Application News

GC-MS GCMS-TQ™8040 NX

Analysis of Fragrance Components in Aroma Oils Using GC/MS Off-Flavor Analyzer

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User Benefits

- Using GC/MS Off-Flavor Analyzer, components analysis can be easily performed even on non-odorous fragrances.
- The fragrance of samples can be easily evaluated by the sensory information registered in the database of GC/MS Off-Flavor Analyzer.
- By using GC-MS/MS, more reliable identifications are possible even for samples containing contaminants.

■ Introduction

Fragrances are known to have various effects such as promoting relaxation, masking unpleasant odors, and increasing appetite.

Therefore, the importance of fragrance is emphasized in many fields, including daily necessities, cosmetics, and foods. While sensory analyses by professionals are often used to evaluate fragrances, analyses with gas chromatography enable detailed components analyses of complex fragrances.

GC/MS Off-Flavor Analyzer is a database of expert information for odor analyses. It is also known that odor-causing substances produce pleasant smells as well, depending on their concentrations and compositions. In this article, we applied GC/MS Off-Flavor Analyzer to the analyses of non-odorous fragrances. Belows are the examples of how we used GCMS-TQ8040 NX gas chromatograph mass spectrometer to investigate the characteristics of aroma oil fragrances.

■ GC/MS Off-Flavor Analyzer

Although GC/MS is widely used for odor analyses, it requires a great deal of effort to determine the analysis conditions and to perform post-analysis. GC/MS Off-Flavor Analyzer is a system that allows the efficient analysis of the odor-causing substances. Fig. 1 shows the analysis flow of the GC/MS Off-Flavor Analyzer. The system is compatible with three types of liquid-phase columns. It can also be used for analysis with pretreatment techniques, such as MonoTrap, solid-phase microextraction, and headspace sampling, and supports analyses using a sniffer.

Furthermore, the calibration curve information can be registered after the analysis of the sample for correction, which enables approximate quantifications of detected components.

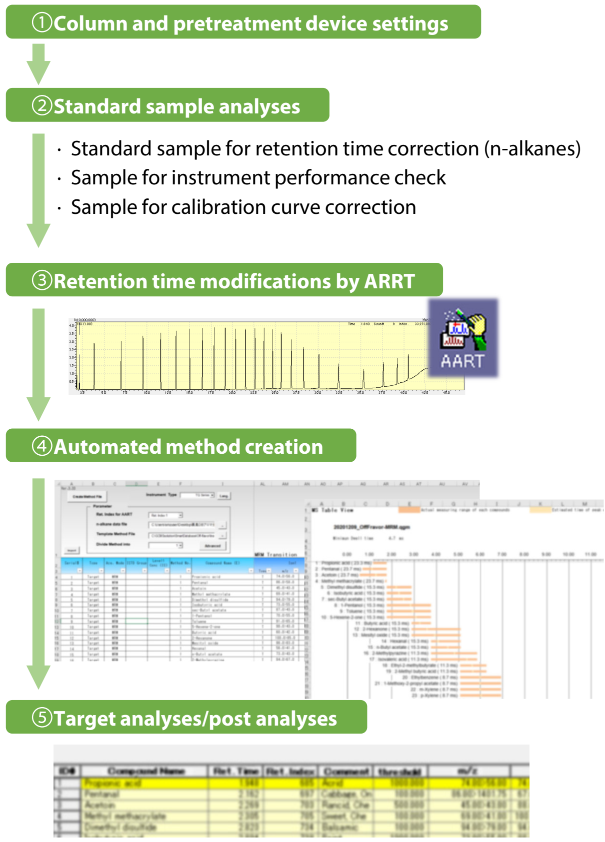


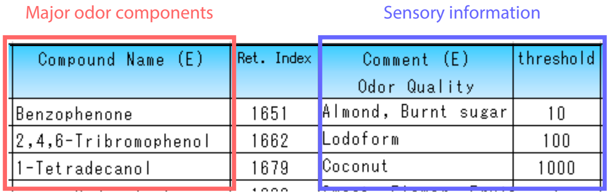
Fig. 1 Analysis Flow with GC/MS Off-Flavor Analyzer

Table 1 Instrument Configuration and Analysis Conditions

Auto sampler	: AOC™-20s Plus	**[AOC]**	
Auto injector	: AOC-20i Plus	Injection volume	: 1 µl
Instrument	: GCMS-TQ8040 NX		
[GCMS]			
Flow control mode	: Pressure (44.5 kPa)	Column temp. program	: 50 °C (5 min) → 10 °C/min → 250 °C (10 min)
Injection mode	: Split (1 : 5)	Interface temp.	: 250 °C
Carrier gas	: He	Ion source temp.	: 200 °C
Purge flow rate	: 3 mL/min	*m/z* range	: 45-500 amu
Column	: InertCap 5MS/Sil (30 m × 0.32 mm I.D., 0.5 µm)	Event time	: 0.1 min (scan) 0.3 min (SIM/MRM)

■ Analysis Conditions

Table 1 shows the instrument configuration and analysis conditions used. Note that GC/MS Off-Flavor Analyzer supports both SIM and MRM analyses. We performed simultaneous scan-SIM and scan-MRM analyses using the analysis conditions registered in GC/MS Off-Flavor Analyzer. Here we adopted a simple test by directly injecting liquid samples.



Compound Name (E)	Ret. Index	Comment (E) Odor Quality	threshold
Benzophenone	1651	Almond, Burnt sugar	10
2,4,6-Tribromophenol	1662	Lodoform	100
1-Tetradecanol	1679	Coconut	1000

Fig. 3 A Part of Database in GC/MS Off-Flavor Analyzer

■ Analysis of Aroma Oils

Four commercially available aroma oils (Jasmine, Lavender, Rose, and Citrus) were diluted to 0.1 % with ethanol and then analyzed.

The graphs in Fig. 2 shows the ratios of major components and fragrance characteristics obtained by SIM and MRM analyses. The vertical axes refer the ratios based on the value of the largest component in each analysis.

These graphs show major 8 components quantified by MRM analysis, excluding those detected only by SIM analysis.

The notes in the graphs show the "odor quality" of each components. As shown in Fig.3, the fragrance characteristics of detected components can be easily confirmed by the "odor qualities" registered in the database of GC/MS Off-Flavor Analyzer. For example, the components detected in Jasmine had fragrances suggestive of plants. It is also found that Lavender had many refreshing fragrances such as mint, camphor, and anise, while Citrus, as its name suggested, contained a high level of citrus fragrance.

Fig. 2 Ratios of Major Components and Flavor Characteristics of Four Aroma Oils
Vertical axis: ratio based on the value of the largest component in each analysis

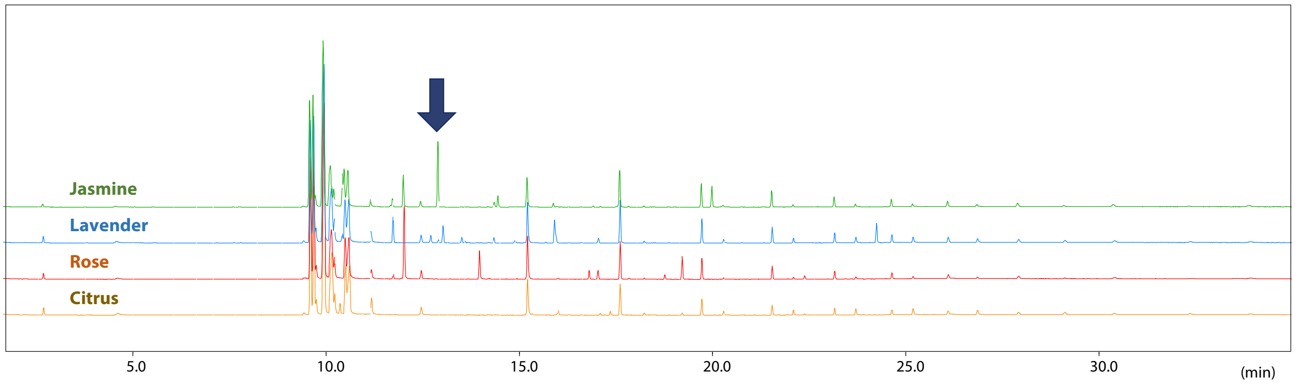


Fig. 4 TIC Chromatograms Obtained by Scan Analyses of Four Aroma Oil Samples (0.1 %)

Using the automated method creation tool of the GC/MS Off-Flavor Analyzer, users can easily configure SIM and MRM analysis conditions. Furthermore, scan analysis and SIM or MRM analysis can be performed at the same time. Fig. 4 shows the TIC chromatograms of four aroma oil samples obtained by simultaneous scan-MRM analyses. With a chromatogram from scan analysis, the peaks can be identified based on a widely-used mass spectral library search. For example, the distinctive peak (arrow in Fig. 4) in the chromatogram of Jasmine was identified as benzyl acetate through the library search. Benzyl acetate is a major ingredient of essential oils of flowers such as jasmine, exhibiting a sweet scent. As in this example, simultaneous scan-SIM/MRM analysis allows us to identify even the components not registered in the database of GC/MS Off-Flavor Analyzer.

■ Accurate Identifications by MRM Analysis

Fragrances are diversified by the combination of many components. Furthermore, perfumes often contain nature-derived ingredients such as plants, which contain a lot of contaminants. These facts make it difficult to analyze the composition of fragrances. For analysis of such samples, MRM analysis is more suitable than SIM analysis because of its higher selectivity. Below shows comparisons between SIM and MRM analyses.

N-decanal in Jasmine sample (Fig. 5) was not identified in SIM analysis due to the mismatch of the ion intensity ratio, but was identified in MRM analysis. In addition, n-dodecanal (Fig. 6) was identified in SIM analysis because the ion intensity ratio was within the acceptable range, but not identified in MRM analysis.

In SIM analysis, contaminants can affect the peaks of the target ions, preventing accurate identification. In the two examples above, it is assumed that MRM analysis, the two steps of fragmentation enabled more accurate identification.

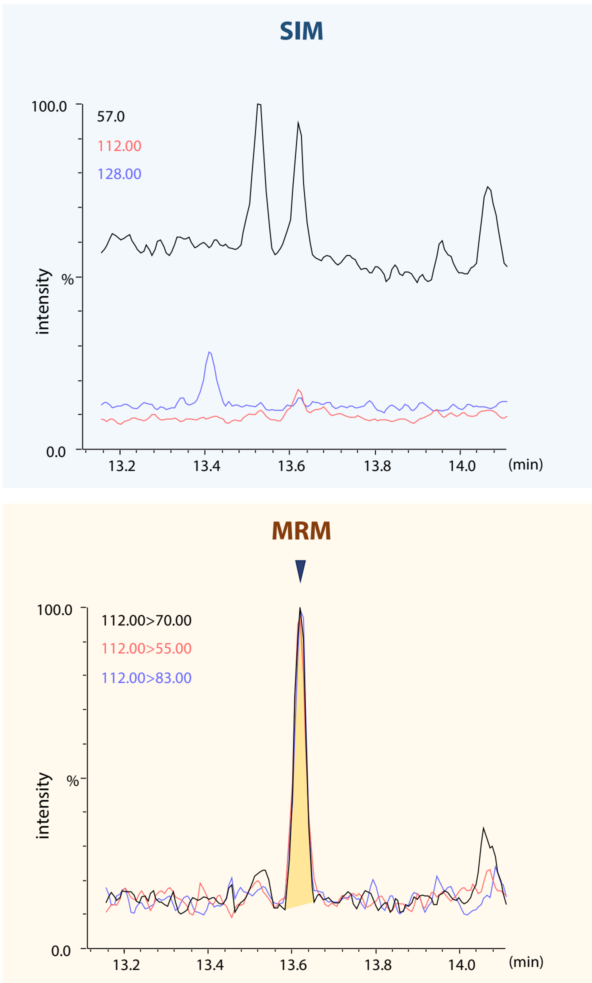


Fig. 5 Chromatograms of n-decanal in Jasmine Sample

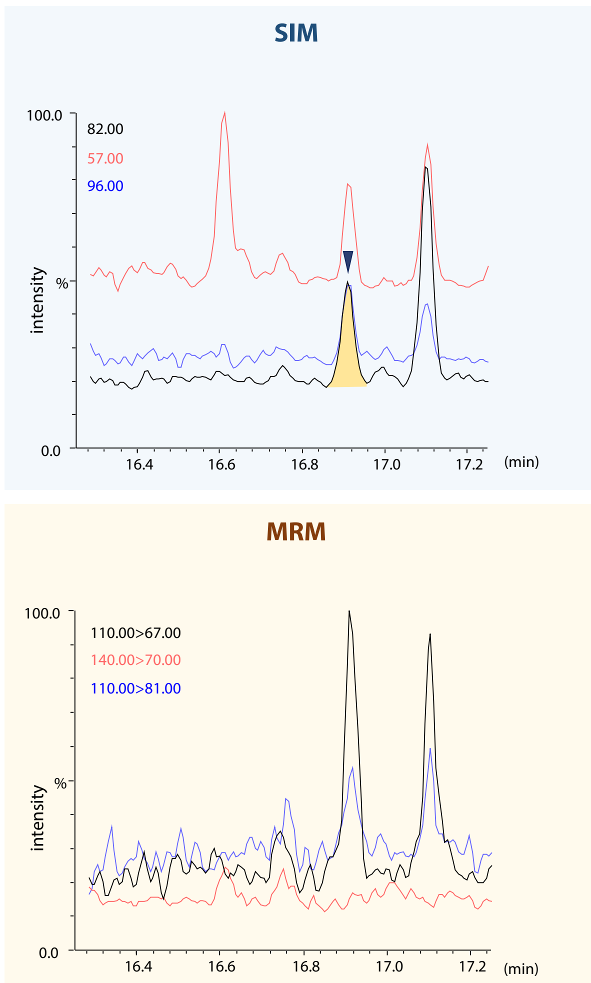


Fig. 6 Chromatograms of n-dodecanal in Jasmine Sample

We next analyzed Jasmine sample intentionally contaminated with a small amount of mineral oil, imitating a nature-derived sample with contaminants. Fig. 7 shows the chromatograms. In the SIM analysis, the presence of mineral oil affected the ion peak of α-terpineol. MRM analysis, however, enabled correct identification even in the sample spiked with mineral oil.

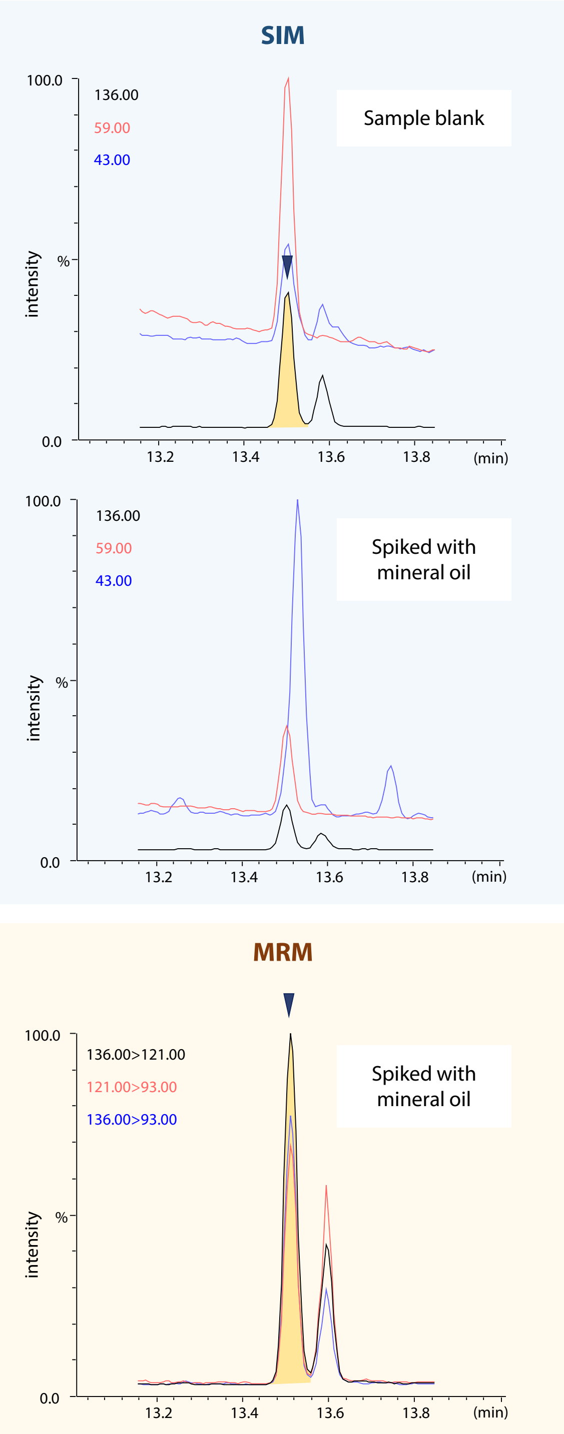


Fig. 7 Chromatograms of α-terpineol in Jasmine Sample

■ Conclusion

Commercially available aroma oils were analyzed with GCMS-TQ8040 NX gas chromatograph mass spectrometer (Fig. 8). Using GC/MS Off-Flavor Analyzer, we could easily confirm the fragrance characteristics of four different aroma oils. Simultaneous scan and SIM/MRM analysis enabled qualification of components not registered in the database of GC/MS Off-Flavor Analyzer.

Furthermore, although compositional analysis of perfumes such as aroma oils is sometimes difficult due to the nature-derived contaminants, the use of MRM analysis provided highly reliable identifications of components. It resulted in more accurate evaluation of the fragrances even with complex components.

Fig. 8 GCMS-TQ™8040 NX + AOC™-20i+s

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