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Development of a flavor release analysis method on volatile compounds of citrus fruits by DART MS

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BC BioChromato

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Introduction

Gas chromatography is typically used for analysis of the volatile compounds represented by aroma compounds. Nevertheless it's important to find relationship with the results of chemical analysis and perceptive aroma, it is hard to measurement flavor release from foods in real time. DART MS with closed-chamber interface system is effective to monitor volatile compounds within seconds successively. Although there are a lot of structural isomers in volatile compounds and it is difficult to specify a target compound by SIM analysis, we developed a simultaneous analysis method using MRMs of flavor compounds which have the similar structure by LC-MS/MS at last this conference.
Here, we tried to measure the flavor release phenomena on volatile compounds of several kinds of citrus fruits.

Methods and Materials

Limonene, gamma terpinene, octanal, linalool and alpha terpineol were used for volatile compounds. Limonene and gamma terpinen (molecular weight 136), linalool and alpha terpineol (molecular weight 154) are a structure isomer, respectively. Triple quadrupole mass spectrometer LCMS-8030 (Shimadzu Corporation, Kyoto, Japan) was used for the analysis of these components. All compounds were ionized by APCI except for octanal, but all compounds were ionized with the DART (DART-OS ion source; IonSense Inc., MA, USA), so MS conditions like compound-dependent parameters and MRM transitions of each compound were optimized using volatile compounds analysis system with DART.
Next, the DART-MS/MS system was coupled with closed interface, Volatimeship (Bio Chromato, Inc., Japan) in order to achieve high sensitivity and real time flavor response and then several kinds of citrus fruits were analyzed.

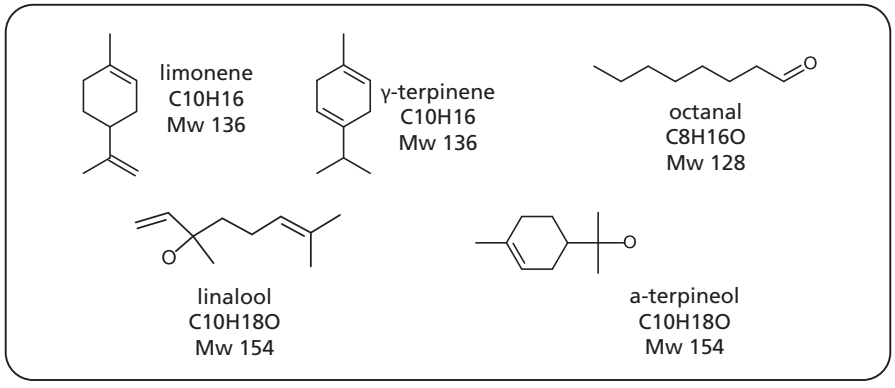

Figure 1 Structure of volatile compounds in this study

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UFMS

Ultra Fast Polarity Switching
- 15msec

Ultra Fast Scanning
- 15,000 u/sec

Ultra Fast MRM
- Max. 555 transition /sec

Figure 2 LCMS-8030 triple quadrupole mass spectrometer

Result

Method development for volatile compounds

Five volatile compounds were used for this study because they were known as characteristic voltile compounds in citrus fruits. Limonene has an aroma of an orange peel, octanal has a sweet aroma of citrus with low concentration, and linalool has an aroma of flower. Regarding ionization ability of these components, all compounds (their volatile) were ionized by DART. These compounds were interestingly ionized, that is, they had detected not molecular related ions (M+H) but several ions which might be fragment ions; precursor ion signals of limonene were detected by positive m/z 135, 151, 153 and terpinene, 170, 186. All compounds were ionized high-sensitively at positive ion mode. For improvement of the selectivity of these compounds, MRM transitions of each compound were set using volatile compound analysis with DART. This was very easily implemented by only bringing sample vials close to the DART ionization area without injection. Then we found each compound specific MRM transitions; terpinene was detected intensively at Q1/Q3=170/153 (positive) which transition could specify terpinene.

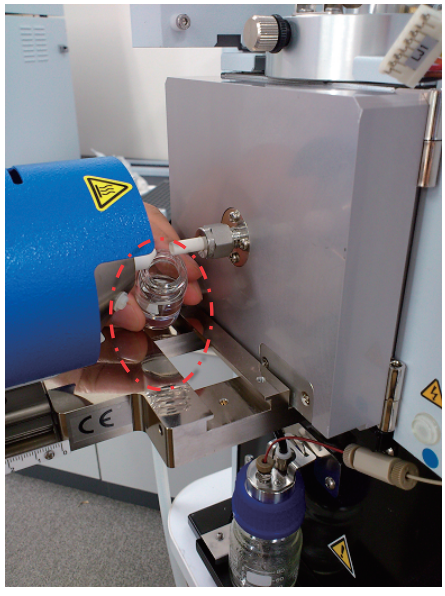

Figure 3 Standard sample (vapor) analysis by DART-MS

Sample vial opened its cap was placed under DART gas flow.

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limonene

a-terpineol

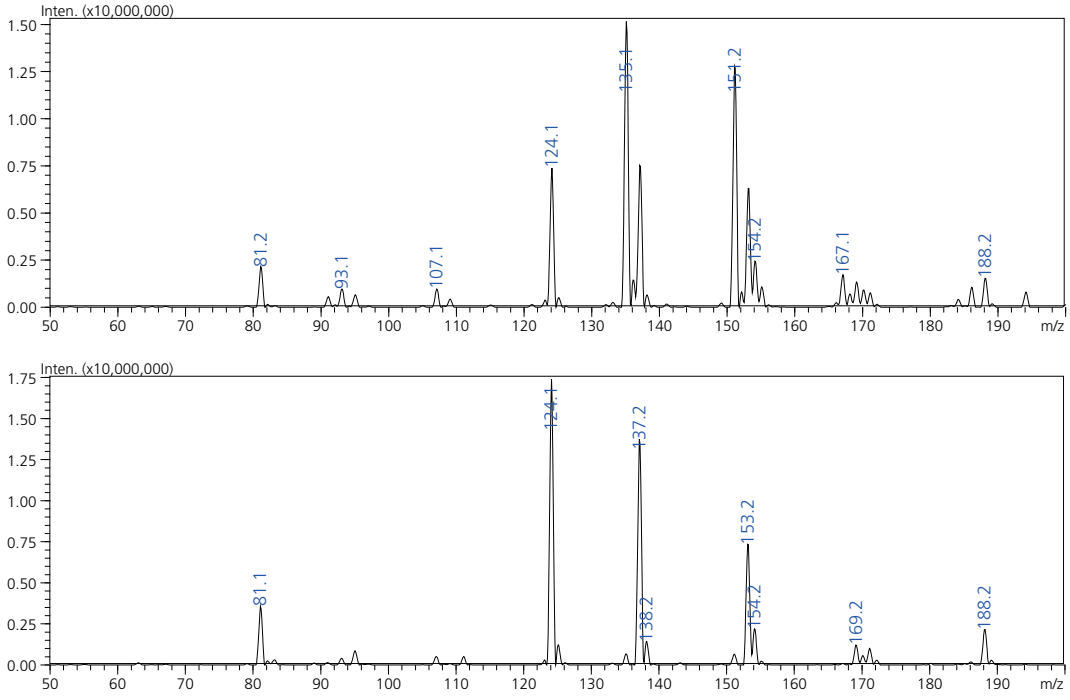

Figure 4 Positive mass spectra of limonene and a-terpineol

Analysis by DART-MS

Next, the DART-MS/MS system was coupled with closed interface in order to achieve high sensitivity and real time flavor response and then several kinds of citrus fruits were analyzed. Whole citrus fruit were put in the closed sample cage and crushed, with analyzing the volatile compounds released from citrus fruit by closed DART system. Shonan Gold (local citrus fruit in Japan) and orange were analyzed successively.

Figure 5 Shonan Gold

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Total analyzing time for each citrus fruit is within 5 minutes. Terpinene, Limonene, Linalool and Terpineol were detected at citrus fruits set into sample flask and their intensity were changed just after squeezing the fruits. The changes were different from each fruit. This system detected flavor compounds within sub-second resolution and may be useful for breeding and singularity analysis of flavor fruits.

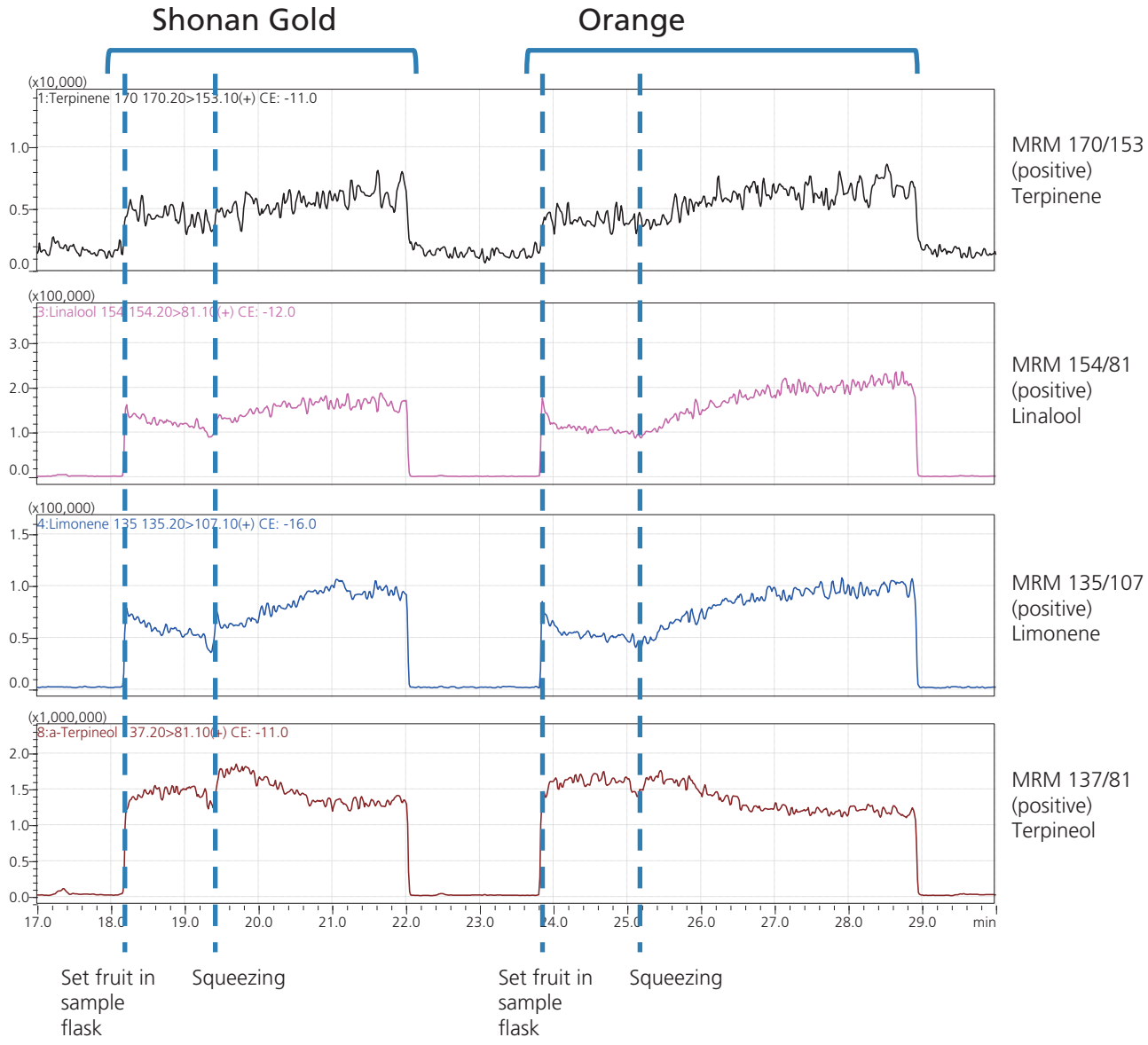

Figure 5 MRM chromatograms of Shonan Gold and Orange

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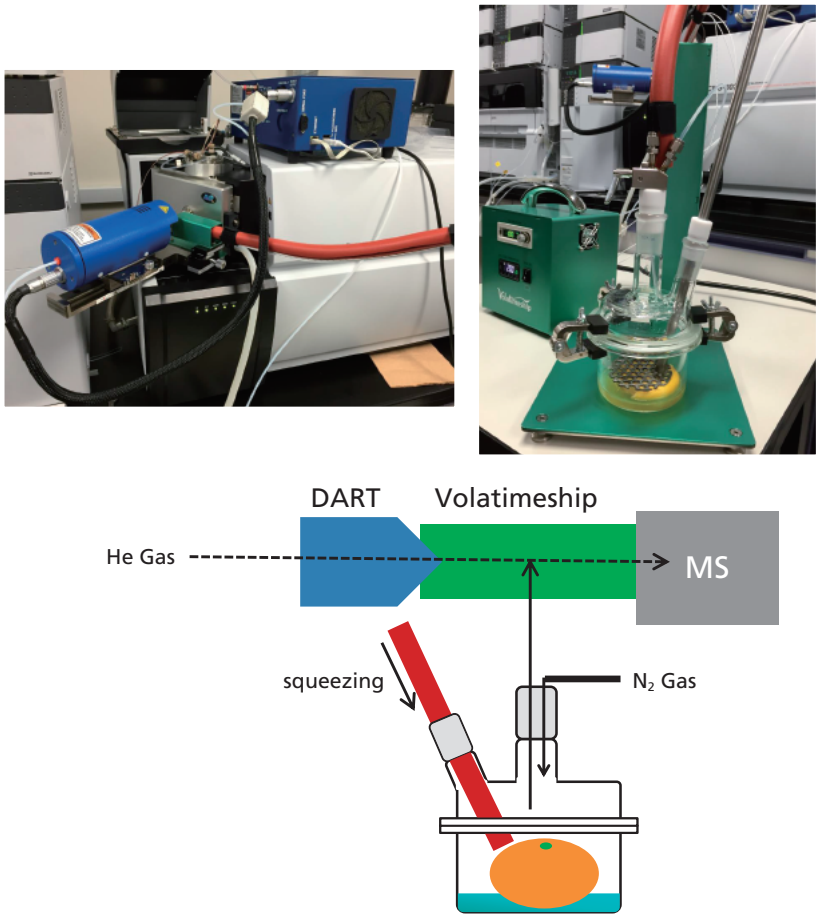

Figure 6 DART & Volatimeship System

Conclusions

Volatile compounds in citrus fruits could be detected with real time analysis by closed DART MS/MS system.

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