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# Headspace Techniques for Flavor Characterization and Off-Odor Detection within the Food Industry

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## INTRODUCTION

More often than not the difference of odors and flavors in the same product is due to very small concentrations of specific chemicals. Potency of the odor is a function of both concentration of odor causing chemicals in the sample and their affinity to the sample matrix (partition coefficient). Any modification of matrix such as digestion, addition of solvents or salts may alter the composition of the headspace as compared with the original odor. It is desirable to characterize the odors/flavors over condensed matrices without any sample preparation or any typical analytical manipulations used to enhance the headspace concentration. The headspace conditions should not be altered from the conditions of human sensory testing.

## DISCUSSION

Analysis of a single aliquot of the headspace (regular headspace sampling) is often used to determine the profile of a consumer food product but is typically insufficient to provide the necessary sensitivity to determine the cause of a “bad” taste or odor. This type of trace detection work requires much larger volumes of headspace and an ability to pre-concentrate these contaminants prior to GC analysis. The Markelov HS9000 Static and Dynamic Headspace system utilizes a patented sampling needle which is comprised of two passages as shown in *Figure 1*.

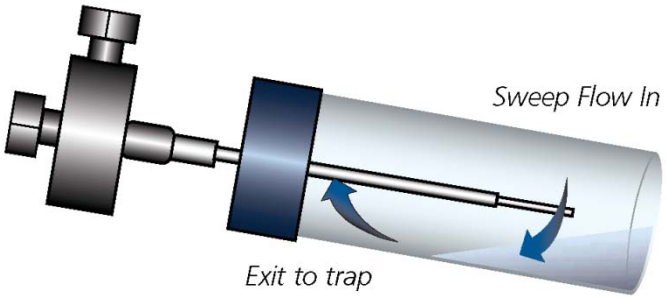


**FIGURE 1 Patented dynamic headspace dual needle design.**

## RESULTS

The flavor absorption of d-limonene and a-pinene by polymeric packaging materials, such as low-density polyethylene has been extensively investigated (K.S.M., Sheug, S. Min, and S. K. Sastry 2004). A small loss of flavor compounds will lead to serious changes in flavor quality (K.S.M., Sheug, S. Min, and S. K. Sastry 2004). These two key favor compounds were examined in this study. The challenge is that d-limonene appears in high part per million levels and a-pinene can be in low parts per billion levels see *Figure 2*. The instrument operating conditions can be seen in *Tables 1&2*

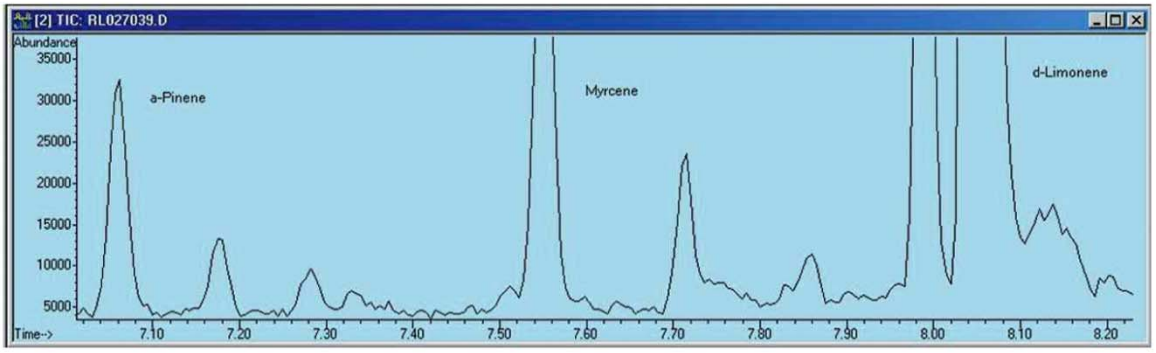


**FIGURE 2 Orange juice carton NT**

A container of orange juice obtained from the same distributor as in the research (K.S.M., Sheug, S. Min, and S. K. Sastry 2004) was used to examine 2 techniques. The first technique was the traditional loop fill and inject generally used for part per million levels of analysis. The second technique was achieved by simply switching the method in the software to the new dynamic sweep and trap technique that allows parts per billion levels of detection. A sealed sample was heated and mixed to reach equilibrium.

The sample vial was then placed onto the dual needle (a needle within a needle). Inert gas flows at a prescribed rate for a programmed period of time through one passage of the needle, displacing the headspace in the vial. The analytes were carried out of the vial through the second passage of the needle and concentrated onto a cool adsorbent trap. See Figure 1 The adsorbent trap is then heated and back-flushed with the GC carrier gas transporting the analytes to the GC column.

## TABLE 1
## Instrument Operating Conditions

| | |
|---|---|
| GC Cycle Time | 25 |
| Constant Heat Mode | enable |
| Vial Type | 20 |
| Pressure Check | cont |
| Sample Vial Temp | 45 |
| Sample Equilibration Time | 15 |
| Mixing | On |
| Mixing Speed | med |
| Post Mix Stabilization Time | 10 |
| Line Temp | 155 |
| Valve / Loop Temp | 150 |
| Standby Flow | On 40 |
| Economy Mode | program |
| Sample Mode | 2N Trap |

## TABLE 2
## Instrument Operating Conditions

| | |
|---|---|
| Trap | Tenax |
| Trap Ready Temp | 40 |
| Pretrap Ready Temp | 25 |
| Sweep Flow Rate | 20 |
| Sweep Time | 2 |
| Trap Dry Sweep Temp | 25 |
| Dry Sweep Flow Rate | 80 |
| Dry Sweep Time | 1 |
| Trap Desorb Preheat Temp | 210 |
| Trap Desorb Temp | 220 |
| Trap Desorb Time | 2 |
| Trap Bake Temp | 220 |
| Pretrap Bake Temp | 25 |
| Bake Flow Rate | 85 |
| Bake Time | 10 |

The same needle was used to perform traditional static headspace injections giving the analyst the ability to make both static and dynamic injections within the same sample schedule. The HS9000 not only delivers improved sensitivity with the dynamic sweeping ability but also delivers the proven technology of static equilibrium headspace analysis all in the same instrument. The two main favor compounds of interest in this study (d-limonene and a-pinene) were easily examined by the automated techniques see *Figures 3 & 4*.

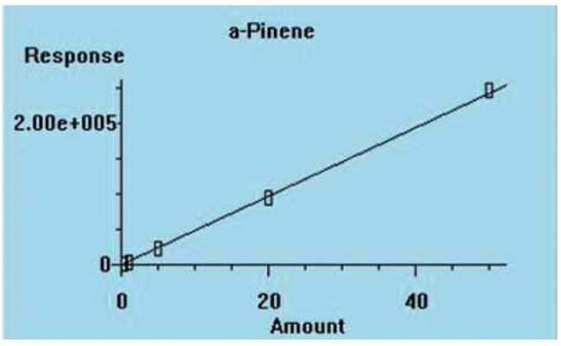


**FIGURE 3 0.5-50 ppb Cal curve**

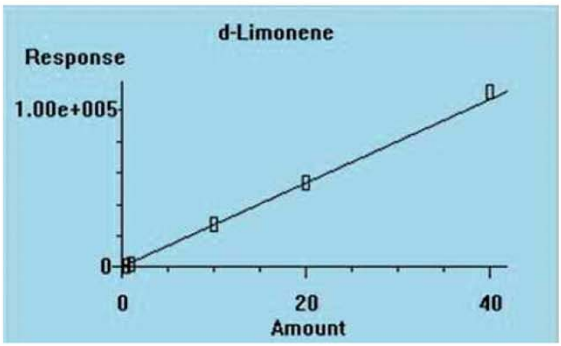


**FIGURE 4 0.5-40 ppb Cal curve**

The high precision and linearity examining the volatiles present in a complex mixture at such a wide range of levels is extraordinary Tables 3& 4. Method optimization can be completely automated by testing the variables such as the equilibration times, pressures, sweep time, flow time and temperatures with just a change in the method software  The analysis of product adulteration, impurity contamination, and off-odor characteristics can now be precisely examine with never before simplicity and ease of use.

**TABLE 3**

| Compound | 0.5ppb | 1ppb | 5ppb | 20ppb | 50ppb | Avg | % RSD |
|---|---|---|---|---|---|---|---|
| a-Pinene | 4.940 | 4.697 | 4.769 | 4.728 | 4.961 | 4.819 E3 | 2.55 |

**TABLE 4**

| Compound | 0.5ppb | 1ppb | 10ppb | 20ppb | 40ppb | Avg | % RSD |
|---|---|---|---|---|---|---|---|
| d-Limonene | 2.716 | 2.304 | 2.688 | 2.705 | 2.789 | 2.640 E3 | 7.27 |

## CONCLUSION

A new simple, automated, static/dynamic headspace gas chromatography technique can be used to determine the absorption of flavor absorption into the packaging material.  The new dual needle trapping technique provides a robust sample introduction method at unprecedented low levels of detection of favors in packaging.  Flavor compounds in food exist in trace levels that are very important concern to the food/package manufacturing process. Now researchers have a tool capable of measuring sub ppb levels with ease of use, accuracy and precision.


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