WHITEPAPER

DIRECT, REAL-TIME AROMA ANALYSIS USING SIFT-MS

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ABSTRACT

Generation and release of aroma compounds from food products is a dynamic process. Conventional analytical technologies – such as gas chromatography-mass spectrometry (GC-MS) – are poorly suited to studying changes on a timescale of seconds to minutes. This whitepaper introduces selected ion flow tube mass spectrometry (SIFT-MS) – a start-of-the-art analytical technique for the tool kits of flavor chemists, new product developers, and food processing engineers. SIFT-MS analyzes chemically diverse aroma compounds in real-time, including amines, organosulfur compounds, and volatile fatty acids.

THE SIGNIFICANCE OF VOLATILE COMPOUNDS IN FOODS AND PERSONAL CARE PRODUCTS

Volatile organic compounds (VOCs) impart aroma to food, beverage, and personal care products (PCPs). Development and/or release of aroma compounds from these products frequently occurs over short timescales of seconds to minutes, requiring fast analysis techniques to probe the dynamics.

Conventional analytical technologies used for VOC analysis – most notably gas chromatography-mass spectrometry (GC-MS) – are poorly equipped to address these needs. Factors that preclude high-throughput and real-time analysis using GC-MS and HPLC are that:

- **Chromatography is slow** by its very nature, since it is intended to separate analytes in time.
- Achieving **low detection limits** (part-per-billion-by-volume (ppbV) and below) usually requires preconcentration and/or extraction to be utilized, prolonging sample preparation.
- **Chromatographic columns are discriminatory**, so these methods cannot provide a complete analysis of the sample. Use of derivatization or other analyte modification also increases sample preparation time.
- **Moisture** generally needs to be removed prior to analysis.
- **Data post-processing and interpretation** from chromatograms is typically an off-line activity, delaying decision making – especially on the process line.

Direct, real-time analysis using selected ion flow tube mass spectrometry (SIFT-MS) overcomes these limitations, delivering high sensitivity, high specificity, and high time resolution VOC analysis with simplicity and robustness. This whitepaper gives examples of food and PCP applications where real-time VOC analysis has significant benefits. Broadly, these are organized as food processing, breath analysis, and the ability to probe time-dependence of aroma release.

SELECTED ION FLOW TUBE MASS SPECTROMETRY (SIFT-MS) – A BRIEF INTRODUCTION

SIFT-MS is the leading real-time analytical technique for comprehensive gas and headspace analysis to ultra-trace concentrations. SIFT-MS (Figure 1) uses ultra-soft, precisely controlled chemical ionization (CI) coupled with mass spectrometric detection to rapidly quantify VOCs and permanent gases to sub-part-per-billion concentrations by volume (ppbV). Up to standard eight chemical ionization agents (reagent ions) are available in Syft Technologies SIFT-MS instruments, such as the Syft Tracer™: H_3O^+, NO^+, $O_2^{+\cdot}$, OH^-, $O^{-\cdot}$, $O_2^{-\cdot}$, NO_2^-, and NO_3^-. These reagent ions react with analyte VOCs and inorganic gases in very well controlled ion-molecule reactions, but they have very slow or no reaction with the major components of air (N_2, O_2, Ar, CO_2 and H_2O). This enables SIFT-MS to analyze air at trace and ultra-trace levels without preconcentration or sample drying. Rapid switching of reagent ions provides unsurpassed specificity compared to other direct-analysis techniques. To learn more about SIFT-MS, see the Technology Overview (https://syft.com/assets/Knowledge-Centre/SIFT-MS-Overview_Mar-2023.pdf) and recent review articles (Langford (2023) and Smith *et al.* (2023)).

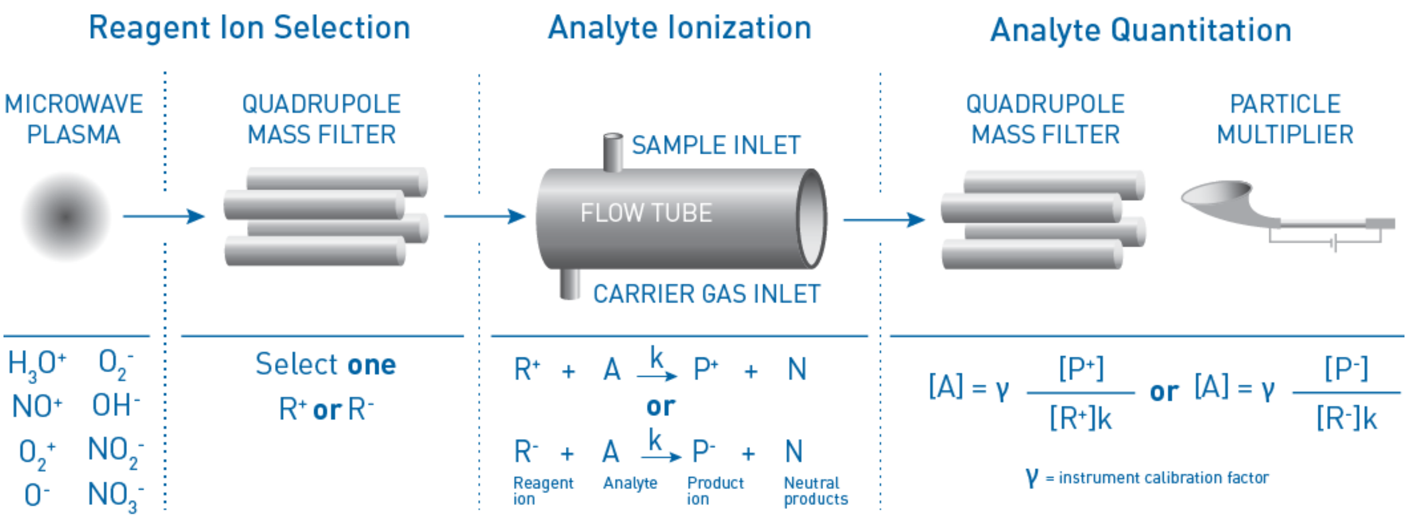

Figure 1. Schematic representation of the SIFT-MS technique.

1. Sample Introduction Options

Real-time analysis using SIFT-MS requires continuous introduction of sample through the instrument's sample inlet (Figure 1) – typically at a standard sample flow rate of approx. 25 standard cubic centimeters per minute (sccm). Depending on the application, sample gas can be drawn continuously from one point or cycled through multiple points (for example, to monitor several processes).

For high-throughput applications, robotic syringe-injection autosamplers are an excellent fit (Langford and Perkins (2024)). These samplers can also be utilized for continuous headspace analysis (CHA) with appropriate minor modification to the flow path (Langford and Perkins (2025)).

The flexibility of SIFT-MS sample delivery facilitates straightforward implementation – including process-line integration – across a wide range of applications.

2. Targeted and Untargeted Approaches

SIFT-MS can be utilized for both targeted (selected ion monitoring, SIM) and untargeted (full scan) analysis, each with distinct benefits. SIM mode enables direct, quantitative analysis of targeted aroma compounds, whereas full-scan mode provides a broader view of the sample and can be used to detect volatiles that are outside a target compound suite – albeit with a modest reduction in sensitivity and response time.

FOOD PROCESSING: OPTIMIZATION AND MONITORING

Real-time, broad-spectrum aroma compound analysis, coupled with simple sample delivery and instant reporting of data, enables SIFT-MS to be readily applied to continuous monitoring of aroma release in food processing. This section illustrates SIFT-MS monitoring of three products: coffee, cacao, and milk powder.

1. Coffee Roasting

Generation of a consistent flavor profile from each coffee roast requires a high degree of skill due to natural variations in beans and the dynamic nature of flavor formation during roasting. Applying SIFT-MS to real-time analysis of volatile compounds during coffee roasting has potential to support production of consistent quality coffee. Figure 2 shows continuous analysis of selected VOCs when a single bean was roasted at constant temperature (200 °C). Most VOCs exhibit two distinct peaks, which to a burst of volatiles released from the well-known “cracks” that occur during roasting.

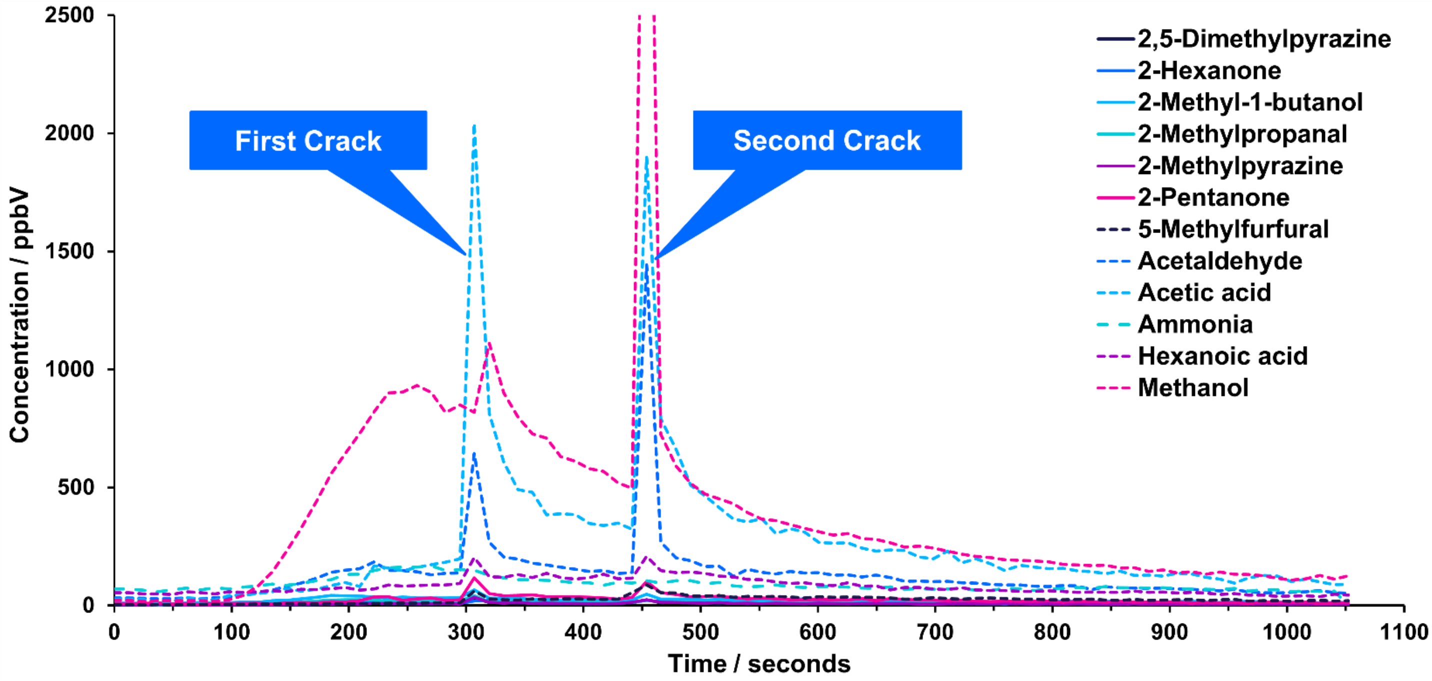

Figure 2. SIFT-MS volatile analysis of a coffee bean during roasting at 200 °C. For clarity, only one third of the compounds targeted are shown.

2. Cacao Roasting

Flavor generation during roasting of cacao beans is derived from the Maillard reaction and is conventionally monitored by trained staff members. SIFT-MS can replace human sensory analysis, providing repeatable, objective data long term.

Cacao flavor generation depends on the roasting temperature, as Huang and Barringer (2011) have shown. Moreover, the rapid response of SIFT-MS enables an optimal roasting time for a particular flavor profile to be achieved, from batch-to-batch.

3. Milk Powder Spray Drying

Spray drying of milk solids is a demanding process, requiring great care to maintain both the integrity of the product and avoid conditions that can lead to exothermic reactions – and potential explosions. The likelihood of explosive combustion is difficult to anticipate, as it depends on several factors, including the composition of the product being dried. The current industry standard for combustion detection utilizes carbon monoxide (CO) detection using infrared spectroscopy, but this approach has drawbacks including potential interference from other environmental combustion sources and inadequate sensitivity for large modern dryers.

SIFT-MS is a very good alternative for explosion prevention monitoring with higher sensitivity, targeting multiple VOCs simultaneously that correlated well with CO in side-by-side studies (Figure 3). Additionally, SIFT-MS readily detects aroma quality markers arising from both oxidation and Maillard (browning), as annotated on full-scan data shown in Figure 4. Hence SIFT-MS reports both safety and quality attributes in a single, continuous analysis with time resolution of a few seconds (in targeted mode).

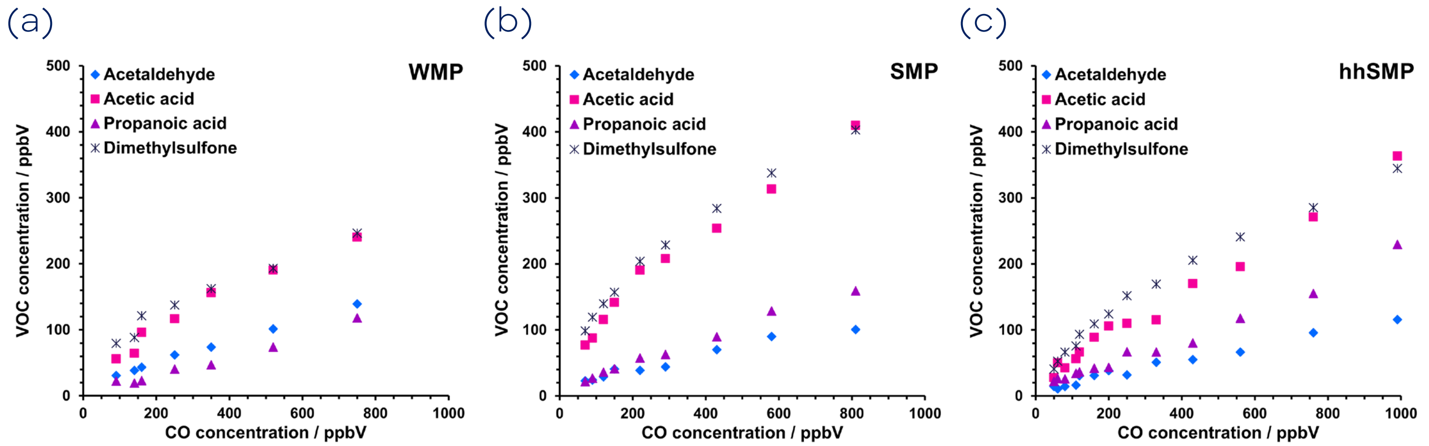

Figure 3. Correlation of conventional CO monitoring data with four VOCs analyzed by SIFT-MS for (a) whole, (b) skim, and (c) high-heat skim milk powders (WMP, SMP and hhSMP, respectively). Data points plotted are those that represent a rise in CO concentration from above baseline to 1000 ppbV.

BREATH ANALYSIS

Flavor is a combination of sensation from taste stimuli and retronasal odor stimuli. The odor component is obtained from air delivered to the nose from the mouth or by sniffing. Conventional chromatographic techniques are not readily applied to this so-called "flavor release" research. In contrast, the SIFT-MS technique enables direct detection from nose or mouth space (Smith *et al.* (2023)). In this section, flavor release from confectionery, tomatoes, and garlic are reviewed briefly.

1. Confectionery

SIFT-MS readily analyses aroma compounds released from confectionery while it is chewed or sucked in the mouth. Typical breath exhalations will show several chewing cycles in a peak "envelope", with the chewing action stimulating greater aroma release. Figure 5 shows the in-nose results obtained for fruit-flavored chewing gum as measured in the nose-space of a volunteer.

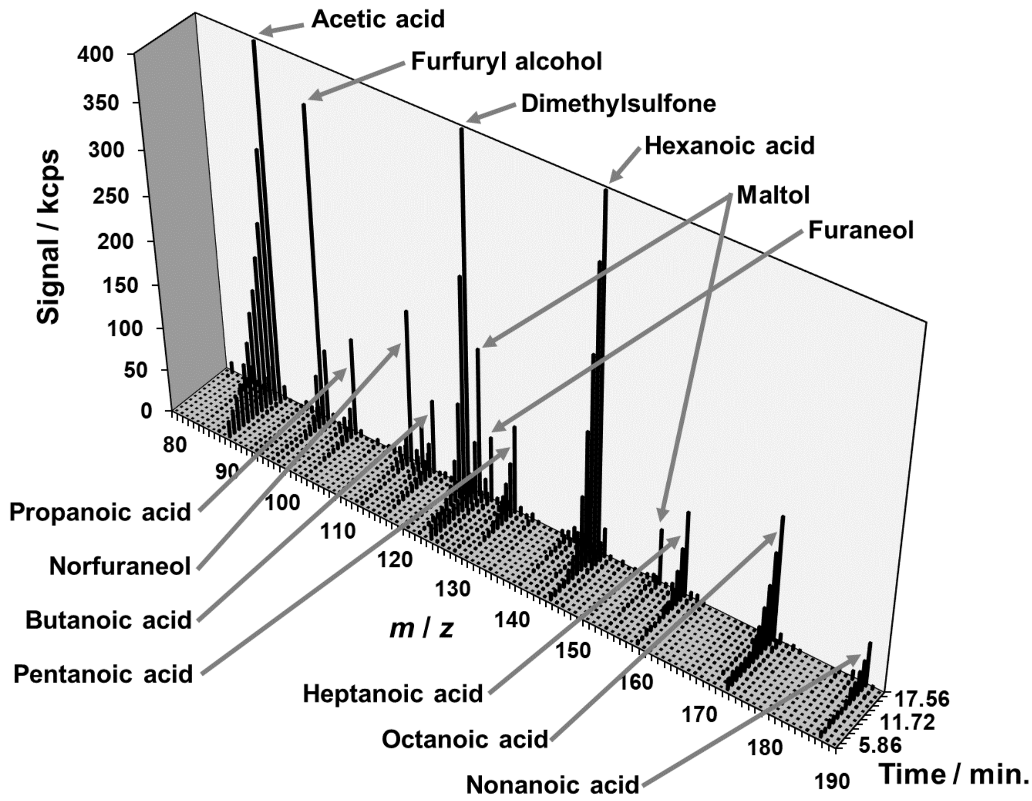

Figure 4. A three-dimensional view of SIFT-MS full-scan data (NO^+ reagent ion) obtained when 1 g of skim milk powder was heated from approximately 80 to 150 °C.

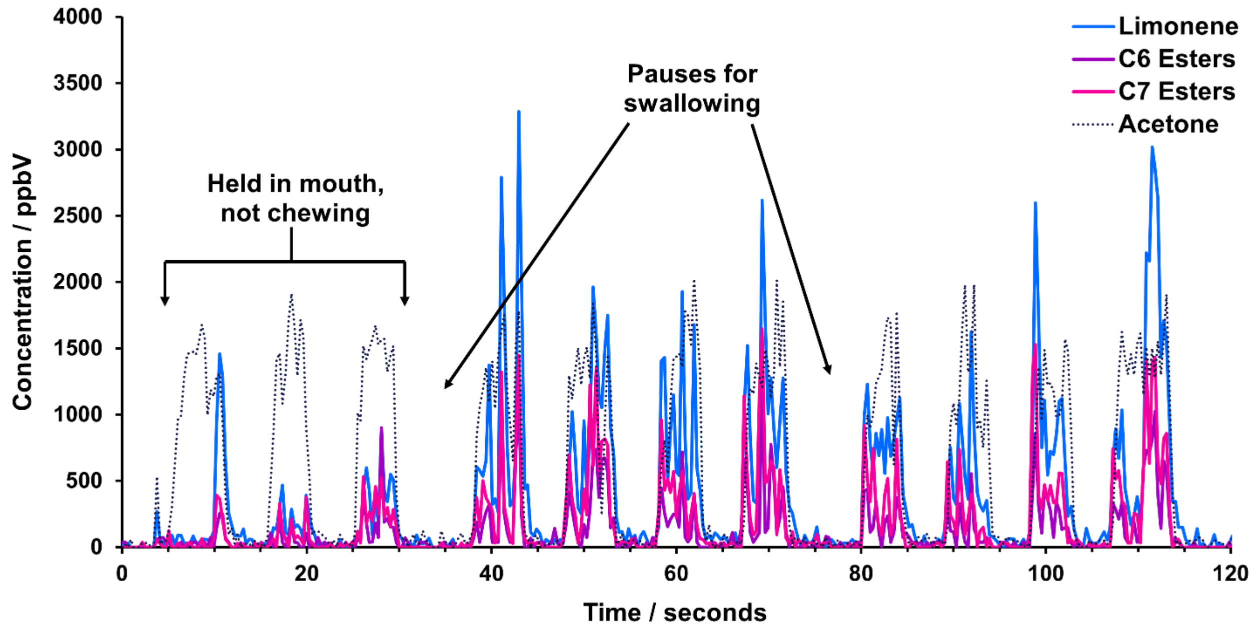

Figure 5. Real-time SIFT-MS analysis of nose-space during chewing of fruit-flavored gum. Acetone is included as a marker of breath exhalation.

2. Tomatoes

Professor Barringer's group at Ohio State University has used SIFT-MS breath analysis to demonstrate that certain aroma volatiles are generated in the mouth (Xu and Barringer (2010)). On chewing whole tomato, lipoxygenase enzyme action occurs rapidly due to tissue damage, resulting in formation of hexanal, *cis*-3-hexenal, and trans-2-hexenal as major volatile compounds. Whereas preexisting aroma compounds in tomato have a steady concentration during chewing, the lipoxygenase-derived volatiles exhibit a rise in concentration. Different rates of formation have been observed for different tomato varieties.

3. Garlic Breath

"Garlic breath" is an unpleasant side effect of consuming garlic and there is considerable interest in decreasing garlic breath malodor. Prior to the work of Barringer and coworkers, most studies utilized headspace analysis. However, the mechanism of deodorization is very different between headspace and the mouth. Factors involved include the time scale of reaction between garlic and treatment, the extent of mixing, and creation and destruction of odor components *in vivo*. By analyzing garlic odor compounds on the breath of human subjects (Munch and Barringer (2014)), SIFT-MS has enabled truly effective deodorization constituents to be identified. See also Castada *et al*. (2017).

PROBING THE DYNAMICS OF VOLATILE RELEASE

Real-time SIFT-MS analysis enables the dynamics of volatile release – from samples as diverse as fruit juices/drinks, muscle rubs, and live basil plants – to be studied in real time. Note that the dynamic SIFT-MS analysis contrasts greatly with the so-called dynamic headspace analysis (DHA) utilized with GC-MS. In DHA-GC-MS, volatiles are accumulated on a thermal desorption tube (TDT) over the duration of the volatile stripping procedure and upon completion the TDT is thermally desorbed and the (single) GC-MS analysis is conducted. Hence no dynamics are measured using DHA-GC-MS.

1. Purging Volatiles from Fruit Juices

In addition to static headspace analysis, SIFT-MS can probe the dynamics of flavor release. For fruit juices and imitation beverages this accomplished simply by analyzing the purge gas into the SIFT-MS instrument inlet. Figure 6 shows results obtained for squeezed and reconstituted orange juices and a reconstituted, orange-flavored powdered beverage when 150-mL of diluted beverage was placed in a bubbler and purged. Although the relative abundances of target compounds differ significantly between beverages, it is evident that the rate of release can also differ for a specific compound – presumably due to differences in beverage matrices. Also, alcohols and aldehydes have a slower decay than limonene due to their much higher affinity for the aqueous solution. These data demonstrate the ease with which purging studies can be conducted using SIFT-MS.

(a)

(d)

(c)

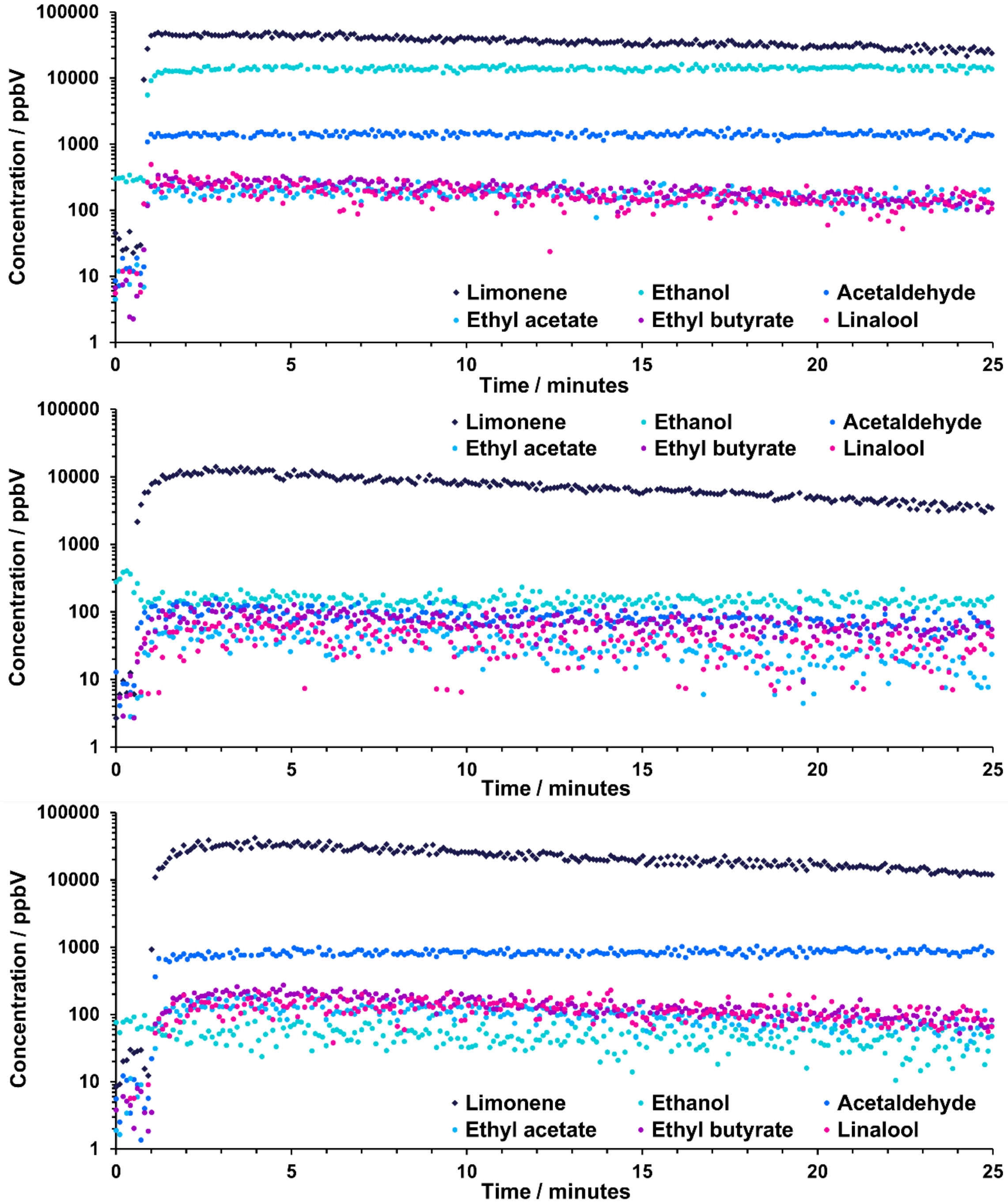

Figure 6. Continuous SIFT-MS analysis of selected VOCs stripped from (a) squeezed and (b) reconstituted orange juices, and (c) a reconstituted powdered orange-flavored beverage (all diluted five-fold with water). Note the logarithmic concentration axis. Elevated ethanol concentrations in laboratory air are evident in (a) and (b) prior to connection of the purge gas (about 1 min. after data acquisition started).

2. Fragrance Release

Temporal changes in aroma composition of PCPs are readily probed using SIFT-MS. Figure 7 shows use of CHA to investigate the release of fragrance compounds from 50 mg of muscle rub at room temperature using an automated CHA-SIFT-MS approach (Langford and Perkins (2025)). Sample was purged continuously with dry zero air at a flow rate of 25 sccm and a data point was acquired every 13 s over a 4-hr period. This popular PCP brand exhibits dramatic changes in fragrance composition over time, which could potentially impact consumer acceptance of the product. Using these insights, the product development processes can be enhanced through previously unavailable quantitative data.

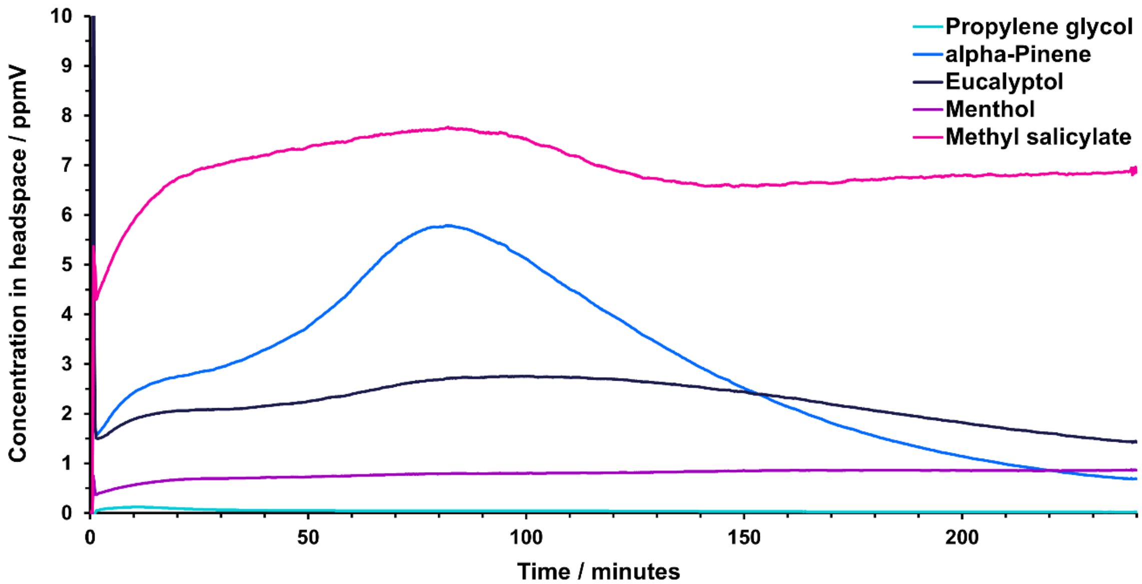

Figure 7. CHA-SIFT-MS of fragrance components as they are slowly released from a popular brand of muscle rub.

3. Plant Response to Wounding

Plant species – even to the cultivar level – usually have distinctive natural aromas. When damaged, additional volatile compounds are generated through enzymatic activity. SIFT-MS readily detects and quantifies volatiles characteristic of both aroma and leaf wounding in real-time, which are of significance to the culinary and agricultural industries, respectively. The dominant VOCs released from basil that contribute to its aroma are terpenoids, such as methylchavicol, linalool, 1,8-cineole (eucalyptol), and eugenol. Like most other plants, basil produces additional VOCs when it is wounded as a rapid, enzyme-regulated response. “Leaf wounding” compounds are commonly C_6 alcohols, aldehydes and ethyl esters, and are thought to be produced as a defense against microbial attack, or to attract predators in the case of insect attack.

The levels of three VOCs released by basil during leaf wounding are shown in Figure 8. Linalool and 1,8-cineole are released continuously by the plant and are detected at concentrations which depend on the plant's proximity to the instrument's sample inlet - hence the observed variability. When leaves are ripped, leaf wounding compounds (here represented by *trans*-2-hexenal) are rapidly released, as are higher concentrations of the terpenoids. When whole leaves are again presented to the inlet, the two terpenoids are still detected (not discernible on the scale shown), but they are not accompanied by leaf wounding compounds. Hence SIFT-MS is well suited to applications both in assessment of cultivar aroma and in research on plant responses to wounding (including insect damage).

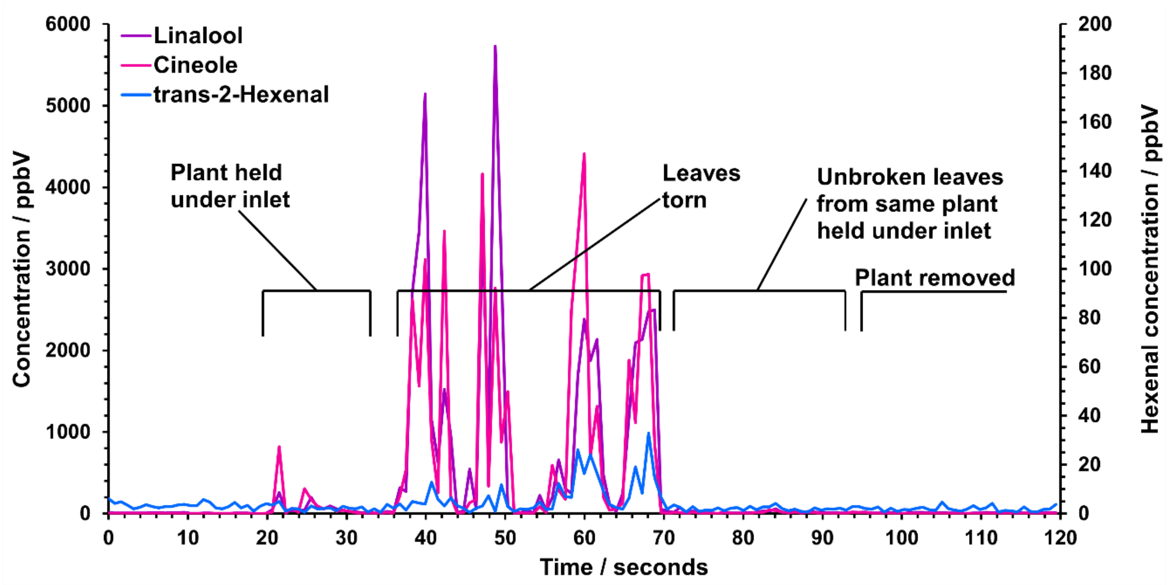

Figure 8. Concentrations of linalool, 1,8-cineole (eucalyptol) and trans-2-hexenal when a basil plant is presented to the SIFT-MS instrument and its leaves wounded by tearing. All compounds were detected and quantified using the NO^+ reagent ion. Note that trans-2-hexenal is shown on the right-hand axis.

CONCLUSIONS

The Syft Tracer™ SIFT-MS platform provides incredibly flexible options for probing aroma development and release in real time. Applications include:

- Direct analysis of flavor release (including direct from breath),
- Process optimization and monitoring, and

- Supporting rapid new product development.

Through rapid, sensitive, and comprehensive analysis, SIFT-MS provides new solutions to challenges facing food R&D and processing applications

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