Flavor Compound Abundances in Orange Juice Comparison Using Purge and Trap

Application Note

Food and Flavor

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Abstract

Before the advent of orange juice from concentrate, people would fresh squeeze oranges for their juice. Concentrate helped make orange juice easier to make and increased its popularity. Following concentrate, manufacturers used flash pasteurization and created ready to drink orange juice and again sales increased. Due to disease in the orange groves and the possibility of the crops getting destroyed by frost, the production of orange juice has gotten more expensive. The increase in production costs creates competition within the orange juice industry to create an orange juice that the public will enjoy and increase their consumption. This application will look at the flavor compound abundances of three different brands of orange juice using purge and trap concentration.

Introduction:

Pasteurization is the first step in the creation of commercial orange juice. This is required in order to kill any harmful bacteria in the juice before it is sold. After the juice is pasteurized, it can be sold. However, since different orange varieties are in season at different times, most juice is squeezed and stored. The storage times vary and with storage there can be other issues. One of these issues is oxidation. Thus, many companies remove the oxygen from the juice before they store it. Between pasteurization and oxygen removal, the juice will lose some of its flavor. This is where juice manufacturers can differentiate themselves.

Flavor compounds and amounts can make a substantial difference in the overall taste, acidity and odor of the juice. So, to create a juice that the public wants to buy, it is essential that the analysis of the flavor compounds and the percentage of these flavors be accounted for. Purge and trap is an excellent technique for the extraction of volatile flavor compounds within a liquid matrix. This is an exhaustive technique and generates reproducible results. This application will compare the reproducibility and ratios of flavor compounds in orange juice using the EST Analytical Evolution purge and trap concentrator.

Experimental:

The EST Analytical Evolution purge and trap concentrator was set up with a Vocarb™ 3000 trap while the Centurion WS autosampler was set to run in soil mode. The sampling system was configured to an Agilent 7890A Gas Chromatograph (GC) and 5975C inert XL Mass Spectrometer (MS) for separation and analysis. As the compounds of interest for this analysis were volatile, a Restek Rxi® 624 Sil MS 30m X 0.25mm X 1.4µm column was affixed in the GC. Sampling and analysis parameters were optimized and are listed in Tables 1 and 2.

Purge and Trap Concentrator	EST Encon Evolution
Trap Type	Vocarb 3000
Valve Oven Temp.	150ºC
Transfer Line Temp.	150ºC
Trap Temp.	35ºC
Moisture Reduction Trap (MoRT) Temp.	39ºC
Purge Time	11 min
Purge Flow	40mL/min
Dry Purge Temp.	ambient
Dry Purge Flow	40mL/min
Dry Purge Time	1.0 min
Desorb Pressure Control	On
Desorb Pressure	6psi
Desorb Time	0.5 min
Desorb Preheat Delay	5 sec.
Desorb Temp.	260ºC
Moisture Reduction Trap (MoRT) Bake Temp.	210ºC
Bake Temp	270ºC
Sparge Vessel Bake Temp.	120ºC
Bake Time	8
Bake Flow	85mL/min
Purge and Trap Auto-Sampler	**EST Centurion WS**
Sample Type	Soil
Sample Fill Mode	Syringe
Sample Volume	NA
Sample Prime Time	NA
Loop Equilibration Time	NA
Sample Transfer Time	NA
Syringe Rinse	Off
Sample Loop Rinse	Off
Sample Loop Sweep Time	Off
Number of Sparge Rinses	NA
Rinse Volume	NA
Rinse Transfer Time	NA
Rinse Drain Time	NA
Number of Foam Rinse Cycles	NA
Water Heater Temp.	85°C
Sample Preheat Time	0.5min
Sample Preheat Temp.	40°C
Soil Valve Temp.	85°C
Soil Transfer Line Temp.	150°C
Minimizer Time	2 min

Table 1: Purge and Trap Experimental Parameters

GC/MS	Agilent 7890/5975
Inlet	Split/Splitless
Inlet Temp.	220ºC
Inlet Head Pressure	12.153 psi
Split	40:1
Liner	Restek Split liner, 1mm x 6.3 x 78.5
Column	Rxi-624 Sil MS 30m x 0.25mm I.D. 1.4µm film thickness
Oven Temp. Program	45ºC hold for 1.0 min, ramp 15ºC/min to 300ºC hold for 5.00 min, 23 min run time
Column Flow Rate	1.0ml/min.
Gas	Helium
Total Flow	44ml/min
Source Temp.	230ºC
Quad Temp.	150ºC
MS Transfer Line Temp.	180ºC
Solvent Delay	0.7 min
Scan Range	m/z 30-350
Scan Speed	4.4 scans/sec

Table 2: GC/MS Experimental Parameters

Three different orange juice brands were purchased at the local market. Ten milliliters of each orange juice was measured and placed in a 40ml vial. Each vial was then deposited in the Centurion WS autosampler to be purged in soil mode and concentrated on a Vocarb 3000 trap in the Evolution concentrator. Finally, the samples were in run in triplicate in order to ensure the reproducibility of the results. Agilent MassHunter Qualitative Analysis software was used for the determination of the flavor compounds in the juice samples. Table 3 displays the reproducibility of the sampling process whereas Table 4 lists a comparison of the flavor compound abundances. Figure 1 is a bar graph comparison of the compounds in the three juice brands while Figures 2, 3 and 4 are the respective chromatograms of the juice samples.

Name	Brand A Reproducibility (%RSD)	Brand B Reproducibility (%RSD)	Brand C Reproducibility (%RSD)
Acetaldehyde	2.10	5.15	2.67
Methanol	1.88	6.26	2.18
Ethanol	1.80	1.53	1.97
Acetone	5.20	6.42	2.69
Ethyl Acetate	3.37	4.37	2.49
Butanoic acid, ethyl ester	3.39	2.56	1.19
Hexanal	3.88	4.04	2.40
.alpha.-Pinene	5.27	3.31	2.48
.beta.-Myrcene	4.33	1.68	1.29
3-Carene	6.18	3.78	4.59
D-Limonene	1.23	1.28	1.72
Limonene	6.92	6.23	4.30
.beta.-Phellandrene	4.71	1.65	1.90
1,6-Octadien-3-ol, 3,7-dimethyl-	3.52	6.00	1.41
Acetic acid, octyl ester	3.61	5.88	0.79
Decanal	3.98	6.18	1.19

Table 3: Reproducibility of the Results

Name	Brand A Compound Response	Brand B Compound Response	Brand C Compound Response
Acetaldehyde	9016366	13731642	12078948
Methanol	3313483	8177045	7982705
Ethanol	101695407	153700906	164460474
Acetone	2538076	10835769	11240111
Ethyl Acetate	3592700	5972570	4067472
Butanoic acid, ethyl ester	49469704	59642493	64367651
Hexanal	7418248	21391191	13975335
.alpha.-Pinene	53387978	50103153	69260326
.beta.-Myrcene	96100518	177738299	202759884
3-Carene	6484328	11635144	14763403
D-Limonene	472403326	578440798	589468049
Limonene	83430644	82319236	90862218
.beta.-Phellandrene	17865392	24628051	27113920
1,6-Octadien-3-ol, 3,7-dimethyl-	41245933	23982142	19518611
Acetic acid, octyl ester	6371908	5131877	5677029
Decanal	22167719	12600309	8187098

Table 4: Flavor Compound Abundance Summary

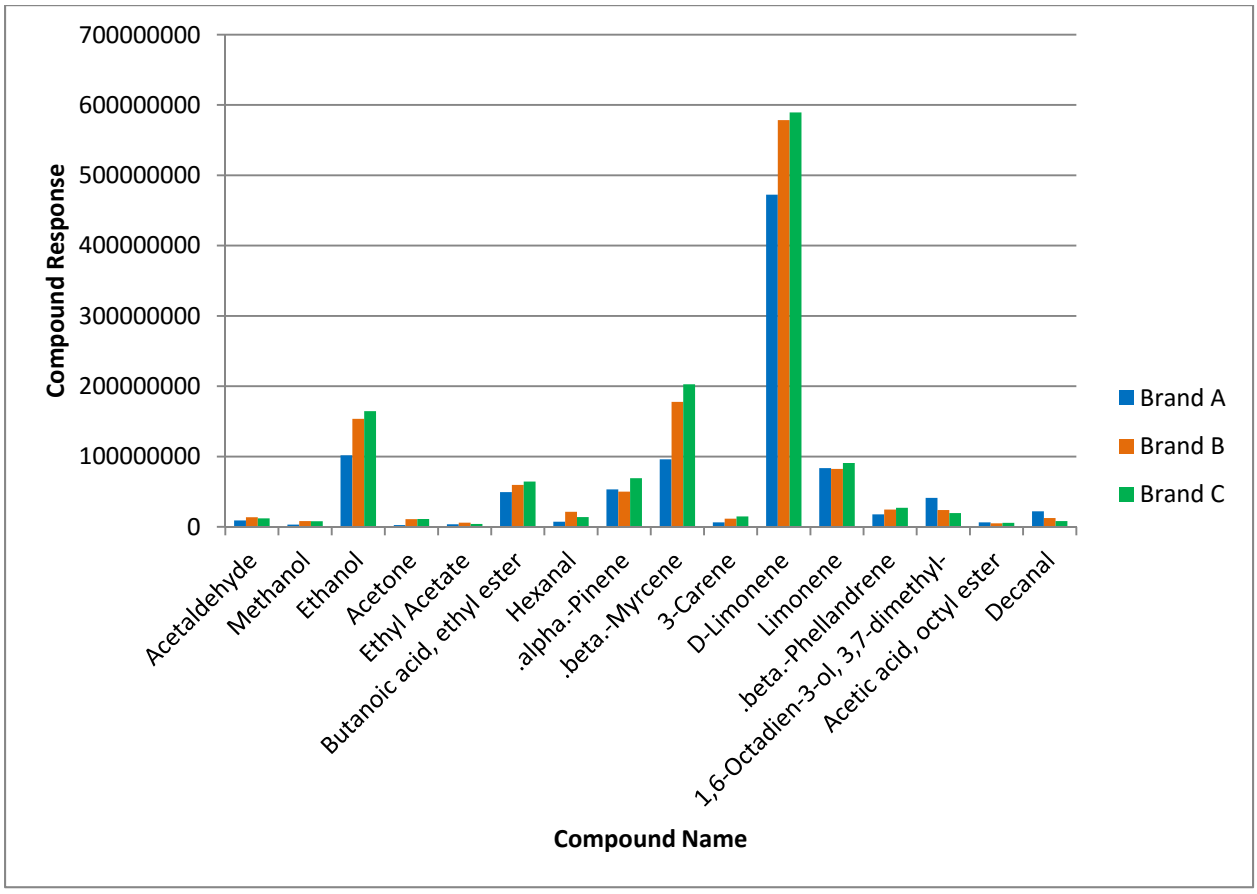

Figure 1: Bar Graph Compound Abundance Comparison

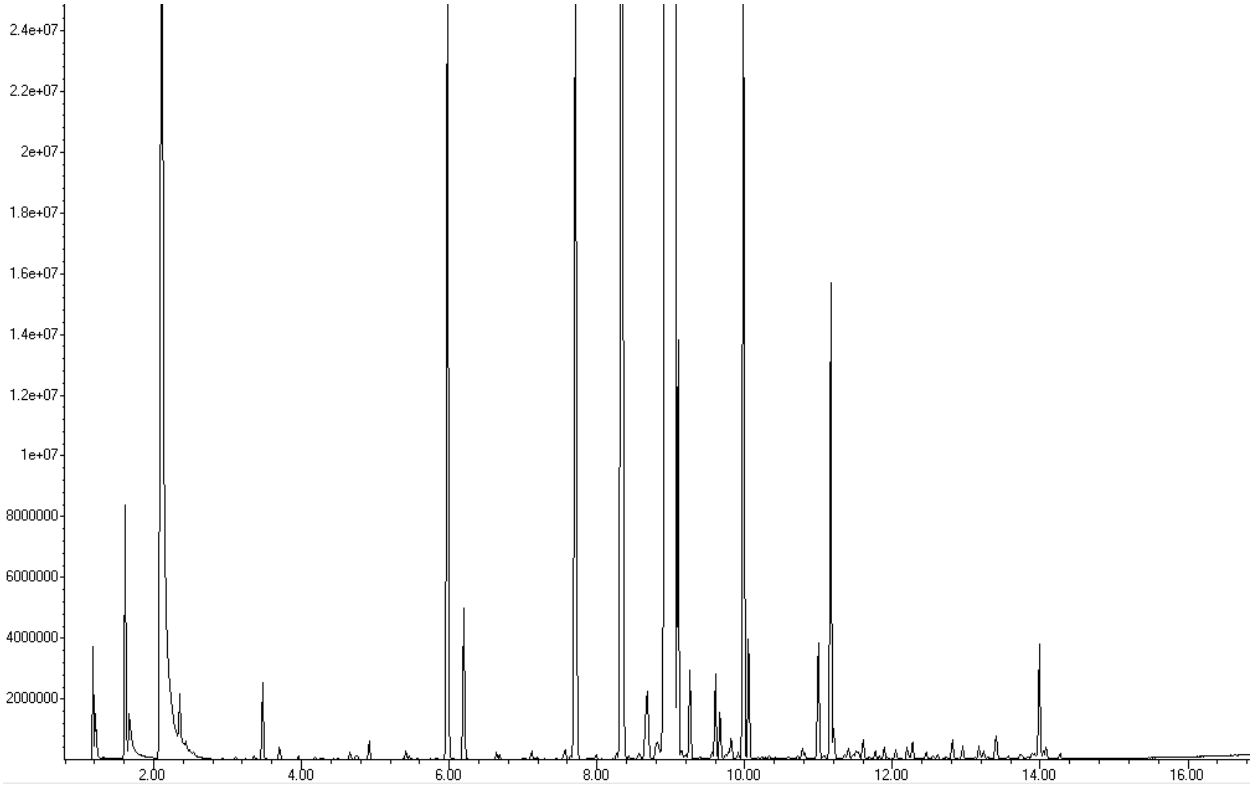

Figure 2: Brand A Chromatogram

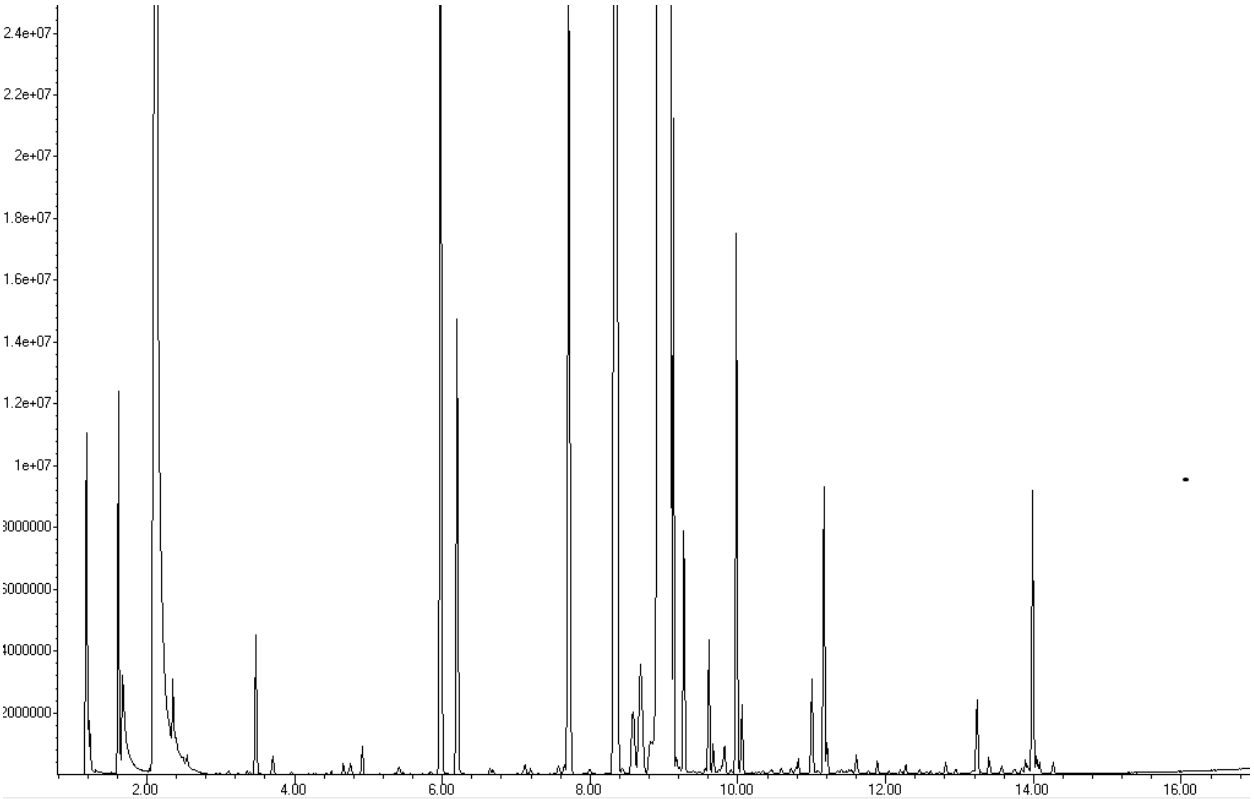

Figure 3: Brand B Chromatogram

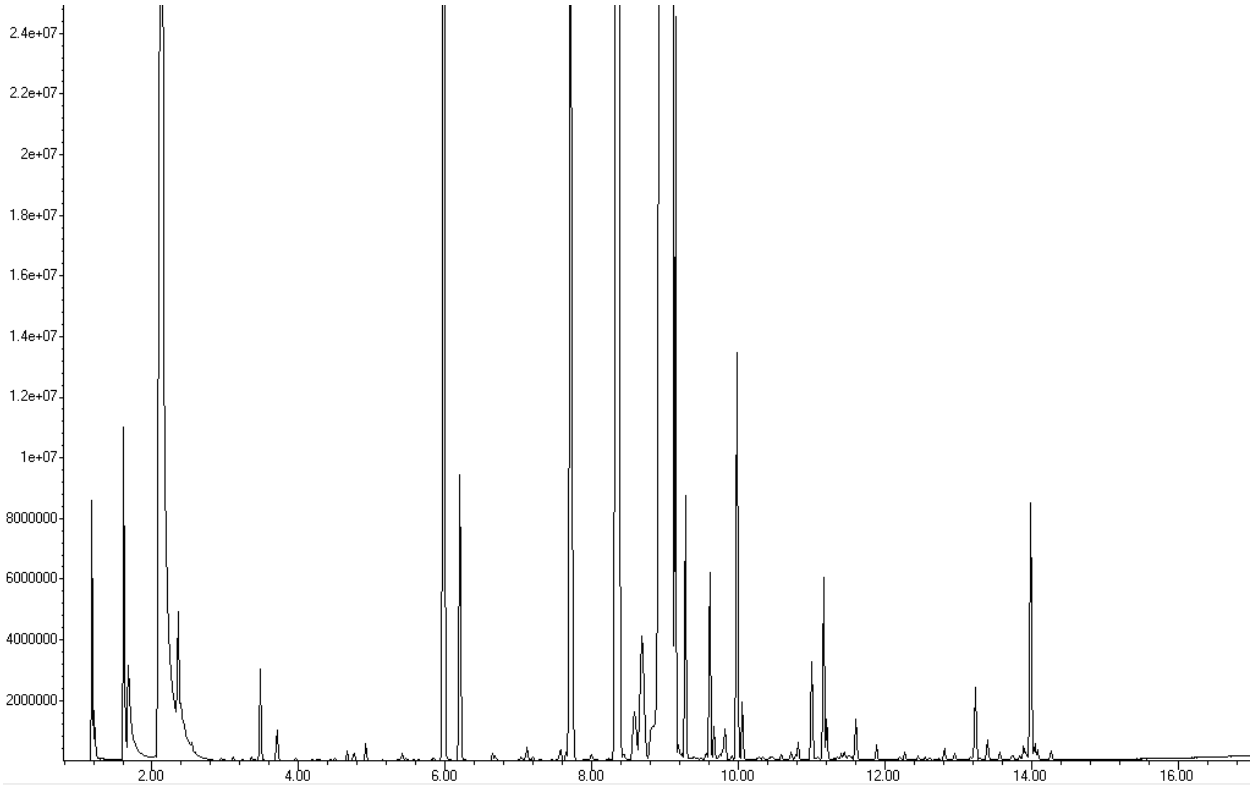

Figure 4: Brand C Chromatogram

Conclusions:

Using the EST Analytical Evolution purge and trap concentrator coupled to the Centurion WS autosampler to evaluate flavor compounds in orange juice samples proved to be a reliable sampling technique. The results were reproducible and the chromatography was excellent. The three different orange juice brands had very similar chromatograms; however upon further analysis many subtle differences in the abundances of the flavor compounds could be discerned. Traditionally, headspace and solid phase micro extraction sampling are used for this type of analysis. However, the purge and trap sampling technique used for this examination offered an excellent alternative for the determination of the volatile components in a juice matrix.

References:

1. Ferdman, Roberto A., "How America fell out of love with orange juice", *Quartz,* February 26, 2014, http://qz.com/176096/how-america-fell-out-of-love-with-orange-juice/, Web. September 20, 2016.
2. "Orange Juice", *Cook's Illustrated,* March 1, 2014m https://www.cookscountry.com/taste_tests/1496-orange-juice, Web. September 20, 2016.

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