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Application News

GC-MS GCMS-QP2050

Analysis of Essential Oil Using GC-MS/FID Detector Splitting System

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User Benefits

- The detector splitting system allows for simultaneous acquisition of MS and FID data in a single measurement.
- Even complex samples can undergo simplified statistical analysis using the eMSTAT Solution™.
- The combination of the compact Brevis™ GC-2050 and GCMS-QP2050 enables space-saving in the laboratory.

■ Introduction

Hinoki (Japanese cedar) has been used as a timber alongside cedar for a long time. It is easy to be processed and highly durable, making it an important resource in construction. Additionally, Hinoki contains various aromatic components, suggesting the potential for people to feel a sense of preference and comfort in wooden structures. Shimadzu Corporation is involved in Kyoto Prefecture Model Forest Movement, contributing to the maintenance of "Shimadzu Corporation Forest" and aiming to contribute to the community and preserve biodiversity. The Hinoki oil and diffuser made from thinned wood from this forest won the Brands & Communication Design category of the prestigious international design award "Red Dot Design Award 2021" in Germany. This is the first time our company has won in this category.

In this application, five types of Hinoki oils, including Hinoki oil made by our company and Hinoki oils from different regions, were analyzed using the detector splitting system between MS and FID. The results from MS were compared using eMSTAT Solution to analyze the composition of the components. Additionally, the quantification of δ-Cadinene, an important aromatic component that is a sesquiterpene was conducted using FID, which showed high calibration linearity over a wide concentration range.

Furthermore, with a compact width of approximately 63 cm, the device configuration of Brevis GC-2050 + GCMS-QP2050 not only contributes to space-saving in the lab but also demonstrates high analytical performance.

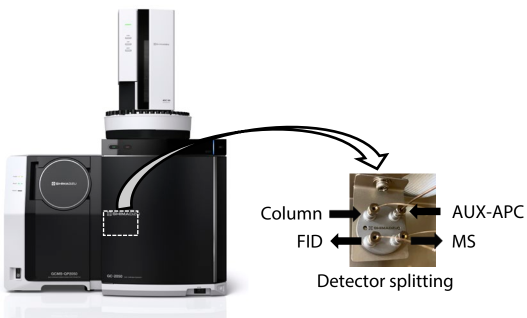


Fig. 1 Detector Splitting System

■ Essential Oil Analysis Using GC-MS/FID

In quality control (QC) of fragrances like Hinoki oil, where a wide concentration range is typically expected, GC-FID, which provides detector response over a broad range, is commonly used. By comparing the chromatogram shapes of product samples obtained during the production process with approved reference samples, the component profile is confirmed. However, in the case of Hinoki oil containing numerous components, qualitative analysis of each peak becomes challenging with an FID detector. Therefore, by using the detector splitting system between MS and FID, it is possible to obtain data from both MS and FID simultaneously in a single measurement. This allows for the usual confirmation of chromatogram shapes with FID and enables qualitative analysis with MS when unknown components are detected.

■ System Configuration and Analysis Conditions

The system configuration and analysis conditions are shown in Table 1 and 2, respectively In this analysis, SMI (Smart Micro Inert) FLOW DEVICE 2-Way Splitter for detector splitting was used (Fig. 1). The SMI FLOW DEVICE is a capillary flow device for analysis with features such as low leakage, low dead volume, low heat capacity, and inertness. This allows for the analysis of highly adsorptive components. We adjusted the inner diameter of the resistors used for MS/FID detector Splitting, as well as the pressure of the AUX-APC (Digital Flow Controller), to set the split ratio at approximately 1:3 for MS:FID. By using eMSTAT Solution for analysis, we visually grasped the differences in component characteristics of each Hinoki oil and identified the compounds contributing to those differences.

Table 1 System Configuration

GC Model	: Brevis GC-2050 / AOC™-30i
MS Model	: GCMS-QP2050 (TMP exhaust: 255 L/sec)
Injection Port	: SPL
Column	: SH-I-5Sil MS (P/N : 221-75954-30) (30 m × 0.25 mm I.D., df= 0.25 µm)
Detector splitting	: SMI FLOW DEVICE 2-Way Splitter with APC (P/N : 221-88100-43)
Software	: Labsolutions™ GCMS eMSTAT Solution

Table 2 Analysis Conditions

GC	
Injection Temperature	: 250℃
Flow Control Mode	: Pressure (He 230 kPa)
Initial Flow Rate	: 3.24 mL/min
Purge Flow	: 5 mL/min
Injection Volume	: 0.8 µL
Split ratio	: 10
Column Oven Temp. Program	: 80℃ → 5℃/min → 230℃
AUX-APC	: He 40 kPa
Resistance tube	: 1.7 m × 0.15 mm I.D. (MS) 2.0 m × 0.25 mm I.D. (FID)
Split ratio	: MS: FID of 1:3.2
Initial Flow Rate	: 1.66 mL/min (MS) 5.29 mL/min (FID)
MS	
Ion Source Temperature	: 230℃
Interface Temperature	: 300℃
Measurement Mode	: Scan
Scan Range (*m/z*)	: 45-500
Ionization Mode	: EI
Event Time	: 0.3 s
FID	
Detector Temperature	: 300℃
Makeup Gas	: N_2 24 mL/min
Detector Gas	: H_2 32 mL/min、Air 200 mL/min

■ Sample Preparation

The following two types of samples were prepared:

(1) Essential Oil samples

Five types of Hinoki oils were prepared as follows:

① Branch and Leaf oil (Branch and Leaf)

This essential oil is extracted through steam distillation using thinned wood from "Shimadzu Corporation Forest" as the raw material.

② Shimadzu oil (Shimadzu)

Based on ①, this aroma oil is a blend of 24 types including Kyoto Hinoki, Ho wood, Abies sachalinensis, Rosemary, Orange, Grapefruit, and Anise.

③ Commercial Hinoki oil from Kiso (Kiso)

④ Commercial Hinoki oil from Yoshino (Yoshino)

⑤ Commercial Hinoki oil from Shimanto (Shimanto)

Essential oil samples were prepared by diluting each of the five Hinoki oils with hexane to a 1% volume ratio.

(2) Standard solution for quantification

δ-Cadinene was diluted with hexane to a 1% volume ratio. This was further diluted with hexane to 0.5-100% (volume ratio) to prepare the standard solution for quantification.

■ Multivariate Analysis

A total of 60 compounds, including terpenes, were identified from the five types of Hinoki oils. Using this result, a Principal Component Analysis (PCA) was conducted with eMSTAT Solution. The score plot is shown in Fig. 2. It was revealed that the five types of Hinoki oils are divided into four main groups. Among them, the Hinoki oils from Shimanto, Yoshino, and Kiso are positioned closely together, indicating relatively similar component characteristics. Next, we examined the characteristic components of each Hinoki oil using loading plot (Fig. 3). For the Hinoki oils from Shimadzu and Kiso, which were significantly apart on the score plot, it was confirmed that (-)-Borneol and δ-Cadinene are relatively abundant in each. The MS chromatograms of these two compounds are shown in Fig. 4.

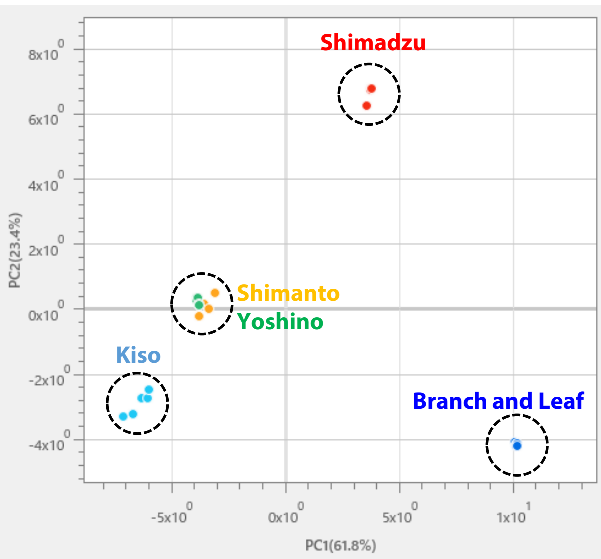


Fig. 2 Score Plot

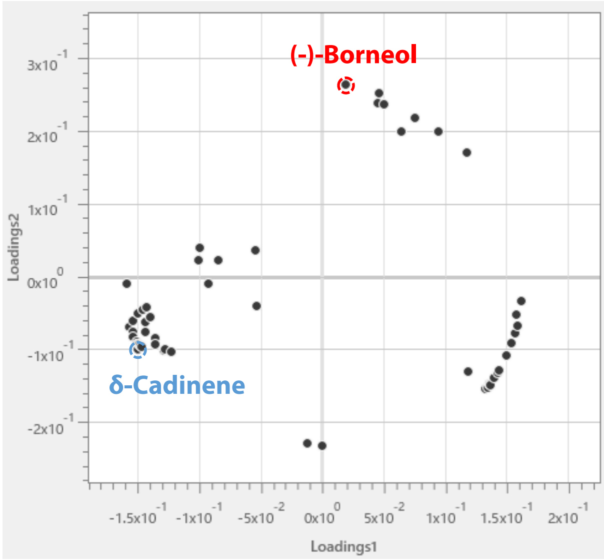


Fig. 3 Loading Plot

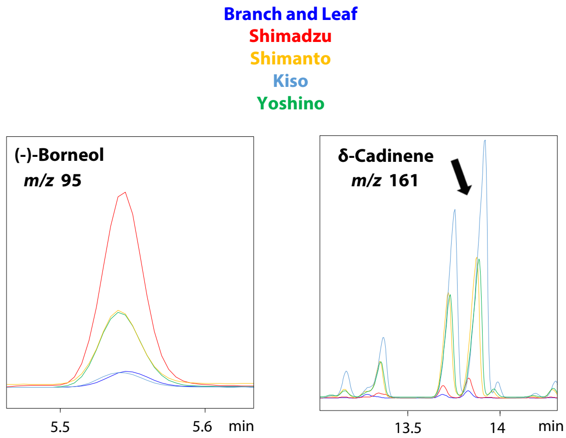
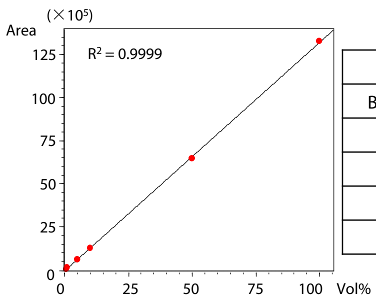


Fig. 4 Chromatograms of (-)-Borneol and δ-Cadinene

■ Quantification of δ-Cadinene Using FID

Using multivariate analysis based on the MS results, it was found that δ-Cadinene is relatively abundant in the Hinoki oil from Kiso and contributes significantly to its component characteristics. Therefore, accurate quantification was carried out using FID, which has a wide linear range for calibration. The calibration curve and Vol% values are shown in Fig. 5.



	Vol%
Branch and Leaf	1.5
Shimadzu	4.5
Shimanto	20.9
Kiso	46.1
Yoshino	28.3

Fig. 5 Calibration curve and quantification values of δ-Cadinene

■ Simultaneous MS and FID Measurement

In simultaneous MS and FID measurements, the retention times of each component almost match. Therefore, when unknown peaks are observed in FID, it is possible to qualitatively identify them using the MS spectrum results. In the chromatogram of Hinoki oil shown in Fig. 6, the peak positions of (-)-Borneol and δ-Cadinene in MS (TIC) and FID are almost identical. Furthermore, when looking at the library search results, (-)-Borneol and δ-Cadinene show high values of 97 and 93, respectively, indicating that qualitative identification with high accuracy can be achieved from FID peaks using MS spectra (Fig. 7).

■ Conclusion

Five different types of Hinoki oils were analyzed using a detector splitting system with MS and FID. When conducting multivariate analysis with the results from MS using eMSTAT Solution, it was possible to visually capture the differences in component characteristics. It was revealed that (-)-Borneol is a characteristic component of Shimadzu oil. Additionally, accurate quantification of δ-Cadinene, which varies significantly in concentration among samples, was achieved using FID.

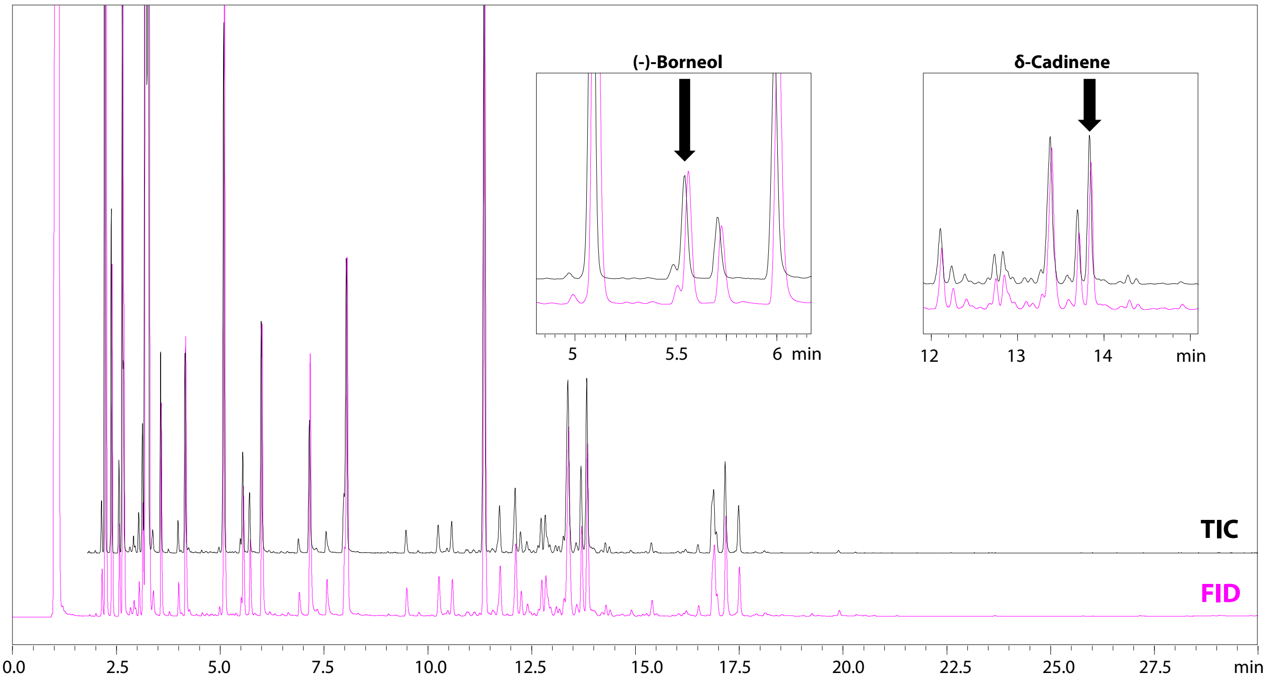


Fig. 6 TIC and FID Chromatograms of Shimadzu Oil

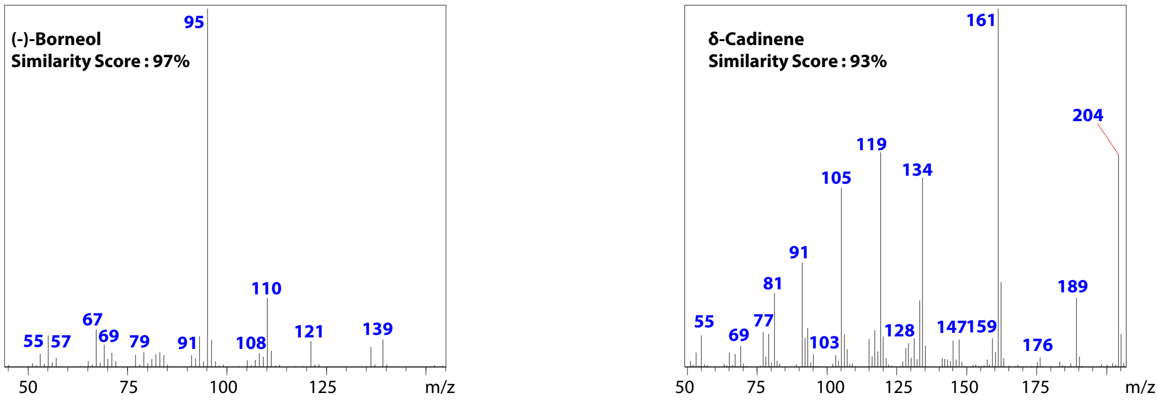


Fig. 7 MS spectra of (-)-Borneol and δ-Cadinene

01-00819-EN First Edition: Jan. 2025

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