Qualitative Analysis of Peach Juice by using HS-SPME-GC-QMS

Product used: Mass Spectrometer (MS)

Introduction

Food flavor compounds are known to be an important element of good taste, and off-flavor compounds, such as rancid odors, are also an important element of food quality. Gas chromatography-mass spectrometry (GC-MS) is often used to analyze these food flavor components due to the high volatility of flavor components and the complex composition of many components.

Qualitative analysis by GC-QMS is generally performed by searching the library database (DB) using the measurement data of the Electron Ionization (EI). However, if qualitative analysis is performed using only the similarity with the library spectrum as an index, several significant candidates may be obtained depending on the compound, or erroneous candidates may be selected as the identification result. In such cases, confirmation of molecular ions by the soft ionization (SI) such as the photoionization (PI) is effective. Therefore, in 2021, we released msFineAnalysis iQ, an integrated qualitative analysis software that automatically combines the analysis results of EI and SI methods measured by GC-QMS. Furthermore, in August 2024, we released msFineAnalysis iQ Ver.2, which includes a target analysis function for rapidly searching for specific compounds such as off-flavor components, additives, isomers, and structural analogs. Using this software, high-quality qualitative analysis results can be obtained.

In this MSTips, we report the analysis results of aroma components in peach juice using a gas chromatography quadrupole mass spectrometer (GC-QMS) and msFineAnalysis iQ. Additionally, we present the results of target analysis of γ-lactones, aroma components characteristic of peaches, using the new functionality target analysis function.

Experimental

Commercially available 100% peach juice was used as sample. Peach juice 10 mL was sealed in a 20 mL vial. The SPME mode of the HT2850T autosampler (HTA S.R.L.) was used as the sample preparation device, and volatile components in the headspace area of the vials were targeted for the measurement. A GC-QMS (JMS-Q1600GC UltraQuad™ SQ-Zeta, JEOL Ltd.) was used for the measurement (Figure 1). We performed HS-SPME-GC-QMS measurements using both EI and photoionization (PI) modes with a combination EI/PI ion source. The qualitative data processing was performed with msFineAnalysis iQ (JEOL Ltd.). Detailed measurement conditions are shown in Table 1.

Figure 1 JMS-Q1600GC with HT2850T autosampler

Table 1 Measurement conditions

SPME	
SPME Fiber	50/30 µm DVB/CAR/PDMS 2mm (Merck)
Sample amount	10 mL
Extraction temp.	50 °C
Extraction time	30 min
Desorption time	3 min
GC	
Column	ZB-WAX (Phenomenex) 30 m×0.25 mm I.D., df=0.25 µm
Inlet	250℃, EI=Split 20:1, PI=Splitless
Oven	40℃ (2 min) →10℃/min→250℃/min (5min)
Carrier flow	He, 1.0 mL/min (Constant Flow)
MS	
Ion Source	EI/PI combination ion source
Ionization mode	EI+ (70 eV, 50 µA), PI+ (D2 lamp, 8～10 eV)
Mass range	*m/z* 33-500 (Scan mode)

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Results and Discussion

Results of Non-Targeted Analysis of Aroma Components

Figure 2 shows the TICC of EI data and the annotation results for the top seven highest intensity compounds. High intensities of aroma compounds were detected such as alcohols (e.g., ethanol, 2-hexenol), esters (e.g., ethyl acetate), aldehydes (e.g., furfural, benzaldehyde), and γ-lactones (e.g., 2(3H)-furanone, 5-hexyldihydro- (γ-decalactone)). Among them, lactones are known to be characteristic aroma compounds of peaches, generated during the softening of peach fruit[1]. Therefore, to conduct a detailed analysis of γ-lactones, this data was subjected to targeted analysis.

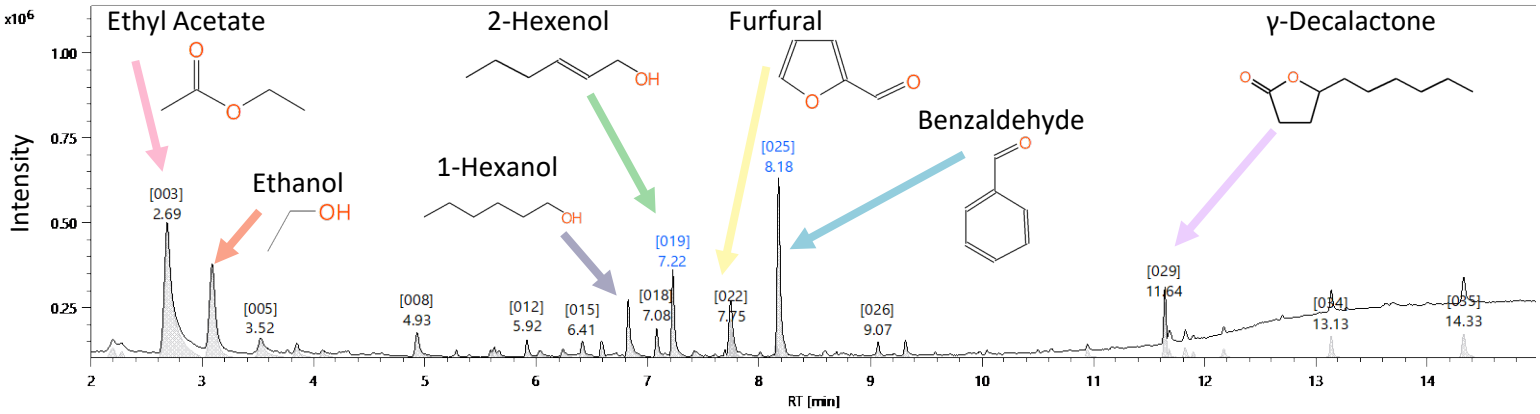

Figure 2 Total ion current chromatogram of EI data

Preparation for Target Analysis of γ-Lactones

Figure 3 shows the EI mass spectrum and structure formula of γ-decalactone, a γ-lactone detected at high intensity in this study. *m/z* 85 was detected as the base peak in the mass spectrum, indicating the desorption of an alkyl chain in γ-decalactone. Since γ-lactones is thought to have *m/z* 85, we decided to perform targeted analysis on components with an *m/z* 85. The targeted analysis function in msFineAnalysis iQ automatically searches for compounds using molecular ion and fragment ion information, as well as *m/z* values registered in advance in a target list. The target list used in this analysis is shown in Figure 4.

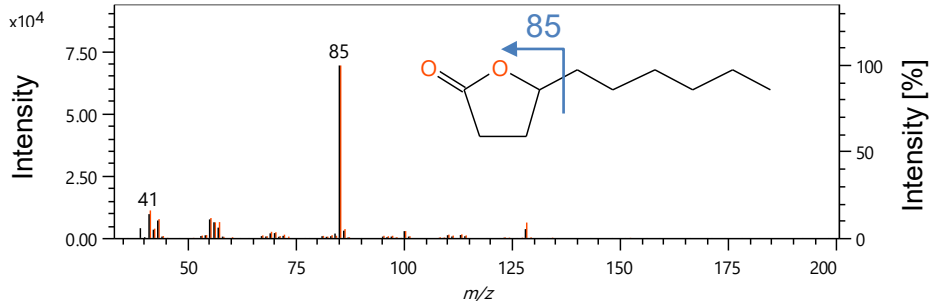

Figure 3 EI mass spectrum of γ-Decalactone
(Black: Measured mass spectrum, Red: NIST DB mass spectrum)

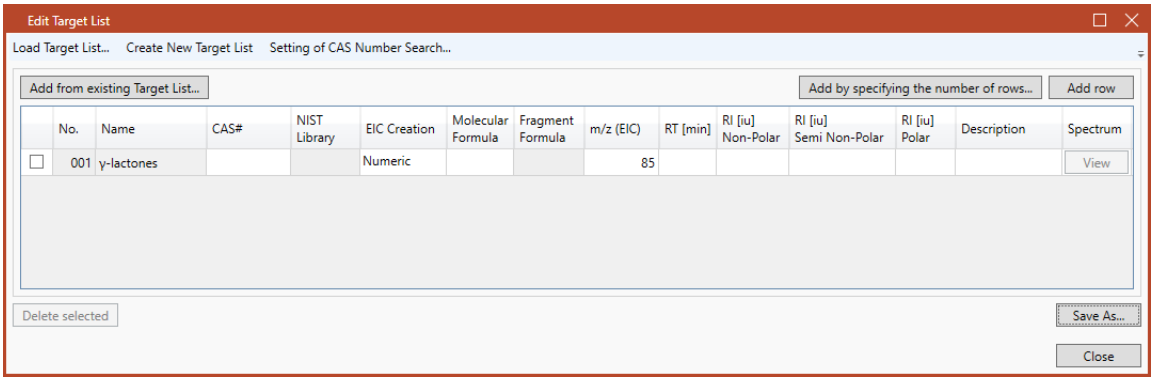

Figure 4 Target list

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Target Analysis Results for γ-Lactones

Figure 5 shows the target analysis results for γ-lactones. In this study, four compounds containing *m/z* 85 were detected (ID: C001, C002, C003, C004). However, it is difficult to estimate whether these compounds are γ-lactones based solely on chromatographic information. Therefore, integrated qualitative analysis was carried out simultaneously. As an example, the integrated qualitative analysis result of C002 is shown in Figure 6. This component was presumed to be γ-octalactone because it had a good match factor of 908 with 2(3H)-Furanone, 5-butyldihydro- (γ-octalactone) in the NIST DB search, with a retention index error (ΔRI) of 45 [iu]. The other components were also subjected to the integrated qualitative analysis, and all four components were confirmed to be γ-lactones (Figure 5). Although C002 and C004 are minute peaks and could be overlooked when exhaustively analyzed by non target analysis, they could be detected by utilizing the target analysis function of msFineAnalysis iQ.

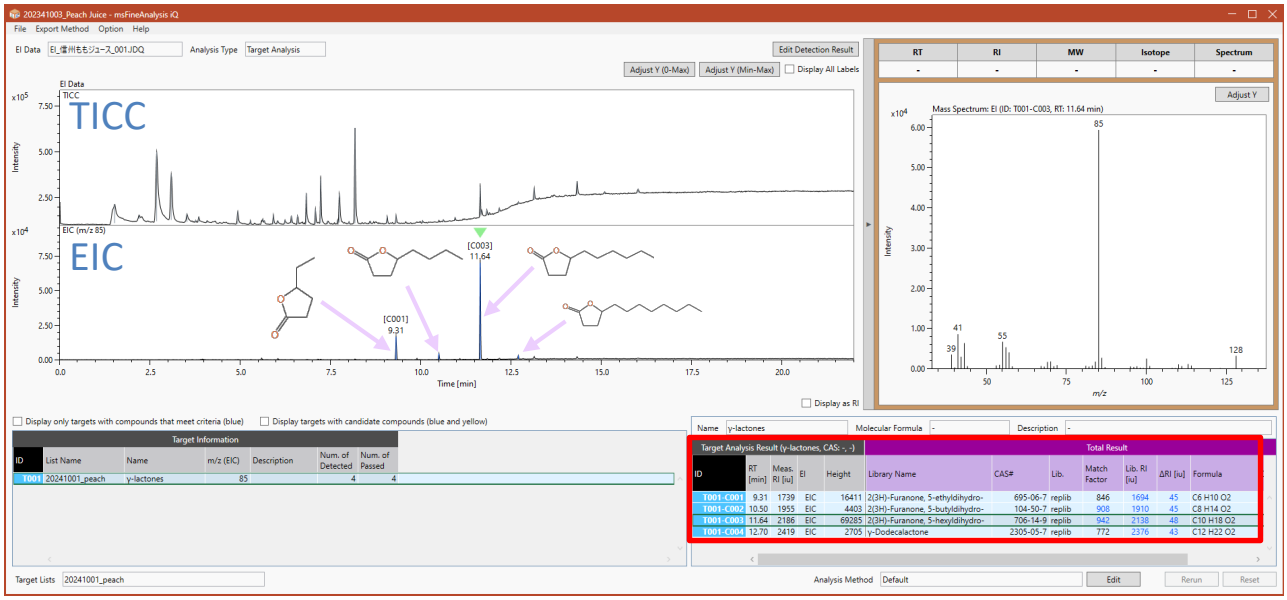

Figure 5 Target analysis result

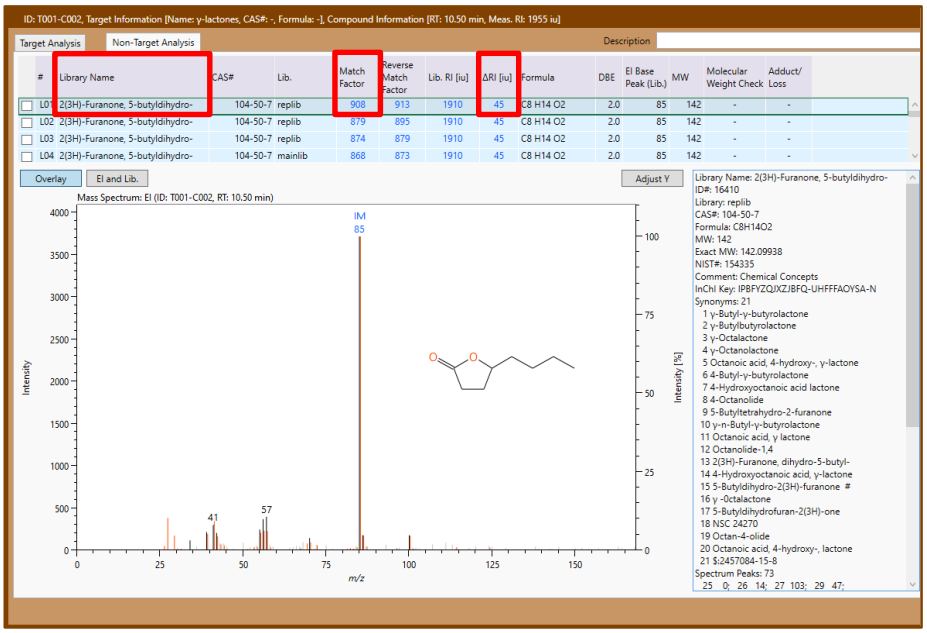

Figure 6 Integrated qualitative analysis result of C002

Conclusions

In this MSTips, we reported the analysis results of aroma components in peach juice using a GC-QMS and msFineAnalysis iQ. As the result, high intensities of aroma compounds were detected such as alcohols , esters , aldehydes , and γ-lactones. Furthermore, target analysis for γ-lactones characteristic aroma compounds of peaches. Although some of the detected compounds were minute, the software is also capable of automatically performing integrated qualitative analysis, and by using this result in combination, the final confirmation was made that four γ-lactone components were present. This software is expected to improve the qualitative accuracy and efficiency of qualitative analysis in foods using GC-QMS.

Reference

1) Kakiuchi, N., et al. "Effect of holding temperature on the development of volatile constituents of white peaches." *Journal of the Japanese Society of Food Preservation Science*, vol. 17, 1991, pp. 14. (In Japanese)

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