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Application News

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Gas Chromatography Mass Spectrometry

Qualitative Analysis of Aroma Components in Beer Using Peak Deconvolution

The gas chromatograph mass spectrometer (GC-MS), which has excellent qualitative analysis capabilities, is used in analyses of the aroma components in foods and beverages. However, when the sample contains heavy matrix or is highly concentrated by pretreatment, many peaks are detected, and the target components may be hidden beneath other peaks. In some cases, it may be difficult to discover the target components from the total ion chromatogram (TIC).

The Automated Mass Spectral Deconvolution and Identification System (AMDIS) program supplied by NIST carries out peak detection by extracting a single spectrum from overlapping mass spectra by deconvoluting the GCMS data, and also searches all detected peaks from the mass spectrum library and identifies the detected compounds. Deconvolution makes it possible to detect and identify peaks, even when the target peak is overlapped by an adjoining peak.

This article reports the results of identification of the aroma components in beer by using peak deconvolution.

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Samples and Analysis Conditions

A commercial Pilsner-type beer was used as the sample. 10 mL of the beer and 3 g of NaCl were introduced into a 20 mL vial, which was then set in an AOC™-6000 multifunctional autosampler. SPME Arrow, which enables high enrichment of compounds, was used in sample introduction. Fig. 1 shows the appearance of the GCMS-QP™2020 NX and AOC-6000 multifunctional autosampler used in the analysis. Table 1 shows the instrument system and analysis conditions.

Fig. 1 GCMS-QP™2020 NX + AOC™-6000

Table 1 Analysis Conditions

Autosampler	: AOC-6000
GC-MS	: GCMS-QP 2020 NX
Column	: InertCap Pure Wax (Length 30 m, 0.25 mm I.D., df=0.25 µm) (GL Sciences)

SPME Arrow conditions

SPME Arrow	: DVB/Carbon WR/PDMS (O.D. 1.1 mm, film thickness 120 µm, length 20 mm)
Conditioning Temp.	: 250 °C
Pre Conditioning Time	: 5 min
Incubation Temp.	: 80 °C
Incubation Time	: 10 min
Agitator Speed	: 250 rpm
Sample Extract Time	: 30 min
Stirrer Speed	: 500 rpm
Sample Desorb Time	: 2 min (250 °C: GC Injection Temperature)

GC conditions

Injection temperature	: 250 °C
Injection mode	: Splitless (1 : 5)
Purge flow rate	: 3.0 mL/min
Control mode	: Constant pressure (83.5 kPa)
Column oven temperature	: 50 °C (5 min) → 10 °C/min → 250 °C (10 min)

MS conditions

Interface temperature	: 250 °C
Ion source temperature	: 200 °C
Ionization method	: EI
Measurement mode	: Scan
Event time	: 0.1 s

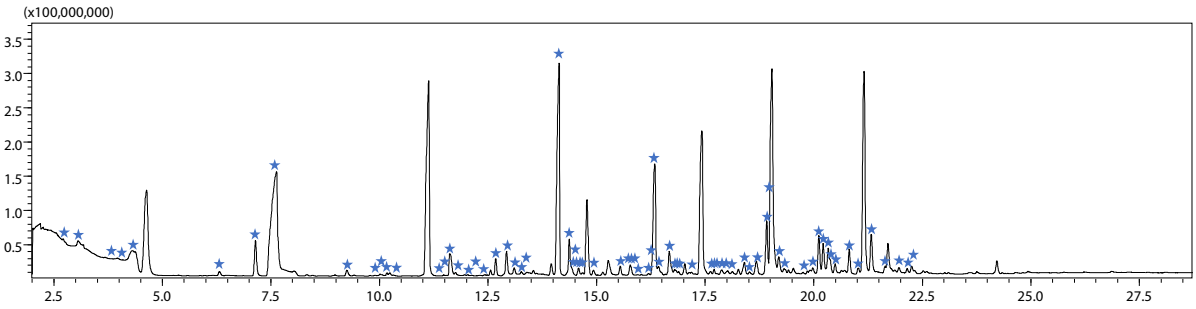

Fig. 2 Total Ion Chromatogram (TIC) of Beer (Stars : Identified Peaks by Target Analysis of Aroma Components)

Data Analysis by Peak Deconvolution

By using the AMDIS linked add-in function of GCMSsolution™, peak detection and identification by deconvolution with AMDIS can be conducted seamless from GCMSsolution. The retention time information of the peaks that are detected and identified and the related compound information are also reflected in GCMSsolution, enabling more detailed analysis.

The compounds associated with the peaks deconvoluted by AMDIS are identified by using a designated mass spectrum library (target analysis). In addition, the qualitative analysis of the detected peaks can also be conducted from the NIST library or other libraries by using the automatic qualitative analysis function of GCMSsolution (nontarget analysis).

Fig. 2 shows the total ion chromatogram (TIC) in this analysis. The aroma compound library (NISTFF) in AMDIS was used in the qualitative analysis of the deconvoluted peaks. In the analysis by AMDIS, 1,384 peaks were detected, and the compounds of 78 of those peaks were identified.

Target Analysis of Aroma Compounds

As mentioned above, 78 compounds were identified in the target analysis using the aroma compound library. These peaks included some compounds that were difficult to detect in the TIC, but could be identified by peak deconvolution. As one example, Fig. 3 shows the chromatogram of geraniol. In the TIC in this analysis, geraniol was hidden beneath the neighboring peak of caproic acid, and thus was impossible to identify in the TIC. It may be noted that geraniol is one type of monoterpene alcohol, and is known to be an aroma compound derived from hops in beer.

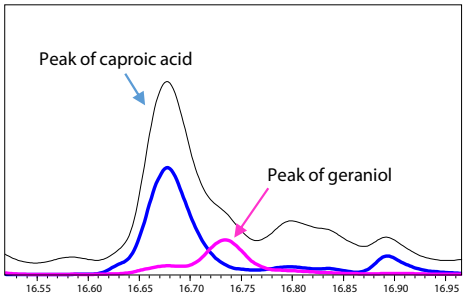

Fig. 3 Mass Chromatogram and TIC of Geraniol

Non-Target Analysis

A non-target analysis is carried out when comprehensive identification of compounds other the aroma compounds is desired. Automatic qualitative analysis from the retention time information of the peaks detected by deconvolution by AMDIS can be conducted with the NIST library by using the automatic qualitative analysis function of GCMSsolution (Fig. 4). In this analysis, a highly accurate qualitative analysis of the peaks that were hidden in the matrix is possible by appropriately setting the reverse search function.

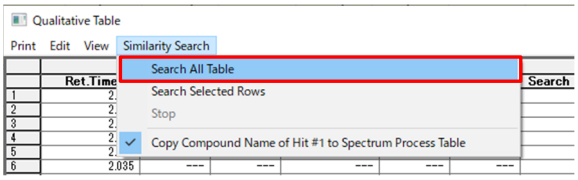

Fig. 4 Automatic Qualitative Analysis by GCMSsolution

As an example of the results of a non-target analysis, the peak with the retention time of 14.44 min shown in Fig. 5 was identified as ethyl trans-4-decenoate.

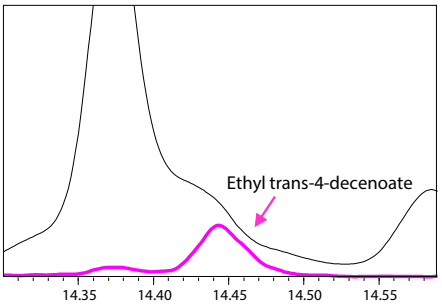

Fig. 5 Mass Chromatogram and TIC of Ethyl Trans-4-Decenoate

Conclusion

By using peak deconvolution, single peaks could be extracted from overlapping peaks and the compounds could be identified. Use of the AMDIS add-in function of GCMSsolution supports analysis by peak deconvolution. It is also possible to search the contribution of the identified compounds to smell by using multivariate analysis.

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