Purge and Trap Extraction of Tea Flavor Components

Application Note

Food and Flavor

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Abstract

Solid Phase Micro Extraction (SPME) is the most prevalent sampling technique when examining the flavor composition of tea. However, SPME is not a comprehensive sampling technique, thus some of the compounds may not sample as well as others. Purge and Trap, on the other hand, is an exhaustive sampling technique and is commonly used for the detection of volatile components in water. This application note will examine how effective Purge and Trap is for the detection of flavor compounds in tea.

Introduction:

Purge and Trap (P&T) sampling is a universally accepted technique for the analysis of volatile compounds in water. The United States Environmental Protection Agency (USEPA), the World Health Organization (WHO) and the American Society for Testing and Materials (ASTM) have all published methods using P&T for water matrices. However, food and flavor manufacturers have yet to use this technique for the analysis of the volatile flavor components of their products. SPME has become the more accepted technique for flavor analysis.

Both SPME and P&T have their limitations. P&T is recommended for volatile analytes and does not work when compounds have higher boiling points. Still, P&T is a reliable technique that has the ability to purge out all of the volatile analytes of a system and detect very low concentrations of those analytes. On the other hand, SPME can be used for sampling a larger amount of semi-volatile compounds and fiber coating selection aids in better discrimination of analyte sampling. Yet, SPME is not an exhaustive sampling technique thus there are detection limitations to the technique. This application note will examine the analysis of white tea samples using P&T sampling.

Experimental:

The EST Analytical Evolution Purge and Trap 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 1.8um 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 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	10mL
Sample Prime Time	NA
Loop Equilibration Time	NA
Sample Transfer Time	NA
Syringe Rinse	Off
Sample Loop Rinse	Off
Sample Loop Sweep Time	NA
Number of Sparge Rinses	0
Rinse Volume	0mL
Rinse Transfer Time	NA
Rinse Drain Time	NA
Number of Foam Rinse Cycles	0
Water Heater Temp.	85°C
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 220ºC hold for 1.33 min, 14 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 35-300
Scan Speed	5.2 scans/sec

Table 2: GC/MS Experimental Parameters

For the sample preparation, white tea was obtained in tea bags. The tea bags were cut open so as to weigh out 2 grams of tea. Next, 120mls of reagent water was heated to 80ºC. Once the water reached temperature, a stir bar and the 2g of tea were added to the water. The tea was allowed to steep and stir for five minutes. After the five minutes the tea was filtered in order to ensure no tea leaves were in the sample. Finally, 10mls of the tea were added to each 40ml vial and placed on the tray of the Centurion WS for sampling and analysis. Four replicates of the steeped tea were tested in order to ensure repeatability of the analysis. Retention time, response and relative standard deviation results are presented in Table 3 while Figures 1 and 2 display a bar graph of the flavor analyte abundance and a labelled chromatogram of the purged tea sample.

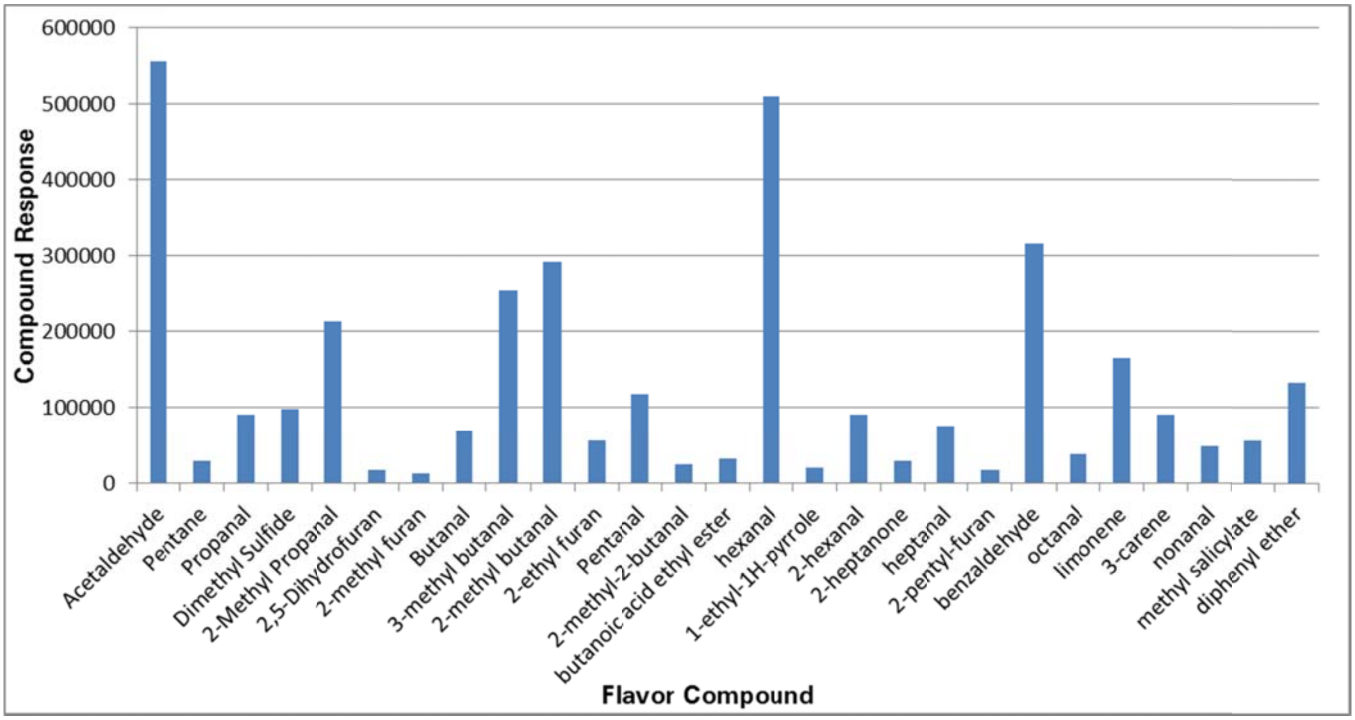
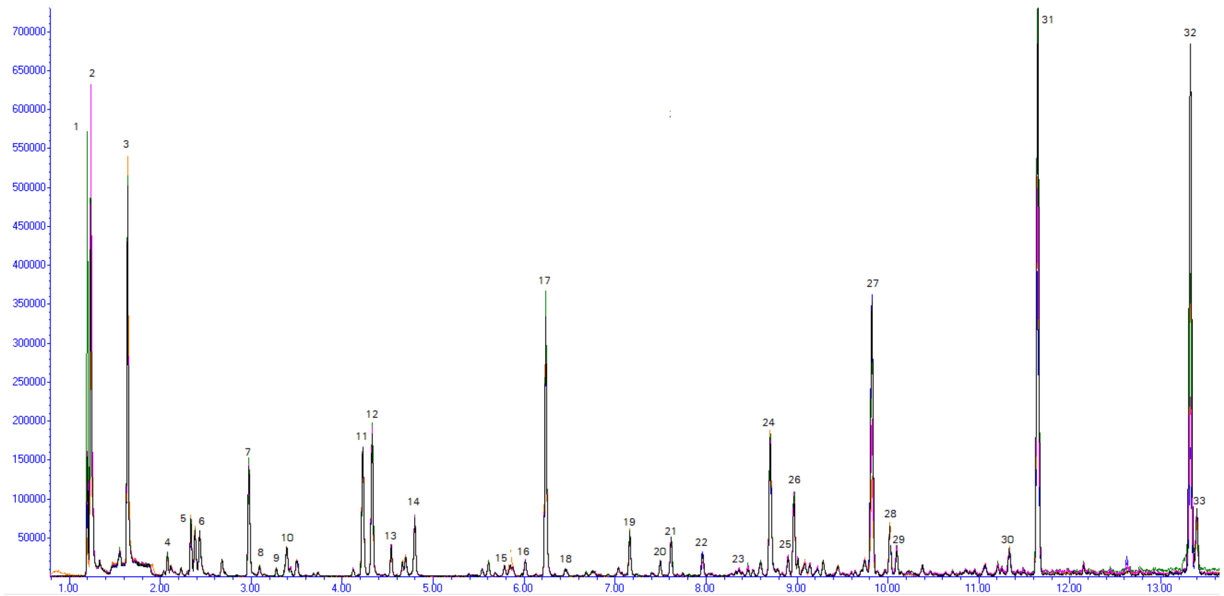


Figure 1: Bar Graph of Flavor Compound Results

Compound	Retention Time	Ave. Compound Response	Std. Dev.	%RSD
Acetaldehyde	1.64	555898	21951	3.95
Pentane	2.08	30073	2822	9.38
Propanal	2.34	89728	3940	4.39
Dimethyl Sulfide	2.43	97988	2536	2.59
2-Methyl Propanal	2.97	213544	4928	2.31
2,5-Dihydrofuran	3.09	17022	1377	8.09
2-methyl furan	3.28	12909	1143	8.86
Butanal	3.39	68267	1717	2.52
3-methyl butanal	4.23	254236	5903	2.32
2-methyl butanal	4.33	291340	5972	2.05
2-ethyl furan	4.54	57420	3007	5.24
Pentanal	4.80	118081	4169	3.53
2-methyl-2-butanal	5.78	24742	909	3.68
butanoic acid ethyl ester	6.02	32680	1458	4.46
hexanal	6.24	509834	12993	2.55
1-ethyl-1H-pyrrole	6.46	20534	544	2.65
2-hexanal	7.16	90358	4787	5.30
2-heptanone	7.50	29350	782	2.66
heptanal	7.61	75424	2915	3.87
2-pentyl-furan	8.46	16802	2217	13.19
benzaldehyde	8.71	315811	13569	4.30
octanal	8.90	38548	3686	9.56
limonene	8.97	164591	7768	4.72
3-carene	10.02	89556	6791	7.58
nonanal	10.10	49746	3983	8.01
methyl salicylate	11.34	56417	5648	10.01
diphenyl ether	13.39	133044	7796	5.86
Average		114159	4755	5.76

Table 3: Retention Time, Response and Repeatability of Purge and Trap Results



1	Carbon Dioxide
2	Carbon Dioxide
3	Acetaldehyde
4	Pentane
5	Propanal
6	Dimethyl Sulfide
7	2-Methyl Propanal
8	2,5-Dihydrofuran
9	2-methyl furan
10	Butanal
11	3-methyl butanal
12	2-methyl butanal
13	2-ethyl furan
14	Pentanal
15	2-methyl-2-butanal
16	butanoic acid ethyl ester
17	hexanal
18	1-ethyl-1H-pyrrole
19	2-hexanal
20	2-heptanone
21	heptanal
22	septum outgassing
23	2-pentyl-furan
24	benzaldehyde
25	octanal
26	limonene
27	septum outgassing
28	3-carene
29	nonanal
30	methyl salicylate
31	septum outgassing
32	septum outgassing
33	diphenyl ether

Figure 2: Labelled and Overlaid Chromatograms of White Tea to Show Reproducibility

Conclusions:

Using purge and trap to evaluate the flavor compounds of the tea samples was a reliable sampling technique. The findings were repeatable and the chromatography was excellent. The resulting flavor compounds were easily purged out of the tea matrix and as the technique is exhaustive, there was excellent detection of the flavor compounds in the system. This sampling technique established purge and trap to be an exceptional method for the determination of volatile analytes in tea.

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