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GC-MS GCMS-QP™2020 NX/HS-20 NX

Application News

Analysis of Acetaldehyde and Limonene in Recycled PET Using an HS-GCMS System (Carrier Gas: H_2)

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User Benefits

- The gas released from the polymer can be easily measured without dissolving the polymer in a solvent, by using a headspace sampler (HS).
- Using an HS-GC/MS system enables qualitative and quantitative analysis of target components in samples with a large number of contaminants that make it difficult to differentiate the targets from other components.
- Analysis costs can be reduced by using hydrogen as a carrier gas, which is more affordable and readily available than helium.

■ Introduction

Plastic is lightweight, strong, waterproof, and very convenient, making it an essential part of our modern lives. On the other hand, because it is a major contributor to environmental issues, such as ocean pollution and global warming, measures to reduce plastic usage are being undertaken in countries around the world.[1)]

In Japan, the Plastic Resource Circulation Promotion Act[2)] came into effect in April 2022. The act does not regulate plastic but rather promotes the establishment of a sustainable circular economy by encouraging collaboration among businesses, local governments, and consumers throughout the overall process of designing, manufacturing, selling, collecting, and recycling plastic products.

One of these initiatives is the recycling of PET bottles for beverage use. When recycling beverage bottles, residual odors can be an issue, with substances like acetaldehyde from water bottles and limonene from citrus beverages known to persist in the containers. To address these residual substances, recycling companies are implementing unique strategies, one of which involves using gas chromatography-mass spectrometry (GC-MS) to quantify the residual materials.

In this article, we present an example of qualitative and quantitative analysis of aldehydes and limonene in PET bottles using the GCMS-QP2020 NX/HS-20 NX, with hydrogen (H_2) as the carrier gas.

■ Device Configuration and Analysis Conditions

Fig. 1 shows the GCMS-QP2020 NX/HS-20 NX system used in this study, while Table 1 indicates the device configuration and analysis conditions.

Fig. 1 GCMS-QP™ 2020NX + HS-20 NS

■ Sample Preparation

The samples consisted of three types of commercially available beverage PET bottles (for grapefruit juice, lemon tea, and water), which were cut up with scissors. Each sample was then sealed in an HS vial, in the amounts specified in Table 2, and analyzed under the conditions indicated in Table 1.

Table 1 Device Configuration and Analysis Conditions

GC-MS Analysis Conditions	
Model	: GCMS-QP2020 NX
Column	: SH-PolarWax (P/N 227-36248-01) (0.25 mm I.D. × 30 m, d.f., 0.5 µm)
Column Temp.	: 40 to 250 °C at 10 °C/min (Total 21 min)
Injection Mode	: Split (1:2)
Carrier Gas Controller	: Constant Linear Velocity (H_2)
Linear Velocity	: 55 cm/sec
Ion Source Temp.	: 200 °C
Interface Temp.	: 250 °C
Measurement Mode	: Scan/SIM (Simultaneous Measurements)
SCAN Range	: *m/z* 10 to 250
SIM	: *m/z* 43, 29, 42 (Acetaldehyde) *m/z* 136, 68, 93 (D-Limonene)
Event Time	: 0.3 sec
HS Analysis Conditions (Trap: Tenax TA)	
Vial Volume	: 20 mL
Injection Volume	: 1 mL
Oven Temperature	: 80 °C
Sample Line Temp.	: 150 °C
Transfer Line Temp.	: 150 °C
Trap Cooling Temp.	: -20 °C
Trap Heating Temp.	: 220 °C
Trap Waiting Temp.	: 25 °C
Multi-Injection Times	: 5
Vial Stirring	: 5
Vial Pressure	: 80 kPa (N_2)
Dry Purge Pressure	: 20 kPa
Vial Heating Time	: 30 min.
Vial Pressurization Time	: 0.5 min
Pressure Equilib. Time	: 0.1 min
Loading Time	: 0.5 min
Load Equilib. Time	: 0 .1 min
Dry Purge Time	: 1 min
Injection Time	: 5 min
Needle Flush Time	: 5 min

Table 2 Quantity of Various PET Bottle Samples Sealed in Vial

Sample	State	Qty
G1	Grapefruit	1g
G2	Grapefruit	1g
G3	Grapefruit	2g
L1	Lemon Tea	1g
L2	Lemon Tea	1g
L3	Lemon Tea	2g
W1	Water	1g
W2	Water	1g
W3	Water	2g

■ TIC Chromatogram

As a representative example of the samples, a TIC chromatogram of sample G1 is shown in Fig. 2.

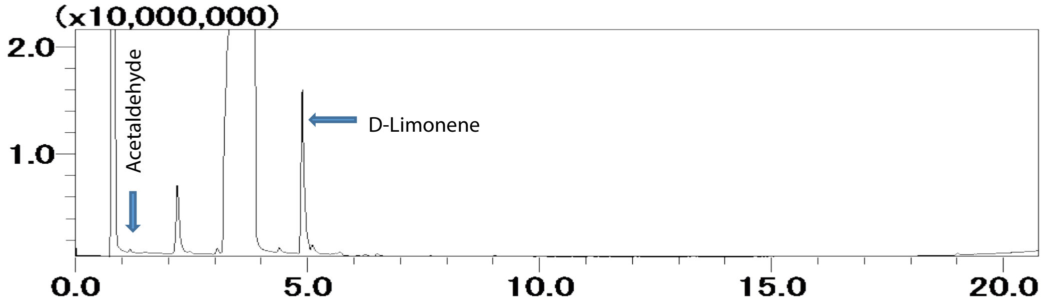

Fig.2 TIC Chromatogram of Sample G1

■ Calibration Curves

2 µL of each calibration curve preparation reagent was sealed in an HS vial and diluted with an acetone solution to prepare 2, 20, 100, and 200 µg quantities of acetaldehyde and 10, 50, 100, 200, 400, and 2000 ng quantities of limonene, assuming that all quantities would be evaporated. The calibration curves for acetaldehyde and limonene are shown in Figs. 3 and 4.

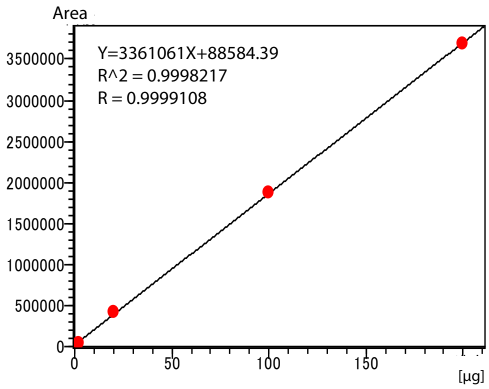

Fig. 3 Acetaldehyde Calibration Curve

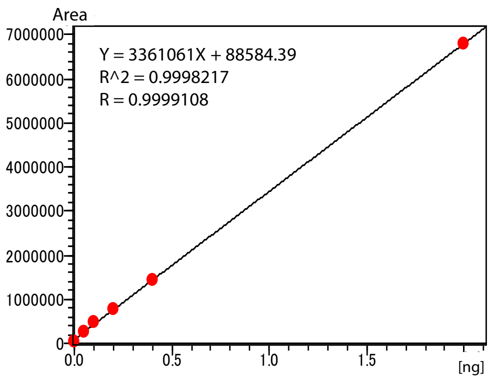

Fig. 4 Limonene Calibration Curve

■ Analysis Results

The results of acetaldehyde and limonene quantification per gram from the amounts of each sample sealed in the vial are shown in Table 3. Although there is some variation in the quantification values due to the differences in the plastic pieces placed in the HS vials, limonene was not detected in the PET bottles that contained water (W1–W3), while acetaldehyde was found to be the most abundant among the three types.

Table 3 Quantification Results of Acetaldehyde and Limonene per 1 g of Each Sample

Sample	Acetaldehyde	Limonene
G1	14.13 µg	2.98 ng
G2	12.61 µg	2.84 ng
G3	16.03 µg	3.61 ng
L1	10.27 µg	6.63 ng
L2	12.15 µg	7.38 ng
L3	11.01 µg	5.68 ng
W1	34.14 µg	N.D
W2	17.63 µg	N.D.
W3	22.15 µg	N.D.

■ Conclusion

By using hydrogen (H2) gas as the carrier gas, we were able to avoid the use of the hard-to-obtain helium gas. Additionally, by employing an HS-GC-MS system, we could qualitatively and quantitatively analyze acetaldehyde and limonene from PET without the need for cumbersome pretreatment.

<References>

1) The Nippon Foundation (2022-09-30)

2) Ministry of the Environment Government of Japan

<Related Applications>

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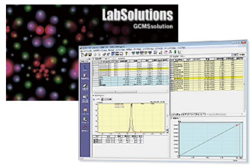

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