2-methylpropyl ester	5.59	0.76
6.546	butanoic acid ethyl ester	5.25	53.36
7.215	butanoic acid, 2-methyl, ethyl ester	4.77	7.06
7.600	1-butanol, 3-methyl, acetate	13.17	0.85
9.105	hexanoic acid, ethyl ester	3.81	0.76
9.256	4-hexen-1-ol, acetate (Z)	7.07	0.21
9.301	acetic acid, hexyl ester	5.84	0.78
10.523	beta-pinene	5.13	0.32

Table 5: Strawberry Cereal Flavor Composition Summary

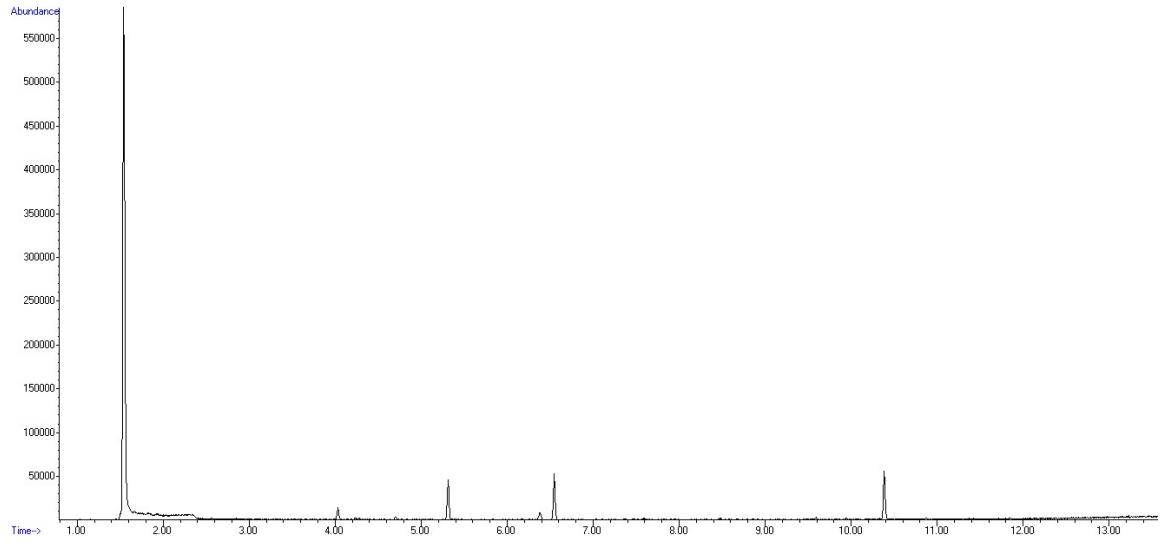

Figure 4: Tobacco Vape Chromatogram

Tobacco Flavor			
RT	**Compound**	**%RSD**	**% of Total**
4.031	ethyl acetate	2.65	8.01
5.311	propanoic acid ethyl ester	2.07	26.09
6.546	butanoic acid ethyl ester	2.50	30.76
10.385	butanoic acid, 3-methyl, 3-methylbutylester	2.72	35.14

Table 6: Tobacco Flavor Composition Summary

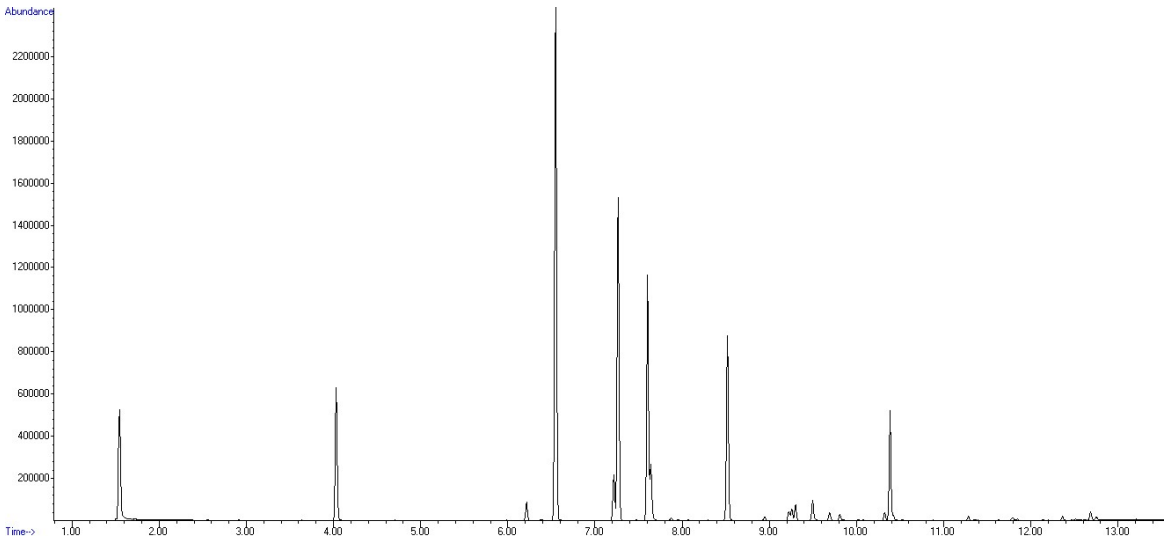

Figure 5: Mystery Vape with 3mg Nicotine Chromatogram

Mystery Flavor with Different Levels of Nicotine				
RT	Compound	6mg Compound Response	3mg Compound Response	0mg Compound Response
4.031	ethyl acetate	1038339	1028996	1910275
5.311	formic acid, propyl ester	452	2502	29890
6.215	acetic acid, 2-methylpropyl ester	150494	148129	318678
6.546	butanoic acid ethyl ester	4066846	3971522	6190253
7.215	butanoic acid, 2-methyl, ethyl ester	382841	391608	788237
7.266	butanoic acid, 3-methyl, ethyl ester	2637681	2637764	2633270
7.604	1-butanol, 3-methyl, acetate	1984070	1989384	2016274
7.639	1-butanol, 2-methyl, acetate	431895	445606	705324
7.870	pentanoic acid, ethyl ester	21175	21044	21312
8.520	butanoic acid, 3-methyl-, propyl ester	1540380	1550821	1460154
8.948	beta-pinene	24905	30119	6539
9.224	pentanoic acid, 2-methylpropyl ester	81832	82305	72041
9.259	3-hexen-1-ol, acetate (Z)	88418	90386	94271
9.301	acetic acid, hexyl ester	124112	130510	246421
9.497	d-limonene	143603	177911	108932
9.694	pentanoic acid, butyl ester	68183	68603	63944
9.813	butanoic acid, 3-methylbutyl ester	49882	51630	74764
10.025	5-heptenal, 2,6-dimethyl	1430	8410	3322
10.324	3-hexen-1-ol, propanoate (Z)	201355	62945	281257
10.385	butanoic acid, 3-methyl, 3-methylbutyl ester	931824	927964	1126356
11.285	butanoic acid, 3-hexenyl ester, (Z)	112532	38767	160149
11.353	acetic acid, phenylmethyl ester	7306	7112	18035
11.793	butanoic acid, 2-methyl-, hexyl ester	48899	33428	54239
12.365	isopulegyl acetate	42616	35727	45382

Table 7: Mystery Flavor Composition Comparison

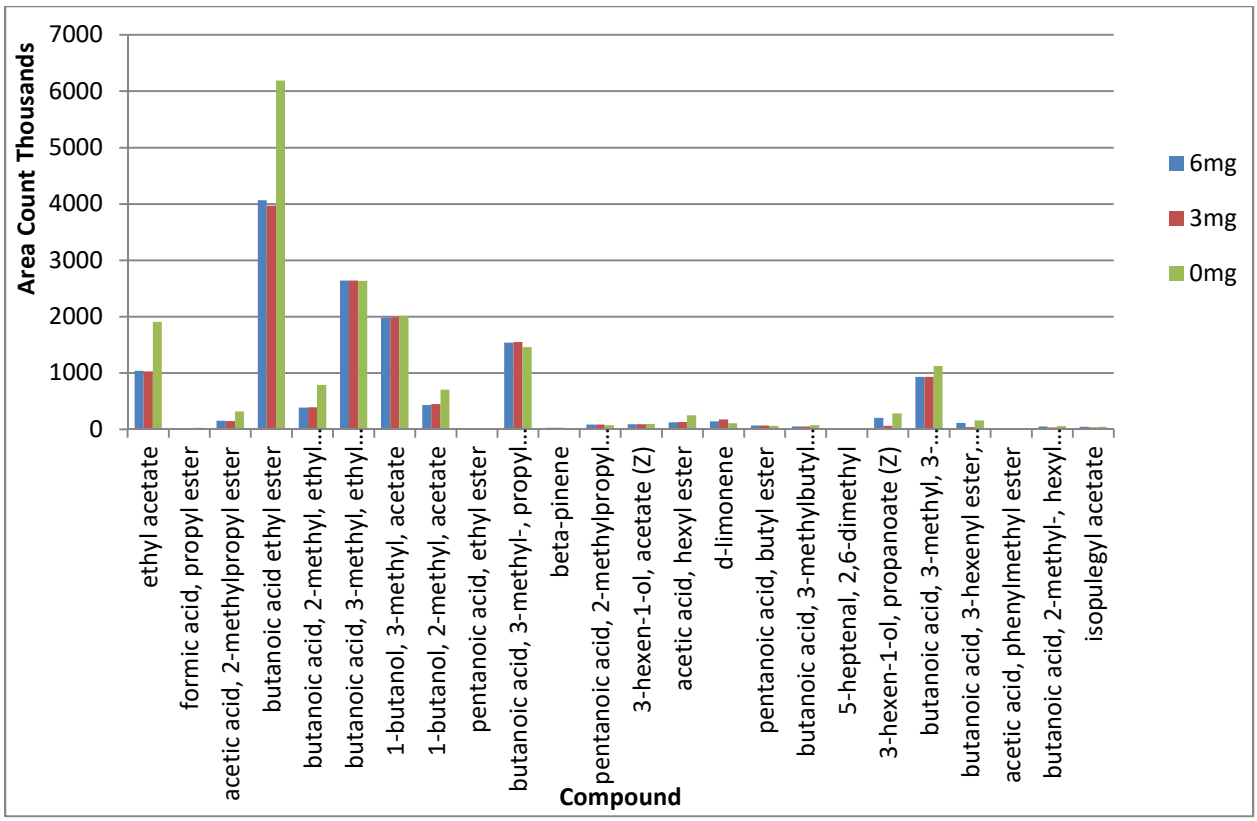

Figure 6: Mystery Flavor Composition Comparison Graphic

Conclusions:

Since vape solution readily dissolves into water, purge and trap sampling proved to be an excellent technique for the determination of the volatile compounds in the E-liquids. Purge and trap sampling is an exhaustive sampling technique, so the results were very reproducible and provided the sensitivity to determine the lower level compounds in the solution. Furthermore, it was found that nicotine addition to the solution has an effect some of the compounds in the solution. So, the taste of the vape liquid is affected by the amount nicotine in the mix.

References:

Chapter 6, E Juice and You, "onVaping", https://onvaping.com/guide/beginners-guide-to-vaping/ch-6-e-juice-and-you/, 2

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