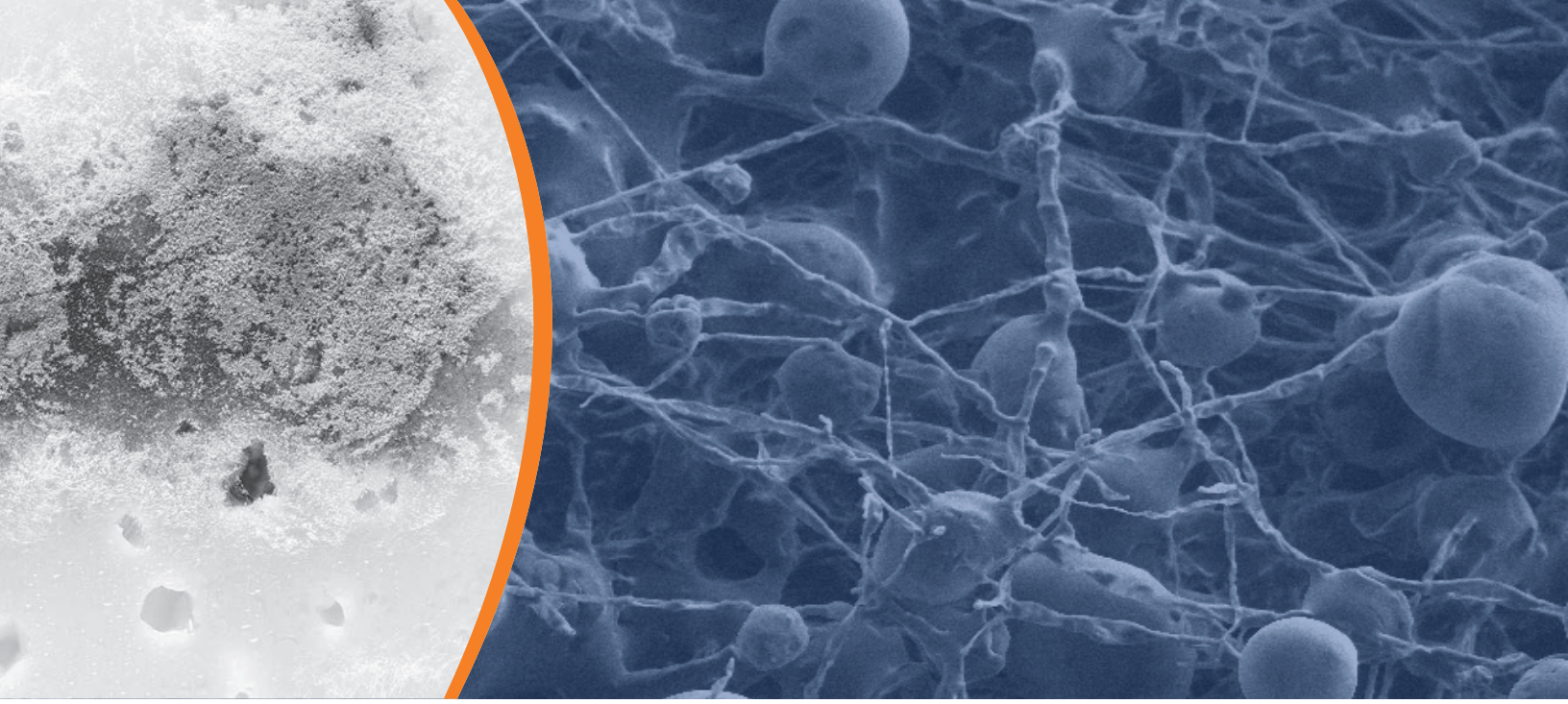

Off-flavor and Odor Investigations
Employing Solid Phase Micro-extraction and Gas Chromatography Coupled with Mass Spectrometry

A EUROFINS WHITE PAPER

Jamie Willems, PhD, Scientist II – Eurofins SF Analytical

eurofins

Table of Contents

Introduction:

The flavor of food is an important component to consumer appeal and is determined by a combination of taste and aroma. Off-odors/flavors are off-putting to consumers and may be a sign of potential problems, such as chemical or microbial contamination, spoilage due to improper handling or storage, or changes/issues in production. It is the volatile compounds which are responsible for the aroma and any off-odors associated with a product. Currently, thousands of chemicals have been identified as flavor active compounds and it is the profile of these compounds which contribute to the overall aroma of a product. By examining these profiles or 'fingerprints', compounds which may contribute to off-odors can be identified and potential sources of the issue identified. It should be noted that certain compounds may be a normal part of the flavor/odor profile of some products but a source of an off-odor/flavor in another. Therefore, one of the most definitive methods for the identification of off-odor compounds is through the comparison of the profile of a control/'good' sample to that of a complaint sample. In this way compounds present in the suspect sample and not in control or off-odor causing compounds present at elevated or suppressed levels can be identified and the source determined.

Gas chromatography coupled with mass spectrometry (GC-MS) is a powerful technique for the analysis of volatile profiles. Coupled with GC-MS, solid phase micro-extraction (SPME) offers increased sensitivity and selectivity for volatile analysis. SPME employs the use of fibers coated with a thin layer of polymer material which acts as an extracting phase allowing for the selective extraction and concentration of volatile compounds, increasing sensitivity and selectivity. SPME fibers are available in a variety of specialized coatings which allows for the targeting of different chemical classes of compounds.

Experimental Technique Summary:

Samples are analyzed for their volatile profiles using SPME-GC-MS. Based on consultations with the client and subjective observations, SPME fiber coatings are selected to target compound classes that are the most likely source(s) of the off-odor/flavor. Samples are incubated in headspace vials, with the temperature being selected based on the volatility of the compounds of interest, with higher temperatures used for compounds with lower volatility and vice versa. The SPME fiber is exposed to the generated headspace gases and allowed to equilibrate. Any adsorbed compounds are then desorbed into the GC inlet and the volatile compounds are separated using a capillary gas chromatography column and detected by mass spectrometry. Identification of components is done based on characteristic fragmentation patterns and library comparisons.

After analysis, the volatile profile obtained is examined for compounds which may contribute to off-odors. Ideally, this is done through comparison between a suspect and a control sample, looking for differences between them. For off-flavors not detectable by volatile analysis (i.e., salty, metallic) metals analysis by inductively coupled plasma - optical emission spectrometry (ICP-OES) can be used to identify elevated levels of certain metals which are responsible for off-flavors.

Case Studies:

Case Study 1: Off-flavor/odor in Packaged Hamburger Buns

Customers complained of a 'vinegar-like' off-flavor/odor in hamburger buns. Samples of control and complaint products were analyzed for their volatile profiles by SPME-GC-MS. Samples were additionally analyzed for their microbial content. Analysis showed that suspect samples had elevated levels of various organic acids and organic acid byproducts including 2,3-butanediol, pentanoic acid, phenylethyl alcohol and cyclohexane carboxylic acid which

likely led to the off-flavor complaint. In addition, sorbic acid (a common antimicrobial) was detected in the control samples and was not present or present at lower levels in the suspect samples. Microbial analysis did not show elevated levels of microbial growth in the suspect samples, suggesting that the off-odor/flavor was a product of production rather than product age or degradation.

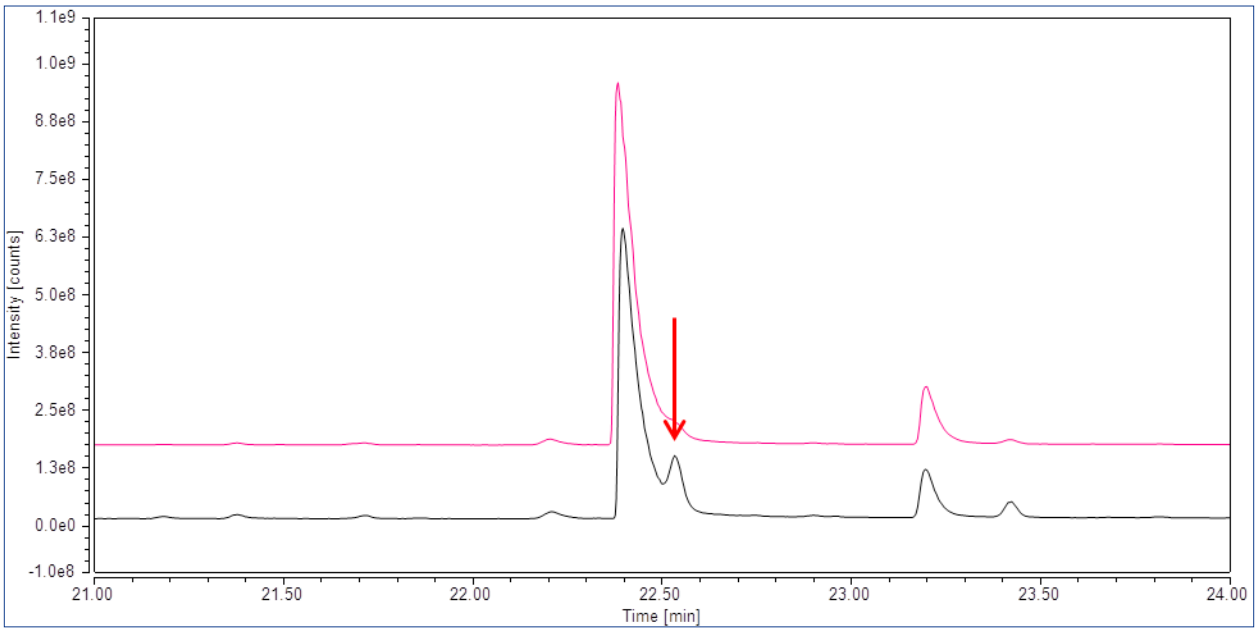

Figure 1. Expanded sample overlay of the SPME-GC-MS profile hamburger buns showing additional butanediol, indicated with a red arrow, present in the complaint sample (black) versus the control sample (pink).

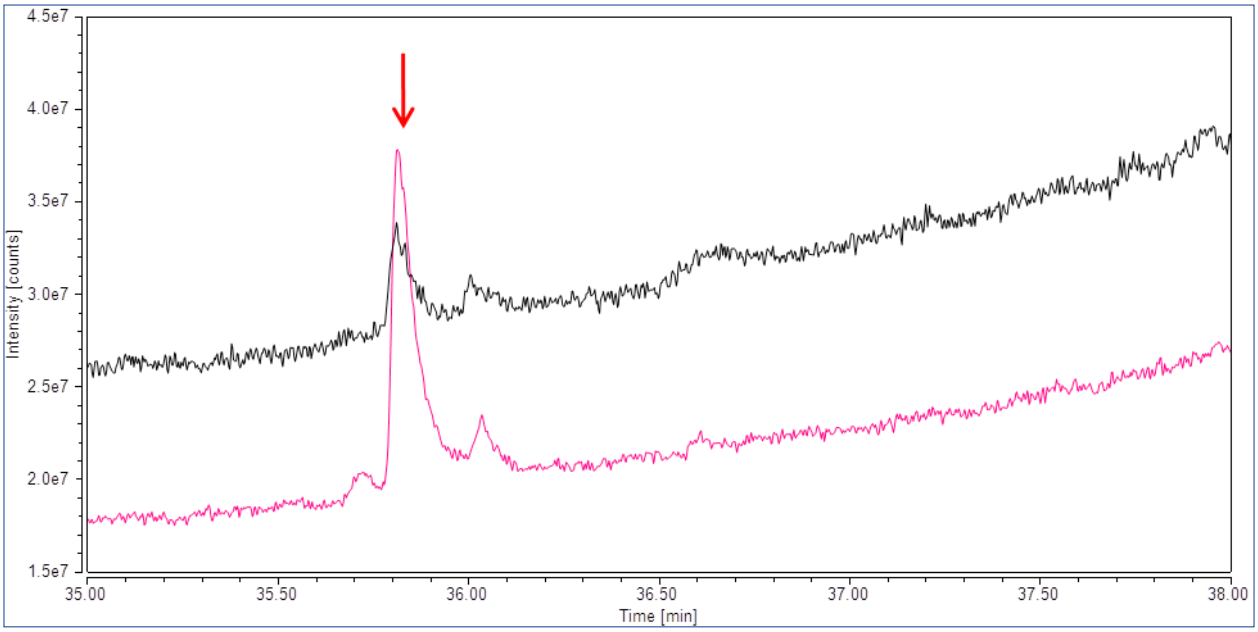

Figure 2. Expanded sample overlay of the SPME-GC-MS profile hamburger buns illustrating the higher amount of sorbic acid, indicated with a red arrow, present in the control sample (pink), with inconsistent or absent sorbic acid in the complaint sample (black).

Case Study 2: Off-flavor/odor in Chocolate Animal Crackers

Complaints were received about an off-taste and odor in chocolate animal crackers. Samples of control (good lot) and complaint products were analyzed for their volatile profiles by SPME-GC-MS. In addition, samples were ashed and analyzed by ICP-OES, in case the off taste was due to the presence of metals. While there was no significant difference observed between the metal content of the control and complaint samples, 1-(2-methoxy-1-methylethoxy)-2-propanol was identified by SPME-GC-MS to be present in the complaint samples, but not detected in the control sample. This compound was determined to be a key ingredient of a cleaner used on the baking line.

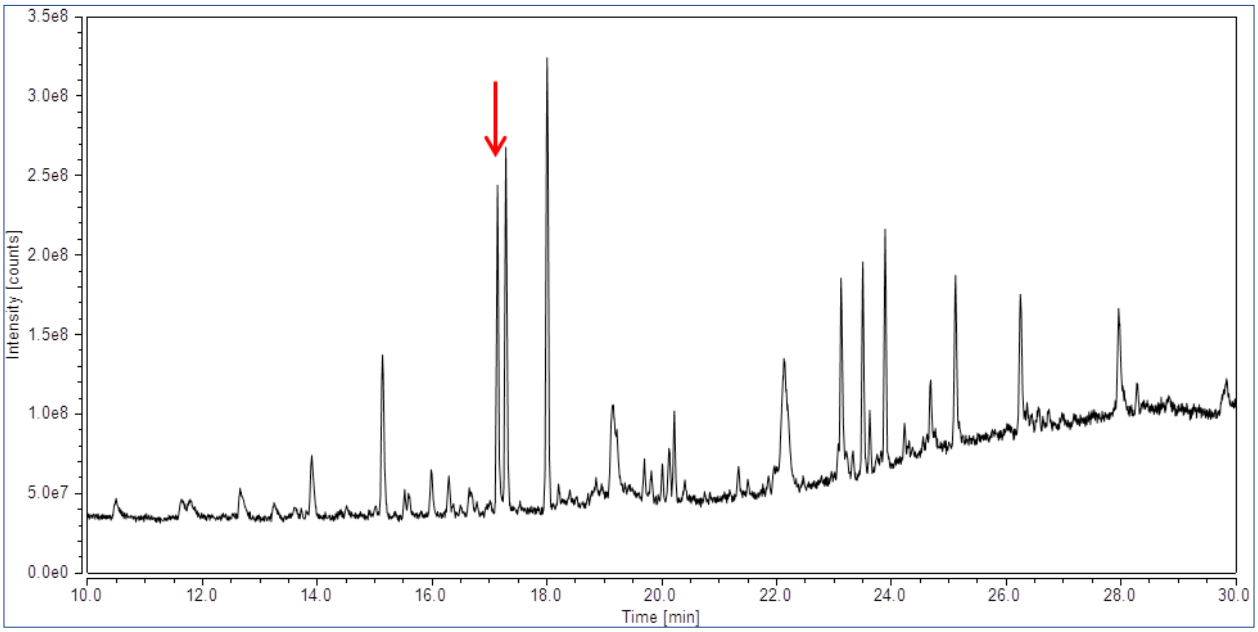

Figure 3. SPME-GC-MS profile of the complaint chocolate flavored animal cracker, 1-(2-methoxy-1-methylethoxy)-2-propanol is identified with a red arrow.

Case Study 3: Off-flavor/odor in Fruit Juice

A juice sample was received with the request that it be analyzed for the source of a perceived off-flavor/odor. The sample was analyzed by SPME-GC-MS and was compared to a control sample without the off-odor/flavor. Chromatograms of the suspect sample showed significant contamination with a variety of compounds, including 1-(2-butoxyethoxy)-ethanol, which were identified to be from common industrial cleaning agents. The between-batch rinse process was modified to avoid these issues in the future.

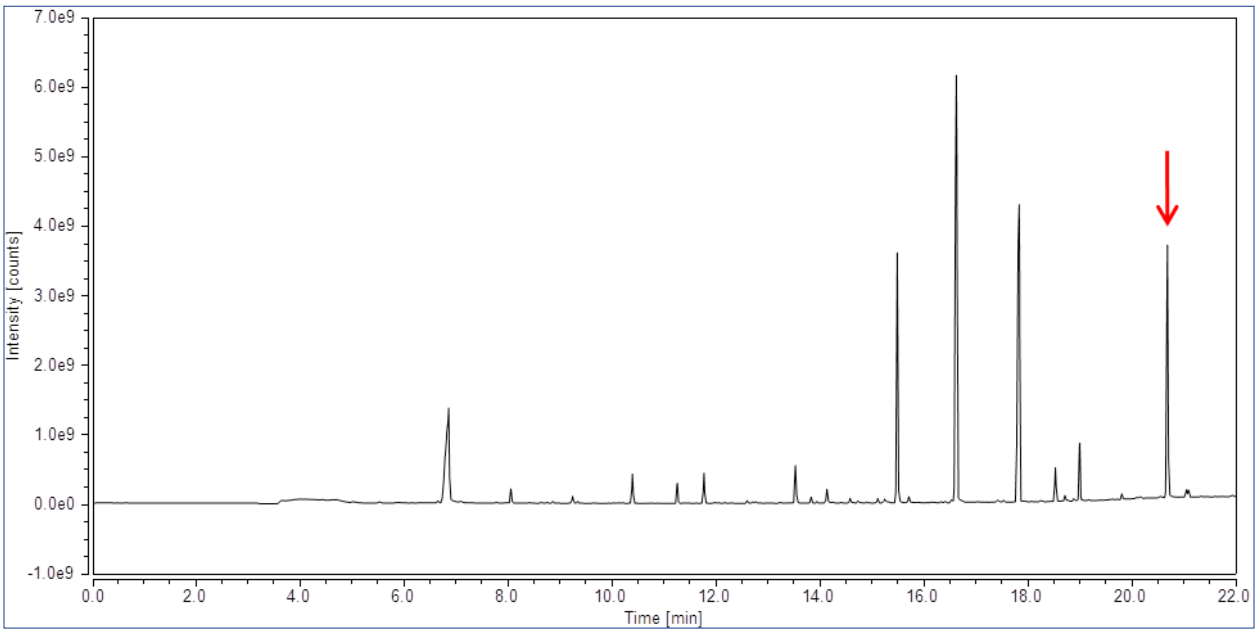

Figure 4. SPME-GC-MS profile of the complaint fruit juice, 1-(2-butoxyethoxy)-ethanol is identified with a red arrow.

Conclusion:

Aroma is an important component of a products flavor and appeal. Off-odors/flavors can be off-putting to consumers and may be indicative of larger concerns around product contamination, spoilage, storage and/or processing. SPME-GC-MS is a powerful tool in the analysis of off-odors/flavors in food and related products. Through the proper selection of SPME fiber and analysis conditions, compounds relating to many off-odors and flavors can be identified and this information used to identify a potential source and corrective action.