



AccuTOF-GCv Series

Aroma Oil Analysis using GCxGC-HRTOFMS Performance Test for AccuTOF GCv 4G

Introduction

The AccuTOF GCv 4G is JEOL's third generation high resolution GC-TOFMS. New, enhanced features of the system include:

- 1) Recording speed: up to 50 spectra/sec
- 2) Mass resolution: 8,000 or more (m/z 614, FWHM)
- 3) Mass accuracy: 1.5 mmu or 4 ppm
- 4) Mass range: m/z 4 to 5,000

Comprehensive 2D GC (GCxGC) is a chromatographic separations technique that uses 2 columns with different polarities arranged in a series. Featuring higher resolution than conventional capillary GC analysis, it is a powerful tool for the measurement of multiple components in a complex mixture. However, because there is a cryo-trap before the 2nd column, the resulting peaks in the chromatograms are extremely narrow. As a result, the system requires a detector capable of high speed data recording. The TOFMS is an ideal detector for the 2D GC system.

In this work, we analyzed aroma oil using a GCxGC-HRTOFMS system, in which the AccuTOF GCv 4G was used with a Zoex GCxGC system to examine the spectrum recording speed and mass accuracy.

Instrument	JMS-T100GCV 4G (JEOL Ltd.) KT2004 (Zoex Corporation)
Injection mode	Split 200 : 1
Injection temp.	270 °C
Oven temp.	50 °C(3min) → 5 °C/min → 270 °C(8min hold)
Injection volume	0.2 µL
Column set	1st: BPX-5 (30 m x 0.25 mm, 0.25 µm) 2nd: BPX-50 (2 m x 0.1 mm, 0.1 µm)
Modulation period	6 sec
Ionization mode	EI+: 70 eV, 300 µA
Ion source temp.	270 °C
m/z range	m/z 35 - 500
Data acquisition	0.02 sec (50 Hz)

Table 1. Measurement condition

Experimental

For the sample, a commercial product of tea tree oil, a type of aroma oil, was used without treatment. Table 1 shows the measurement conditions used for the analysis. GC Image (Zoex) was used for processing of the GCxGC data.

Results and Discussion

Figure 1 shows a 2D map created from the aroma oil TIC. The compounds identified from each EI mass spectrum

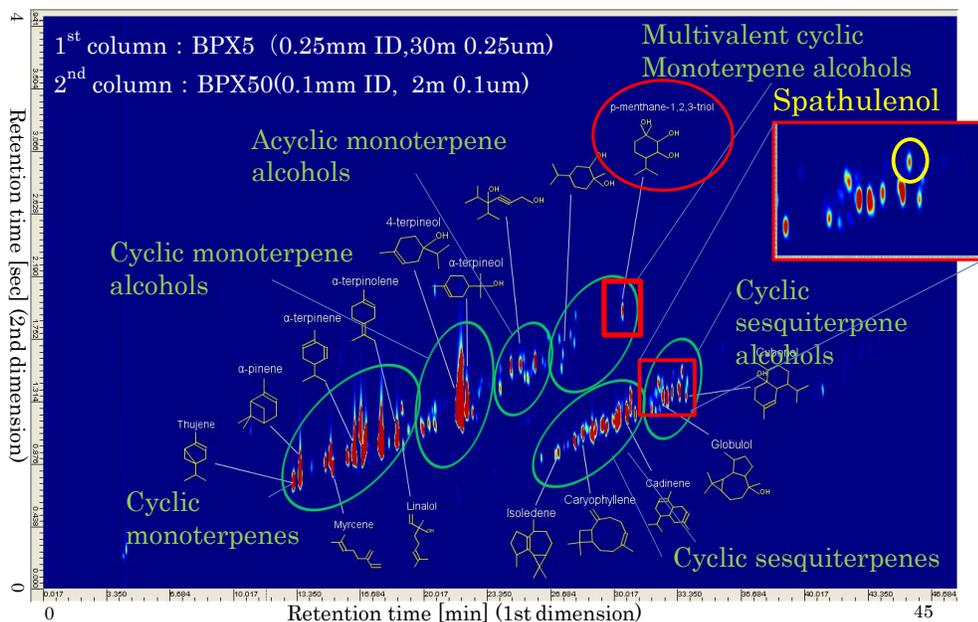


Figure 1. 2D map of tea tree oil sample analyzed by GCxGC-HRTOFMS

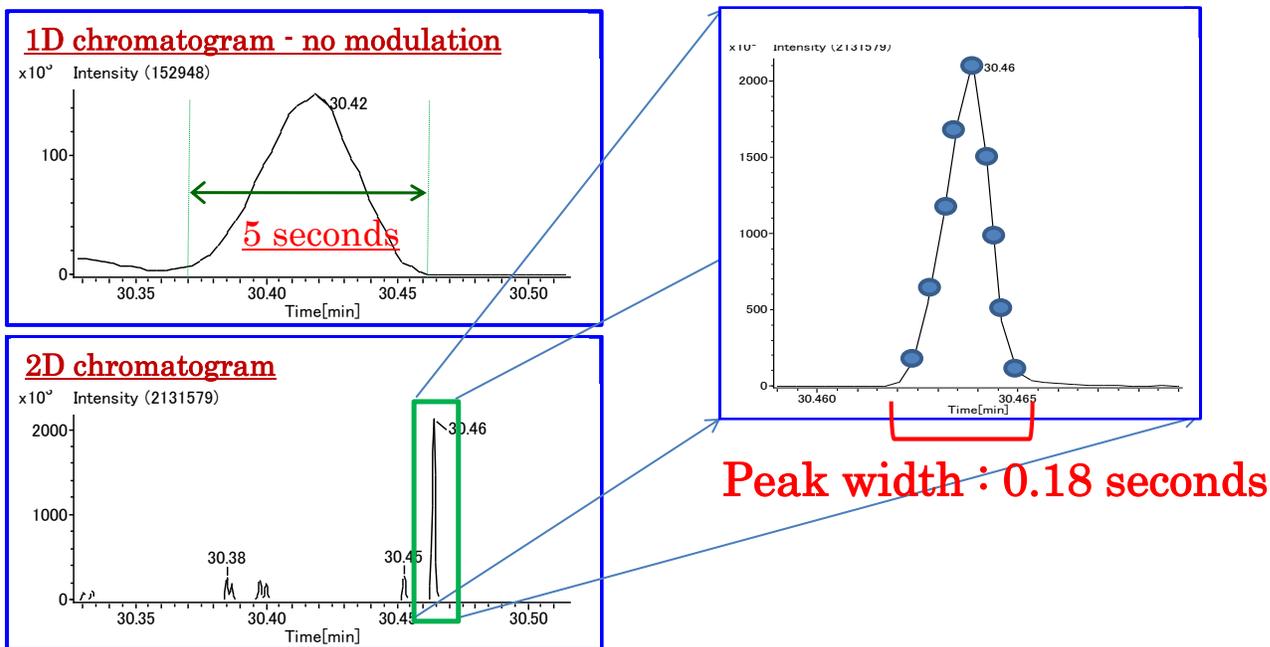
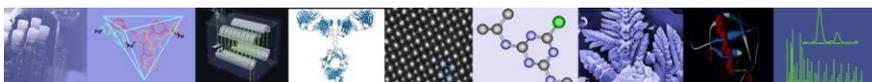


Figure 2. Magnified peak of p-menthane-1,2,3-triol

and their corresponding structures are marked on the 2D map.

Because the column set used for the analysis had a nonpolar 1st column and a polar 2nd column, the components were eluted with reference to their boiling point in the horizontal axis and their polarity in the vertical axis. Figure 1 shows cyclic and acyclic monoterpenes and monoterpene alcohols in groups from 12 to 30 minutes in the horizontal axis. The cyclic sesquiterpenes and cyclic sesquiterpene alcohols were observed between 25 and 35 minutes in the 1st dimension.

Recording speed

The recording speed was examined using the peak width in the chromatogram of p-menthane-1,2,3-triol that was eluted at approximately 30 minutes in Figure 1. Figure 2 shows the 1D and 2D chromatograms of p-menthane-1,2,3-triol at the elution time with and without modulation on the same column set. The figure also shows a magnified chromatogram of the p-menthane-1,2,3-triol peak in the 2D chromatogram.

In the 1D chromatogram without modulation, the peak with p-menthane-1,2,3-triol as its major component had a

peak width of approximately 5 sec. Meanwhile, in the 2D chromatogram, the p-menthane-1,2,3-triol had an extremely narrow peak width of approximately 0.18 sec. This demonstrates that at the recording speed at 50 Hz, the system was able to acquire sufficient data points on the chromatogram, 9 points as shown in the magnified view in Figure 2, compared to the recording speed at 2.5 to 10 Hz that is typically used in 1D GC-MS data acquisition.

Exact mass accuracy

Next, the mass accuracy was examined using the mass spectrum of spathulenol ($C_{15}H_{24}O$, exact mass: 220.18271), a cyclic sesquiterpene that eluted at approximately 33 min in Figure 1. Figure 3 shows the measured mass spectrum for this compound. Table 2 shows the error (mDa) between the measured exact mass and the expected exact mass for the molecular ion when the same sample was analyzed 5 different times. The error ranged from 0.46 to 0.78 mDa, showing extremely high levels of accuracy and stability. These results demonstrate that the system is capable of estimating the composition with high precision during the high speed data acquisition that is required for GCxGC analyses.

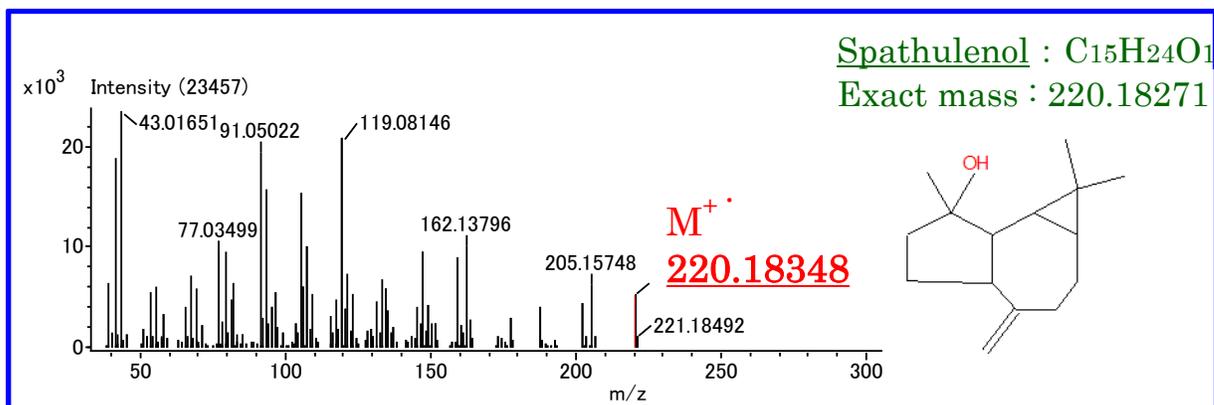
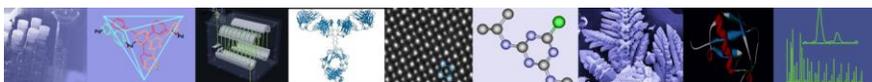


Figure 3. Mass spectrum of spathulenol

No.	Accurate mass (Da)	Error (mDa)
1	220.18348	0.77
2	220.18337	0.65
3	220.18326	0.55
4	220.18349	0.78
5	220.18318	0.46

Table 2. Errors between exact mass and accurate mass of spathulenol