Stable Flavor Formulations, High-impact Aroma Chemicals and More

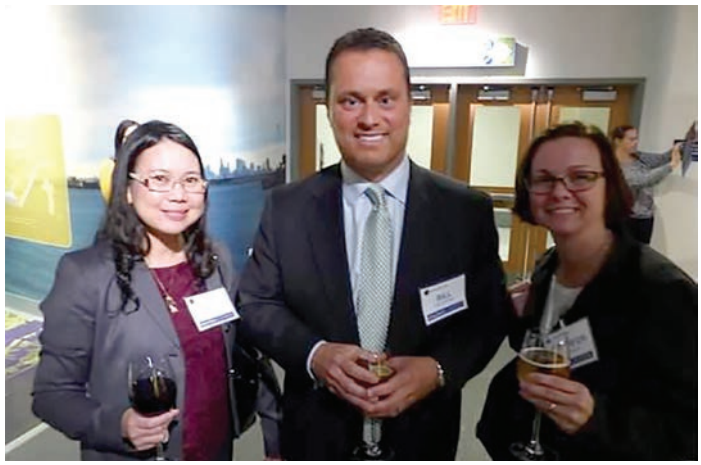
Select highlights from the Society of Flavor Chemists' 2014 symposium.

In 1954 Senator McCarthy's Communist witch hunt ended in formal censure by the US Senate, and the US Supreme Court ended segregation of public schools. It was also the year that Stouffer's released its first TV meals and General Mills introduced Trix—flavors were certainly in demand.

That year the Society of Flavor Chemists (SFC) held its first regular meeting at Little Venice restaurant in New York City, led by chair Jack Bouton of Synfleur.

"The purpose of the organization is primarily social," P&F announced at the time, "but occasionally informal talks on matters of mutual interest will be the order of business."

And so it was appropriate that the SFC's 2014 symposium at the Liberty Science Center in Jersey City, New Jersey, fell on its 60th anniversary, providing an informative two-day event that spanned ingredients, regulations, technologies and more. Below is a sampling of the many interesting conference presentations.



From left: Sally Domingo (Sensient), Bill Aslanides (Synergy Flavors) and Jennifer Thompson (Sensient).

Flavorists Discuss Applied Ingredients

During the general session, Kay Bardsley-Murano (IFF) presented (+/-)-*cis*- and *trans*-1,2-dihydroperillaldehyde (CAS# 22451-50-9; FEMA# 4312), a US nature-identical material found in watercress and curry leaf. The ingredient was floral and cuminlike, appropriate for pumpkin spice and brie flavor profiles, and for deepening and adding a seediness to raspberry flavors, and boosting dairy notes in butternut squash.

Next, Bardsley-Murano presented 6-methoxy-2,6-dimethylheptanal (syn: Aquaflor;[a] CAS# 62439-41-2; FEMA# 4745), which is also used as a fragrance material. The ingredient had a watermelon, fatty, sweaty, watery and aloelike profile appropriate for watermelon, pumpkin, mango, shrimp, kaffir lime and citrus flavors. At low levels, said Bardsley-Murano, the material could serve as a driver of preference.

Kent Zeller (Hershey) presented *cis*-3-hexenol in a strawberry flavor. The material provides a wild strawberry "kick," while the *trans*- form doesn't quite work in the flavor profile. He also showed a Yakima peppermint oil from Citrus & Allied that contained no l-menthol, which was replaced with neomenthol.

Consulting flavorist Brian Grainger, meanwhile, discussed the application of high-impact molecules in flavors. He presented the extreme example of geosmin, the odor impact compound of beetroot, which has an odor threshold in water of 21 ppt. One drop of the ingredient can flavor three Olympic-sized pools.

While many flavor volatiles are quite powerful, Grainger considers "high impact" any ingredient that is present in the finished product at a level of less than 20 ppb while imparting an aroma impact. These are often products that require two dilutions to be usable. For example, 2-methoxy-3-isopropyl pyrazine has a reported threshold in water of 2 ppt.

Grainger noted that 382 sulfur compounds have FEMA GRAS status. They are generally all unpleasant-smelling at high levels; the gap between threshold and unpleasant-smelling is quite narrow. Among these intensely powerful odorants is 1-para-menthene-8-thiol, which is known as grapefruit mercaptan and has a 0.1 ppt threshold. Furfuryl mercaptan is present in brewed coffee and is pleasant at 5 ppb but becomes skunky at 30 ppb.

Effects are not always as expected, Grainger said. For example, dimethyl sulfide added to orange makes it more mandarinlike. It is also important in jammy fruits.

3-Mercaptohexyl acetate (FEMA# 3851) in a demo boosted the passion fruit impact in an orange juice. Meanwhile, 5 ppb of geosmin (FEMA# 4682) added to a corn soup control boosted its sensory qualities.

Sulfur compounds oxidize readily, so Grainger explained that flavorists should always use fresh solutions and avoid chlorinated water. Meanwhile, overdosing can be avoided by knowing thresholds; this is crucial as it is quite easy for one to overdose by 100 times with powerful odorants. Furthermore,

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[a]Aquaflor is a trade name of Bedoukian

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sulfur compounds must be kept out of tasting areas to ensure a neutral workspace.

Essential Oils

The key technologies driving essential oil innovation over the last 60 years have been fractional distillation, chromatography and biotechnology, said Colin Ringlieb (Ultra International). Because field processing is relatively crude, the industry has always followed up with additional refinements to ensure that the oils that reach the market are of an acceptable quality. Field processing involves lots of hand work and may even employ wooden stills packed with clay—even to this day. However, he added, groups like the citrus industry behave more like big business agriculture; they are efficient and employ clean processing.

Today, products such as citrus, savory blends and mint are critical in many flavor systems, said Ringlieb, while herbal oils are also important. Other categories of oils may be only contributory to overall flavor systems.

Citrus materials for flavor applications are likely to be folded, from single to 20-times to produce terpeneless and sesquiterpeneless types for increased solubility. Meanwhile, fractions can isolate key chemicals within essential oils.

A working knowledge of seasonal variation is critical to formulators, said Ringlieb, as is an understanding of the impact of different fractionation techniques, ability to select materials based on the flavor task at hand and economics, stability and details of subsequent processing. Because essential oils are highly unstable naturally, understanding them is crucial. Essential oils stored well will age well, he added.

Authentication is a big concern for today's formulators, said Ringlieb. Are blended and WONF (with other natural flavors) flavors properly labeled? Ringlieb warned that stretching oils by adding non-volatile oils like castor oil and vegetable oil is a common practice amongst unreliable suppliers. Purchasers also have to be wary and screen for synthetic chemical addition.

Meanwhile, the volume of regulatory pressures will put low-volume oils at risk, and so it is crucial for the industry to find and maintain accessible resources for those ingredients. With just in time deliveries, prices can be volatile—particularly as there is no buffer inventory. As a result, big storms or other supply limitations can be crippling.

From left: Glen Kraemer (Sensient), Thomas Massetti (Craftmaster Flavor Technology) and Christina Barrera (Sensient).

From left: Kent Zeller (Hershey) and Jennifer Updegrove (Shanks Extracts).

Cloudy with a Chance of Customer Rejection: Stable Flavors

Cindy Cosmos (Bell Flavors & Fragrances) and Hedy Kulka (IFF) discussed the formulation of stable flavors using solvent combinations such as propylene glycol/triacetin, ethanol/triacetin, ethanol/MCT Neobee, ethanol/MCT/sunflower, etc.

The flavorist encounters many stability issues in formulations, the presenters noted, including cloudiness, which can of course lead to customer rejection. The flavorist's goal, said Cosmos, is to formulate a robust flavor that is clear and will remain stable even if accidentally refrigerated or exposed to extreme cold during shipping. As transportation has become more complicated, products become exposed to ever greater temperature and timing variables.

Assessing the appropriate solvent(s) for a formulation requires the flavorist to consider key parameters, including the actives present, organic compliance (if relevant), flammability, potability, cost, stability and legal/regulatory considerations. Solvents must withstand microbial growth and keep components such as ketals and acetals from reacting in situ.

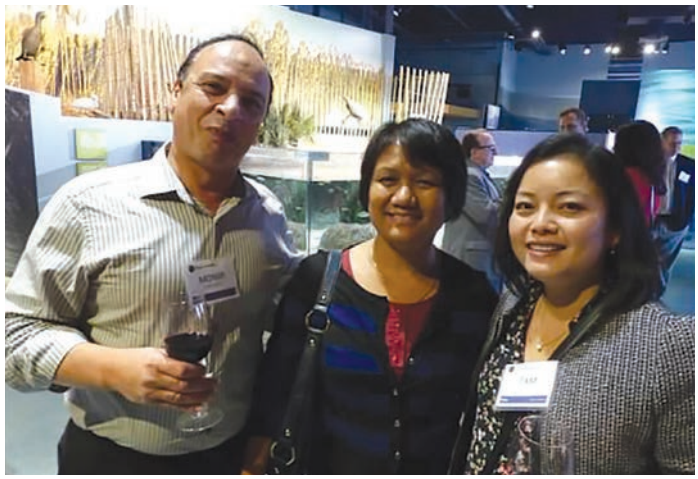
Kulka and Cosmos explained that flavorists must watch for phase separation, like oil slick separation creating layers from oeleoresins, as well as hazing and clouding, and recrystallization. While filtration can clean this up, this is a burdensome process that only confirms the flavor is out of balance and should have been reworked, said Cosmos.

Cosmos and Kulka have undertaken numerous experiments with ingredients and solvents. They presented an oleoresin in amino triglycerides and another in propylene glycol (PG). In the version containing triglycerides, one could observe some sediment.

Ethyl lactate or lactic acid can aid the addition of solids to a solution. These materials taste relatively neutral, so the flavor impact is minimal. On the other hand, triacetin is soluble in water to a certain degree, as well as MCT, PG and others; however, it is a plasticizer, which is a problem in gum. Triacetin is commonly used in combination with MCT to get more solids in an oil-based flavor.

When a formulation is completed, its solubility should be tested via refrigeration or by adding extra solids into the solution to see what, if anything, falls out. If just a small amount of additional solid material causes the formula to destabilize, it

From left: Vincent Moreau and Melissa Aubert (both Metarom Neotech), and Emeline Peridollan.



From left: Monir Zakhary, Delina Sagucio and Tam Luong (all Kerry).

is not sufficiently robust, said Cosmos. She reiterated that the flavorist can never be certain under which conditions a product may be shipped, so these precautions are necessary to ensure quality of flavor performance.

Guesstimates are inefficient, said Kulka. In developing their solvency experiments, Cosmos and Kulka found that many of their predictions of solubility—especially during refrigeration—proved to be incorrect. As a result, the only sure way to know one's formulation is robust is to test it.

Kulka and Cosmos offered a number of formulation tricks, including the use of anisyl acetone in place of oxyphenylon. They did note, however, that oxyphenylon could be desirable in chewing gum flavor systems in which it could contribute to longlastingness. In addition, they said that benzyl alcohol works well in solubilizing oleoresins. Despite that it has some flavoring contribution, its value on the solubility side outweighs any flavor issues. In addition, the presenters said, it's important to remember that "triacetin doesn't like terpenes" and that butter derivatives are hydrophobic.

Encapsulation

Spray drying comprises 85–95% of all encapsulation, which is due to its cost efficiency, said Robert Sobel (FONA). Spray chilling and fluid bed coating are also used, but with much less frequency.

The question of encapsulation centers on how a formulator is able to pay homage to all the different components in a flavor and deliver it to the consumer. How a flavor translates in the spray drier can be mapped out, said Sobel, by factoring the relationship between vapor pressure and temperature, as well as the mix of surface area and activity during the process. Flavor retention in the spray drier is typically 89%, while tray drying is 95%.

Understanding the process can improve outcomes. The ideal flavor systems are composed of a single phase wherein all components share similar chemistry. When a solvent carrier is added to a flavor and that flavor is spray dried, chemical potential energy is minimized, leading to improved retention in spray dried flavor.

Sobel offered some examples of solubility in commercial applications. For instance, inclusion of cinnamon, clove, allspice and nutmeg inhibits yeast's ability to act as a leavening agent, which is good for frozen dough products. For a flavor-changing ice cream, cherry ice cream flavor can be encapsulated with a fat, so the profile goes from vanilla to cherry vanilla. Meanwhile, chewing gum matrices may use multiple encapsulations for cooling or heating.

For information on future meetings,
visit *www.perfumerflavorist.com/events/calendar*.