When Food Leaves A Bad Taste

Flavor Detective Ray Marsili Discovers Why

Off-flavors and odors in foods now constitute the <u>most common complaint</u> in the consumer food industry. According to food and flavor experts, more lawsuits are being filed today over alleged deterioration in foods, customers are rejecting products, and products are being recalled due to unintentional tastes and odors.

But the chemicals behind these off-flavors can be very hard to find. Often they're in very small quantities (such as nanogram or ppb scales), can be polar and therefore more difficult to extract using traditional methods, and may even work in concert in unexpected ways to produce an off-flavor, making isolation of those chemicals even more challenging.

Ray Marsili, an independent consulting chemist who specializes in determining the causes of off-flavors, has earned a worldwide reputation for his abilities as a "flavor detective" of sorts, working with companies to help determine the causes of certain flavors, and figuring out why things can go wrong.

Based in Rockford, Ill., Marsili's company, Marsili Consulting Group, uses GERSTEL products for his many projects involving flavor detection. He's worked with Pepsi, Kraft, Evergeen, and Starbucks, as well as large and small food concerns from South Africa, Colombia, Australia, Canada, and elsewhere (including the United States).

Not all of his customers are in the food industry. "I've had a lot of requests for odor

analysis from non-food places, like mattress makers, to find out what was causing odors in new mattresses." Another company approached Marsili to test its shirts for their ability to reduce body odor after hard exercise.

For all of these and other projects, GERSTEL has been an essential ingredient to his ability to determine the chemicals behind odors and flavors. "GERSTEL technology allows for unique applications," Marsili said. "I would not be able to do what I'm doing without GERSTEL. Their instruments are able to provide more sensitivity than I've ever seen."





AN EARLY, INDIRECT PATH TO CHEMISTRY

why." He was the first in his family to go to college, where one of his professors encouraged him to go on to graduate school. Married and with a master's in physical chemistry, Marsili worked in quality control for a pharmaceutical company and then switched to a food company. "One of the questions that kept coming up was, 'Why does this product taste bad?' The longer I worked there (at the food company), the more focused I was on off-flavor chemistry."

He also observed that analytical techniques like GC/MS were becoming more affordable, and technology like solid-phase microextraction

(SPME) and new analytical instrumentation like the <u>Autosampler for SPME</u> were starting to appear. These techniques helped him solve more problems. Another perk eventually came in handy: his bosses allowed him to publish his research, helping the company document improvements in flavor chemistry and helping Marsili make a reputation for himself.

In a way, he said, he owes a great deal of his career to beer. Using GERSTEL techniques like the SBSE Twister stir bar, "I found chemicals in beer nobody had found. I didn't know how good our data was. Then, a chemist from a beer company was really excited about our data." As more companies wanted to learn about these chemicals and sent him samples to work on, he got to be well known.

Off-flavors and odors can have many causes. These can include microbial contamination, interactions with packaging materials, barrier protection failures, oxidation reactions, or a combination of chemical reactions.

Determining the factors that could lead to these problems is a major issue for food and beverage companies and has become bread and butter for Ray Marsili.

A FLAVORFUL CAREER

A number of projects illustrate how Marsili used GERSTEL technology to resolve a wide range of flavor and odor issues:



▶ A large dairy food company was grappling with increasing off-flavor complaints in milk. This is a common problem in late winter and early spring, but the rate of complaints was increasing every year. SPME GC/MS analysis pointed to lipid oxidation products created by light-induced oxidation of butterfat. But that provided only a partial explanation. Why was the problem increasing each year? Further testing showed that the butterfat was increasingly laden with high levels of linoleic acid. The high acid levels were arising from farmers feeding their

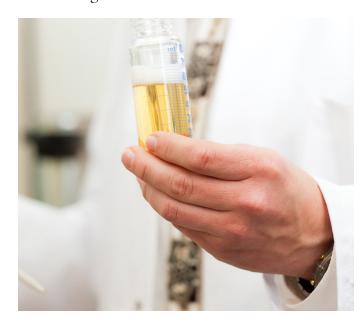
milk cows more soybeans. By asking farmers to reduce soybean levels in their feed and adding Vitamin E (an antioxidant to curb butterfat oxidation), the problem was resolved.

A company that created a new way to make dill pickles taste like fermented dills without the long, expensive, and polluting fermentation process was running into another type of pickle — buyers like McDonald's didn't want to take the risk of adopting the new process. Fermentation processes convert sugars to lactic acid, some acetic acid, and other trace metabolites to create the unique flavor of fermented pickles. Only one paper indicated the main flavor chemical, a terpene alcohol called linalool, responsible for fermented pickle flavor. However, Marsili's lab, using GERSTEL SPME and purge-and-trap GC/MS, could not find any evidence that this chemical correlated to fermentation flavor. Marsili, using the same GERSTEL equipment, discovered that the major odor impact chemicals in traditional fermentation were not linalool, but isomers of



3-hexenoic acid, which had never been reported before. This chemical also is a carcinogen, and nobody supplied it. Fortunately, McDonald's changed its mind and decided to take on Marsili's client and adopt the new non-fermented pickles after all.

▶ Beer raised its head again in 2007, when a major brewery asked Marsili to study off-flavor development. In this project, SBSE, along with the Leco Pegasus HT GC-TOFMS (time of flight mass spectrometry), with peak deconvolution capabilities, could detect several off-flavor compounds produced in beer during aging, as well as from light exposure. Identifying these compounds was challenging considering that more than 800 volatiles such as



alcohols, esters, aldehydes, ketones, volatile acids, terpenes and pyrazines all contribute to flavors in beer and wine, and often exist in the ppb range. In 2015, Marsili applied sequential-SBSE and multi-SBSE using two types of Twister stir bars to detect even more flavor-producing chemicals, and even lower concentrations. "One class of potent odor chemicals that had been difficult to detect was

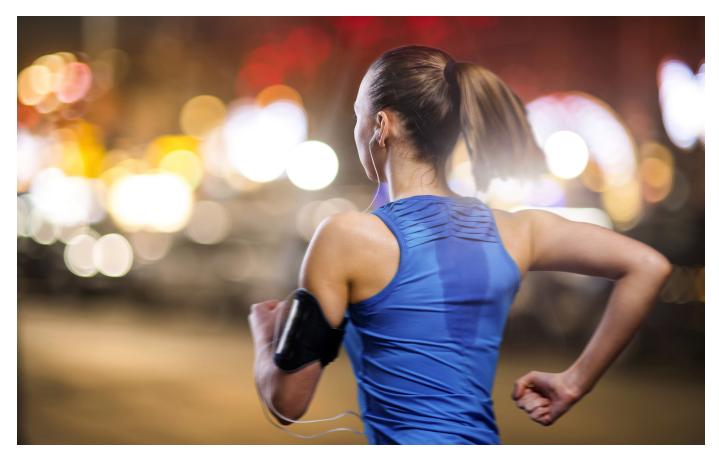
carboxylic acids. With seq-SBSE, and multi-SBSE, these chemicals can now be routinely monitored," Marsili said.

In a detour from food flavor, a sportswear company approached Marsili, asking him to test its athletic shirts for their ability to reduce body odor after exercise. Most exercise-produced sweat and secretions are odorless, but microbes on the skin can break down these secretions. creating smaller molecules that have intense odors. Most of these smaller molecules are volatile carboxylic acids. Fresh from using multi-SBSE to extract the same low levels of acids in beer, Marsili thought they could apply the same technology to the shirts. With one variation: the Twisters were in the headspace instead of in solution. By suspending the soiled shirt fabric from a paper clip punched in a GC vial lid and attaching a PDMS Twister and ethylene glycol silicon Twister to the vial side, they stirred the air in the vial and could extract low ppb levels of axillary carboxylic acids. The shirt manufacturer could thus perfect its fabrics to do an even better job of minimizing body odor from eccrine and apocrine sweat glands and sebaceous glands secretions.

LOOKING FORWARD, WITH A WARNING

progress over the past decades, largely thanks to instrumentation that's more accurate, precise, and cost-effective. Marsili has observed more uses for gas chromatography/mass spectrometry as techniques move from research labs into food manufacturing and quality control. "For food, instrumentation will be used more and more," Marsili said.

While GERSTEL instruments provide a solid foundation to Marsili's work, offering more



sensitivity, new technologies are providing more data. "More data gets a little unruly," Marsili said. "You chromatograph a sample with a lot of chemical peaks, and you run into problems with chemicals co-eluting." One solution to handling these elution and data mass problems is the time-of-flight (TOF) GC/MS, which determines an ion's mass-to-charge ratio by measuring the time the ion takes to reach a detector.

Data demands are also helping expand (if not create) the field of chemometrics. "When we started by doing solvent-based extractions, we might see 10 or 20 chemicals," Marsili said. "Now we'll see several hundred. It's hard to connect with which ones are producing off-flavors." Chemometrics, which uses mathematical and statistical models to analyze chemical data or create the procedures and

experiments tailored for any lab, can find trends in data that can't be found by just manually looking at results. "I think we're seeing a trend in its use now," Marsili said.

Massive volumes of data aren't the only challenge facing food chemists. Marsili said he's seeing fewer investments by manufacturers in flavor and food chemistry. "I think that's hurting them. A lot of food businesses aren't appreciating the contributions food chemistry can make to their business." This is especially problematic in an area where experience matters. "I've never seen a field where experience means so much. The longer you work, the more you encounter different types of problems. You can buy equipment, but it takes a long time to understand how the data you generate relates to the problems you are facing."



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