SSX SUPER SESI X

(Application note)

FOSSILIONTECH

Chocolate tastes best when you swallow.

Monitoring the dynamics of the nose-space cavity and its effect in the flavor experience of chocolate.

#foodomics #flavor #taste #experience

ChemoSens

Centre des Sciences du Goût et de l'Alimentation

INRAE
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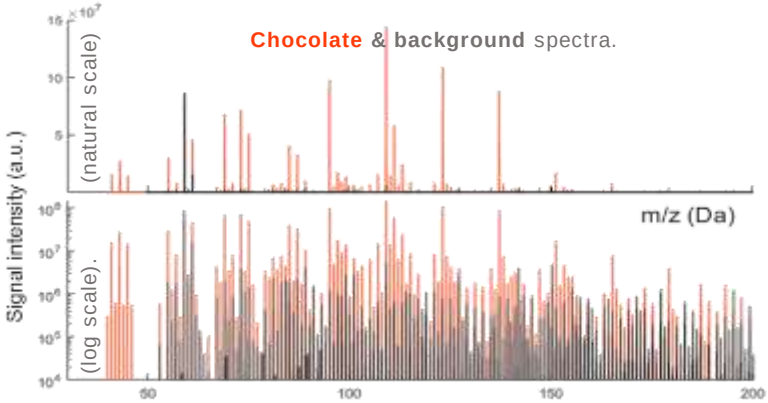
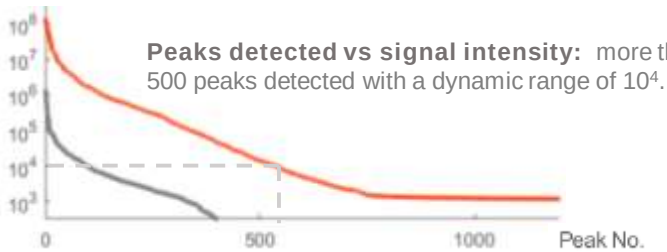
Headspace analysis of chocolate samples:

Methods: to identify the main peaks produced by chocolate, a Super SESI X was coupled with an Orbitrap Exploris 120. Chocolate was introduced in a flask (100 ml) with a custom-designed cap featuring an inlet and an outlet. The flask was heated at 36ºC for 1h to equilibrate the headspace. A flow of Nitrogen was controlled using a SIROCCO unit that incorporates an active charcoal filter and controls the flow rate, the temperature, and the dew point of the gas. The gas outputted by Sirocco swept the flask headspace for 30 seconds to carry it to the Super SESI. The MS collected data in full scan mode. An empty flask was analyzed to capture the background signal of the system.

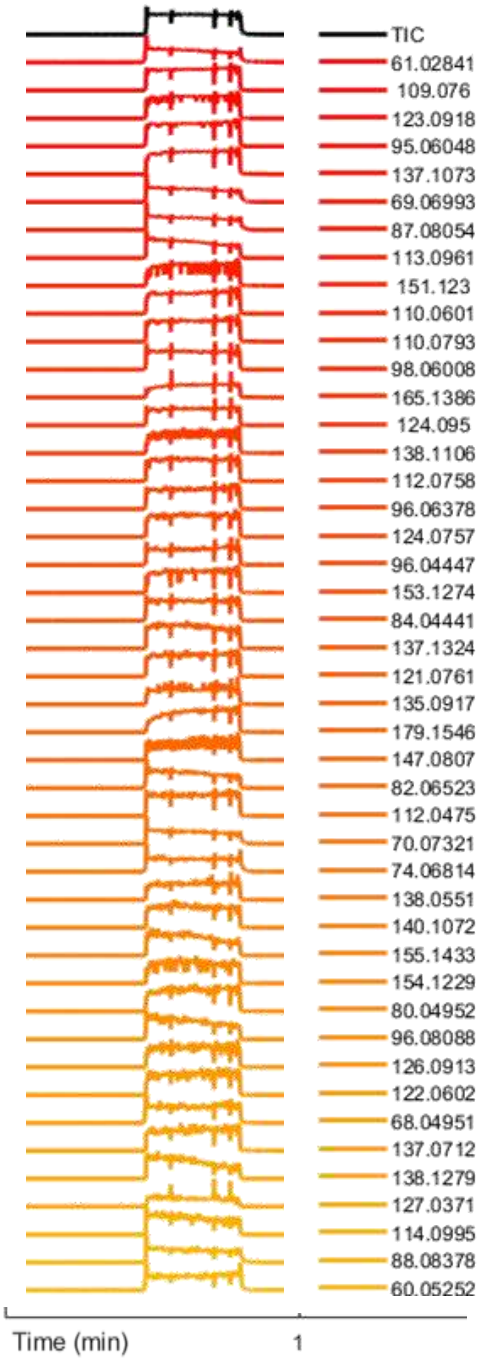


The dominating peaks are shown in natural scale. The log scale shows the depth of the data, with several species covering various orders of magnitude.



Peaks detected vs signal intensity: more than 500 peaks detected with a dynamic range of 10^4.

Results: The background levels of the SIROCCO system are remarkably low compared with the chocolate headspace. This allows for the detection of many compounds with a dynamic range of 104. Most peaks released by chocolate have masses between 40 and 200 Da. In this range, more than 500 peaks were detected after averaging the spectra for the time the headspace gas was introduced in the Super SESI. The time traces of some of the most abundant peaks were plotted along with the TIC curve, and their respective masses.



Time traces of some of the most abundant peaks in chocolate headspace

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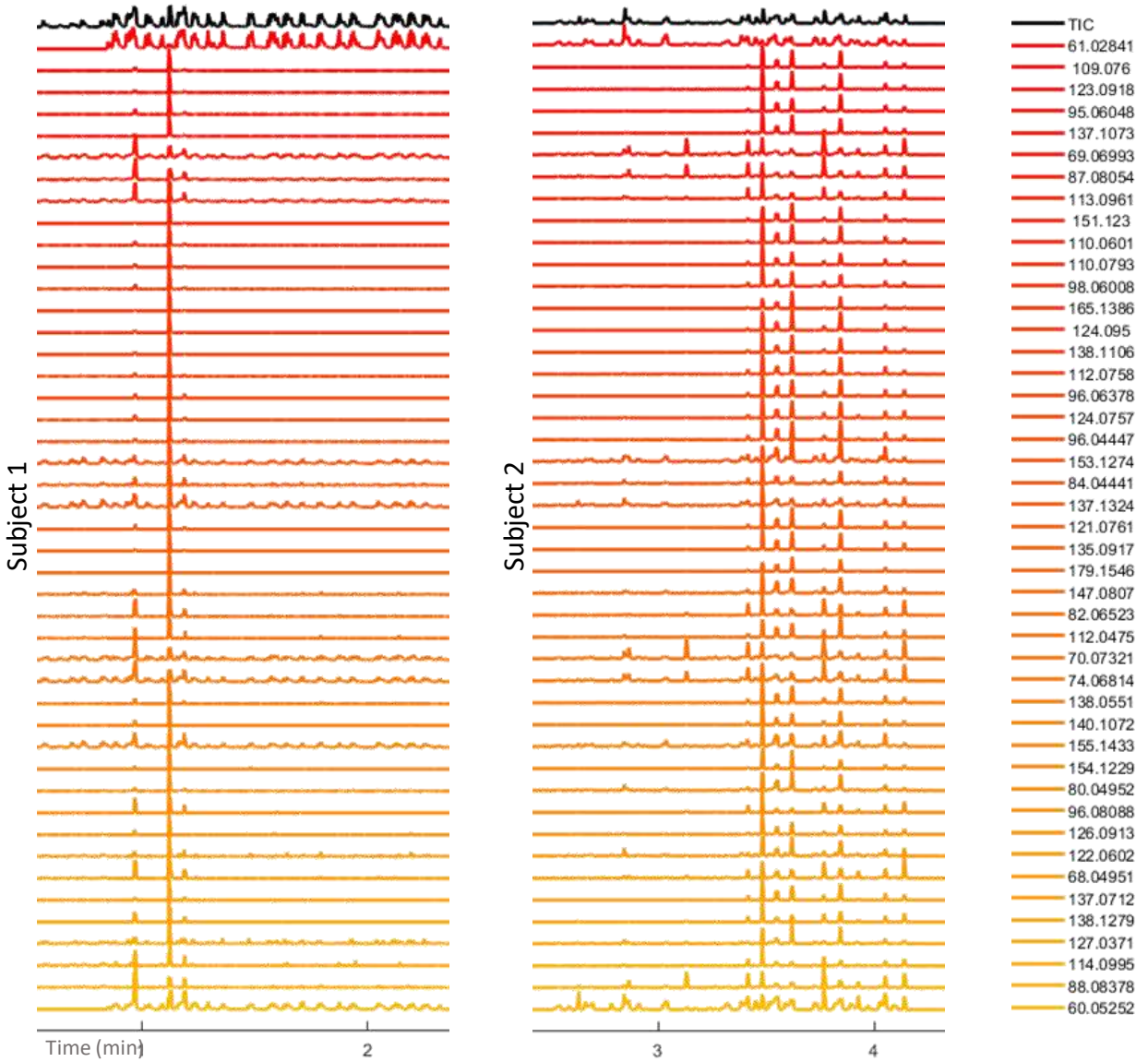
Nose-space *in-vivo* analysis of chocolate eating:

Methods:. Subjects were requested to eat a piece of chocolate while their nose-space cavity was probed and analyzed in real-time with a 3D-printed adaptor connected to the inlet of the SSX. Subjects inhaled room air through the mouth and exhaled through the nose freely. The most abundant chocolate species were detected in the headspace experiments and tracked over time (plotted below). Signals rose during each exhalation.



Results: Some molecules rapidly make their way into the nose-space and are detected after introducing chocolate in the mouth (i.e. Acetic acid, with a mass of 61.0284 Da, which is also detected in natural breath). Other molecules make it to the nose-space cavity only after some exhalations (109.076 and 123.0918 are good examples). For these molecules, the signals produced during some exhalations are substantially high. In these experiments, subject 1 kept a regular breathing pattern (as evidenced by its periodic TIC profile, where the signal goes up each time the subject exhales) and swallowed normally, whereas subject 2 swallowed saliva more often. These experiments indicate that certain maneuvers can have an impact on how molecules reach the nose space, and hence how nuanced flavors are experienced. More tests would be required to assess the effect of the nose-space adaptor.

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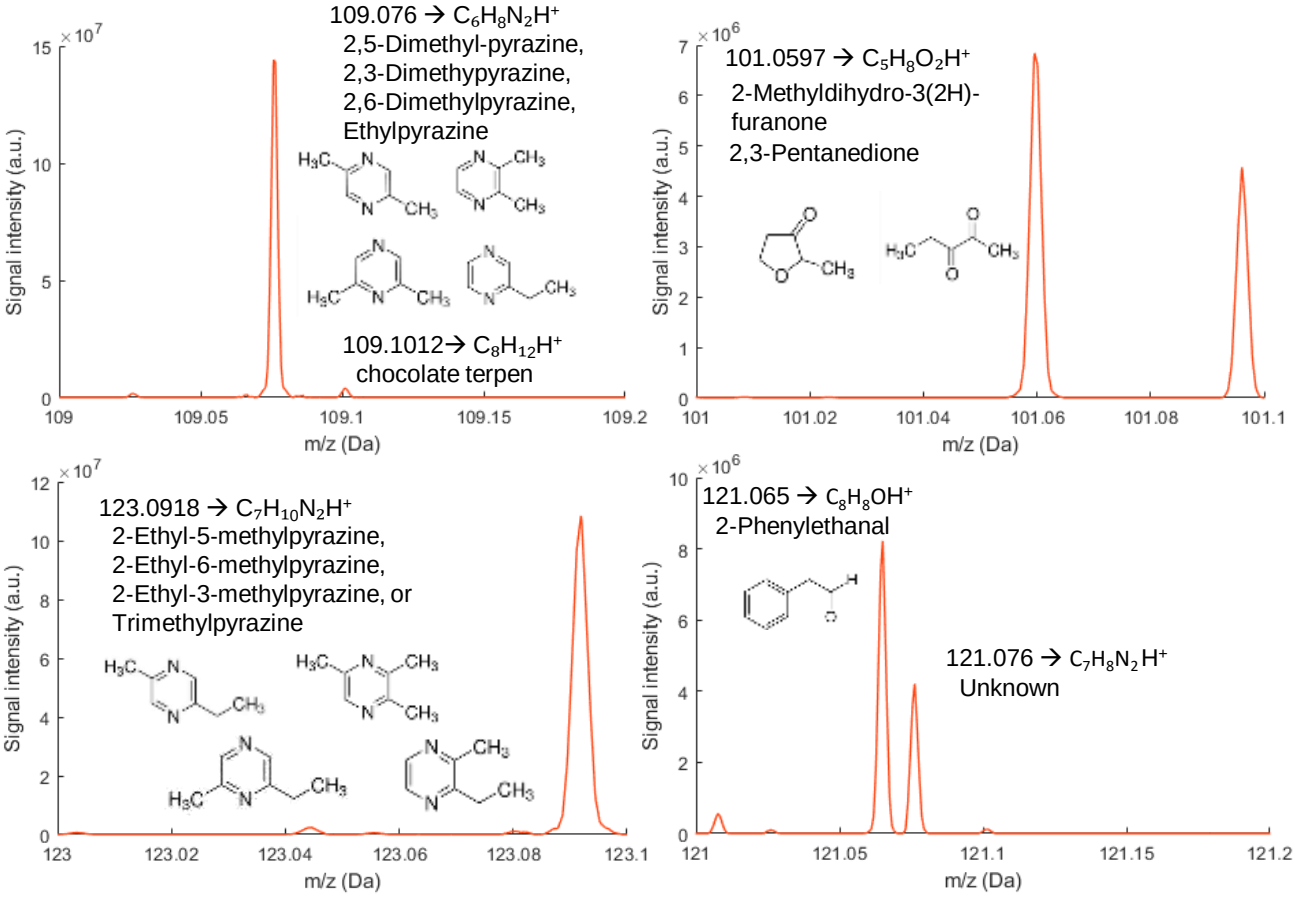
Compound identification:

Of the peaks detected, several compounds were identified in real-time by combining the molecular formula and previous databases for chocolate flavor built by direct injection analysis (Deuscher et al. “Organoleptic properties of dark chocolates investigated by direct-injection mass spectrometry (PTR-ToF-MS) and GC-Olfactometry. Some examples include 101.0597 Da (C_5H8O_2H+) 2-Methyldihydro -3(2H)-furanone, or 2,3-Pentanedione, 109.076 Da (C_6H8N_2H+) Dimethylpyrazine or Ethylpyrazine, 109.1012 Da (C_8H_12H+), a chocolate terpen fragment, or 123.0918 Da ($C_7H_10N_2H+$), an Ethyl-methylpyrazine, or Trimethylpyrazine, or 121.065 Da (C8H8OH+) 2-Phenylethanal. For many more peaks, their molecular formula was automatically generated, but the identity could not be backed by a database. An example of this is 109.1012 Da (C_8H_12H+), a known terpene fragment found in chocolate still to be fully identified. Another example is 121.076 (C7H8N2H+). This shows that there are still many compounds to be identified and cataloged. For this, the Orbitrap Exploris was operated in full scan mode with no fragmentation. The resolution of the instrument was set to 30.000. The data processing software (Xcalibur, by Thermo Fisher Scientific) provides a tentative molecular formula for each peak of interest



Conclusions: We did not observe fragmentation in these experiments. Yet, there are still many compounds to be identified and cataloged. This is because new instruments provide improved resolution and detection limits, which expand the total coverage. Thanks to the extremely soft ionization of SESI, most observed peaks are just protonated. Combined with the high resolution of the orbitrap, this allows for the instant identification of the molecular formula. Further structural details can be obtained by MS-MS analysis.