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HIGH-THROUGHPUT ANALYSIS OF VOLATILES FROM FRUIT USING SIFT-MS

This application note demonstrates the simplicity with which small molecules are detected from fruits using direct SIFT-MS analysis, in contrast to conventional gas chromatographic methods. Automated SIFT-MS also provides significant throughput and method development advantages compared to conventional analytical approaches.

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INTRODUCTION

During ripening, fruits emit a diverse range of low molecular weight compounds arising from various hormonal and metabolic processes. The relative abundances of these volatiles change over time. Ethylene is usually of particular interest because it promotes ripening. Conventional gas chromatography (GC) methodologies are often applied to such analyses, but this is challenging for ethylene and the solvent-type compounds emitted from fruits. A recent application note from a major GC manufacturer demonstrates this,1 with analysis of low molecular weight species from apple taking over 30 minutes per sample, following incubation at 80°C for one hour. This approach means that the apple sample is essentially slow-cooked! In this application note, we demonstrate the application of selected ion flow tube mass spectrometry (SIFT-MS)2 to analysis of small molecules from several fruits,3 demonstrating significantly higher throughput by using automated direct mass spectrometric analysis.

METHOD

The data summarized in this application note were obtained using a Syft Technologies Voice200ultra SIFT-MS instrument (see syft.com/SIFT-MS for more information) integrated with a GERSTEL Multipurpose Sampler (MPS) (GERSTEL, Mülheim an der Ruhr, Germany).

The target compound list was that used in the GC-MS application note (ethane acetaldehyde, ethanol, 1-propanol, 1-butanol, and ethyl acetate), except that pentyl acetate was added subsequently.

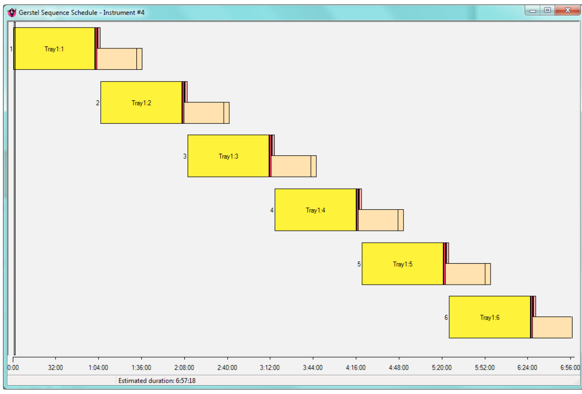
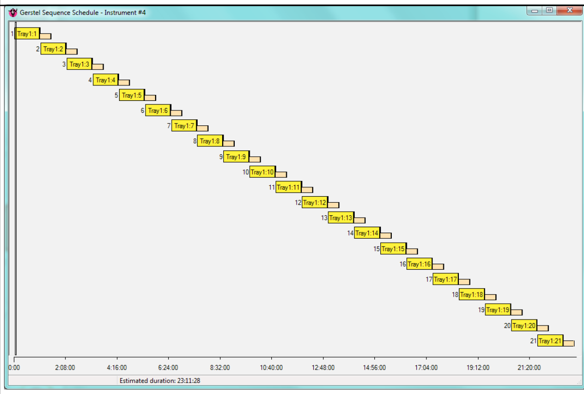
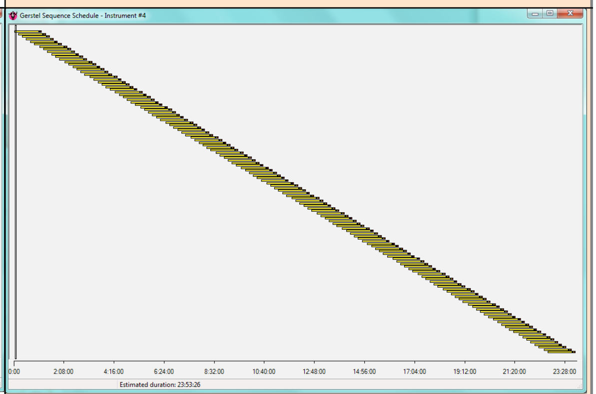
Initial experimentation utilized 1-gram apple samples placed in 20-mL sample vials and incubated at 80°C (Gerstel sixplace incubator/agitator), as used in the GC-MS application note. However, both temperature and incubation time appeared excessive, so in subsequent sample analyses the incubation temperature and time were reduced to 60°C and 15 minutes, respectively, while 2-gram samples were used. This phase compared three apple varieties, a pear variety and a banana variety.

The GERSTEL Maestro software enables efficient scheduling of samples for analysis. The rapid analysis provided by SIFT-MS removes a significant rate limiting step: the long analysis time in GC-MS (Table 1). Under the one-hour sample incubation conditions, six samples can be analyzed nearly 3.5x faster using SIFT-MS rather than GC-MS. Translating this to 24-hour throughput, SIFT-MS can analyze over six times more samples than the GC-MS method — and this with a six-place incubator / agitator device that was designed and optimized for chromatographic analysis, not direct SIFT-MS.

RESULTS AND DISCUSSION

Figure 1 shows data obtained for a 1-gram apple sample incubated for 60 minutes at 80 °C. For illustrative purposes a 90-second trace is shown, which shows the background at the start and end, and the rapid rise in headspace to a steady value when the sample is injected into the SIFT-MS instrument's sample inlet. Comparing the SIFT-MS results with those in the GC-MS application data sheet, the relative levels are the same for both techniques. However, the analysis time is not the same! Compared to GC-MS, each SIFT-MS data point represents acquisition of results equivalent to an entire chromatogram in just a matter of seconds. Because SIFT-MS limits of quantitation improve with increased acquisition time, typically sample averaging occurs over tens of seconds.

Table 1. Comparison of sample throughput for GC-MS and SIFT-MS analysis with a one-hour incubation time using the GERSTEL Maestro software.

THROUGHPUT MEASURE	HS-GC-MS	HS-SIFT-MS
Time to analyze six samples (GERSTEL incubator / agitator capacity)	 Total time: 6 hours, 57 minutes	 Total time: 2 hours 1 minute
Samples analyzed in 24 hours	 21 samples	 128 samples

Clearly there is opportunity to further optimize the method — especially in terms of shorter incubation times and reduced temperature to reduce temperature induced changes to the volatile profiles. This work will be the subject of a subsequent application note, but the GERSTEL Maestro software gives insight into just how efficient this will be with SIFT-MS. Because of the short injection and syringe flush cycles with SIFT-MS, six incubation cycles (in 5-minute steps from 5 to 30 minutes) can be evaluated in less than 37 minutes (Figure 3)! With GC-MS, this evaluation will take 3 hours and 42 minutes, or six times longer.

CONCLUSION

SIFT-MS provides simple and highly sensitive and selective analysis of low molecular weight volatiles from fruits. Efficiency improvements vary depending on the experimental approach, but this application note demonstrates six-fold throughput enhancements even when utilizing automated incubator hardware optimized for slow chromatographic analysis.

Not only does SIFT-MS deliver higher sample throughputs for routine analysis, but it can also be applied to more rapidly determine optimal incubation times and temperatures in the method development phase.

Figure 1. SIFT-MS headspace analysis of a 1-gram apple sample. More details are given in the text.

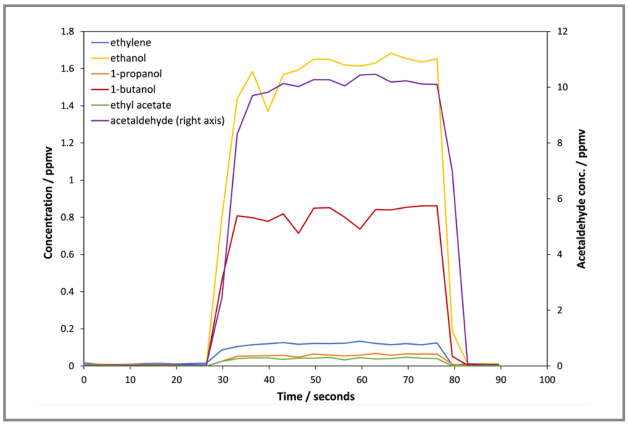


Figure 2. SIFT-MS headspace analysis of several fruits and varieties.

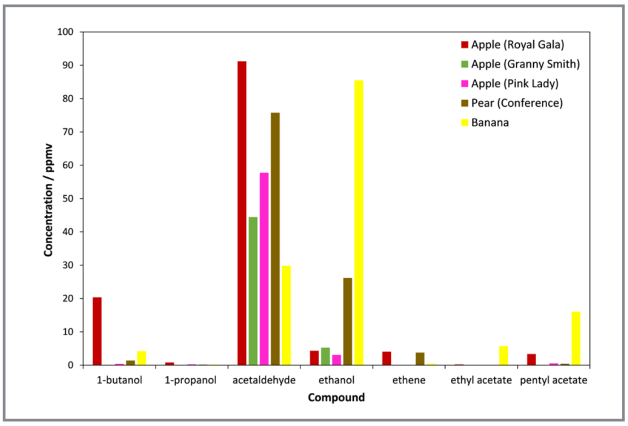
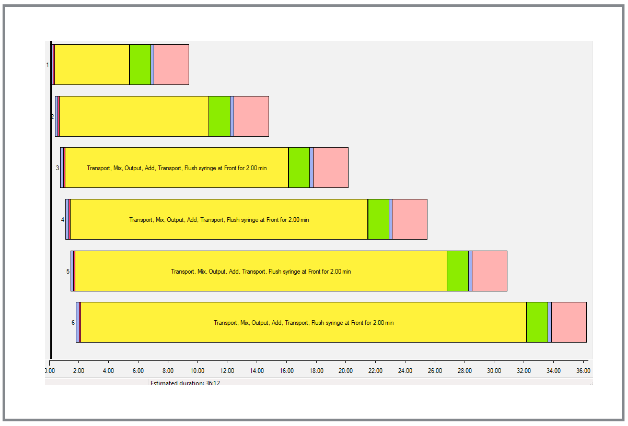


Figure 3. GERSTEL Maestro software illustrates how rapidly direct SIFTMS analysis can determine optimum headspace equilibration time.



REFRENCES

1. Shimadzu Application Datasheet (2015), "Analysis of Ethylene in Food Using GC/MS".

2. B.J. Prince, D.B. Milligan, M.J. McEwan (2010), "Application of [SIFTMS] to real-time atmospheric monitoring", Rapid Commun. Mass Spectrom. 24, 1763; V.S. Langford, I. Graves, M.J. McEwan (2014), "Rapid monitoring of volatile organic compounds: a comparison between gas chromatography/mass spectrometry and [SIFT-MS]", Rapid Commun. Mass Spectrom., 28, 10.

3. For examples of applications of SIFT-MS to food analysis, see:

- N. Sumonsiri and S.A. Barringer (2013), "Application of SIFT-MS in Monitoring Volatile Compounds in Fruits and Vegetables", Curr. Anal. Chem., 9, 631.
- G. Ozcan and S. Barringer (2011), "Effect of Enzymes on Strawberry Volatiles during Storage, at Different Ripeness Level, in Different Cultivars, and during Eating", J. Food Sci., 76(2), C324.
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