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Application News

Gas Chromatograph HS-20 NX/Brevis™ GC-2050

Analysis of Diacetyl and 2,3-Pentanedione in Beer Using Brevis™ GC-2050 (Carrier Gas: N_2)

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User Benefits

- Quantitation of diacetyl and 2,3-pentanedione at ppb levels using an ECD detector without any pre-processing can be performed.
- The only gas required for the analysis is nitrogen (N_2).
- By using the compact design of the Brevis GC-2050, it is possible to achieve high-performance analysis in a space-saving manner.

■ Introduction

There are off-flavors in beer, namely diacetyl and 2,3-pentanedione (collectively known as vicinal diketones or VDKs). VDKs are primarily generated during the early stages of fermentation, and when present above a certain threshold, it imparts a butterscotch-like aroma to the beer. By allowing the beer to undergo a maturation process after fermentation, the VDK levels can be reduced to a certain threshold, effectively removing the off-flavors associated with this component.[1)] This process is known as diacetyl rest.

In this study, the changes in VDK levels before and after dry hopping (DH), a process commonly used in craft beer production where hops are added after fermentation, were investigated using a nitrogen carrier gas. The evolution of VDK levels during the maturation process was also investigated. Furthermore, an analysis of VDKs in commercially available beers was also conducted, and the results are presented.

■ Sample Preparation and Quantification Method

To create the calibration curve, standard solutions of diacetyl and 2,3-pentanedione were prepared by diluting them in water to achieve concentrations of 10, 25, 50, and 100 ppb (v/v) each. A water solution of 2,3-hexanedione was used as an internal standard solution. For each standard sample, 5 mL of the standard solution and the internal standard solution were added to a headspace vial, ensuring that the concentration of the internal standard solution in each standard sample was 50 ppb (v/v). The vials were then sealed, and headspace analysis was performed to create the calibration curve. For the analysis of the actual beer samples, 5 g of the beer without degassing treatment was mixed with the same amount of the internal standard solution as mentioned above. The mixture was then sealed and heated at 60 °C for 90 minutes (*1). After cooling, the sample was placed in the sample tray of an HS-20 NX instrument for headspace analysis. The quantification was performed using the calibration curve that was created earlier.

*1 By performing the heating treatment as a pre-processing step, it is possible to measure the VDK precursors as the total VDK content.

■ Instrument Configuration and Analysis Conditions

Fig. 1 shows Exterior View of Brevis GC-2050+HS-20 NX, Table1 shows analysis conditions. The Brevis GC-2050 with the HS sampler, has a compact system width of approximately 35 in (35.9″). It offers excellent analytical performance without compromising on space efficiency.

Fig. 1 Brevis™GC-2050 + HS-20 NX

Table 1 Instrument Configuration and Analysis Conditions

HS-20 NX	
Mode:	Loop
Oven Temperature:	40 °C
Sample Line Temperature:	90 °C
Transfer Line Temperature:	95 °C
Vial Pressure:	150 kPa
Vial Heat-Retention Time:	40 min
Vial Pressurization Time:	1 min
Vial Pressurization Equilibrating Time:	0.1 min
Loading Time:	0.5 min
Loading Pressurization Time:	0.1 min
Injection Time:	0.5 min
Needle Flush Time:	5 min
Brevis GC-2050/ECD-2050	
Injection Mode:	Split
Split Ratio:	1:20
Carrier Gas:	N_2
Carrier Gas Control:	Constant Linear Velocity (35 cm/sec)
Column:	SH-624 Cap. (P/N 221-75864-60) (60 m × 0.32 mm I.D., 1.80 µm)
Column Temperature:	50 °C - 5 °C/min – 120 °C (6 min)
Detector Temperature:	130 °C
ECD Current:	2.5 nA
Detector Gas:	N_2 30 mL/min

■ Chromatogram of Standard Sample

Fig. 2 shows the chromatograms of the standard samples.

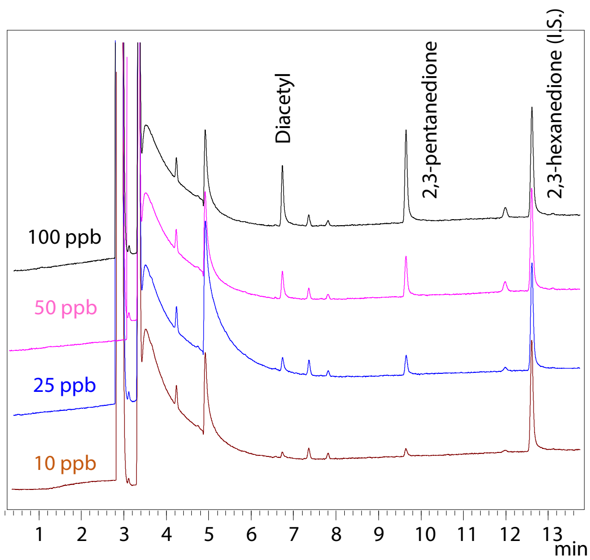

Fig. 2 Chromatograms of Standard Samples

■ Calibration Curves of Standard Samples

Fig. 3 shows the calibration curves of the standard samples.

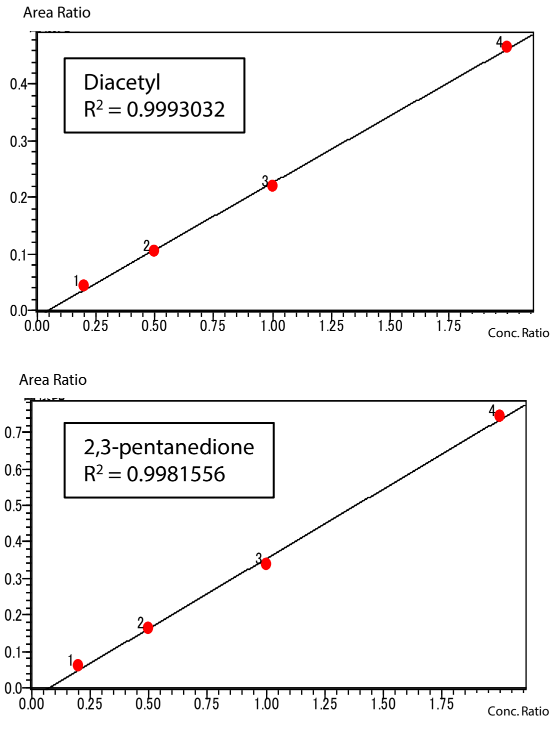

Fig. 3 Calibration Curves of Standard Samples

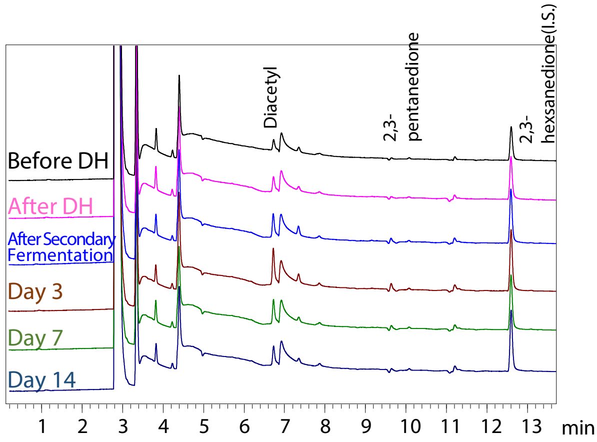

Fig. 4 Chromatograms before and after Dry Hopping (DH) and for Each Secondary Fermentation Period

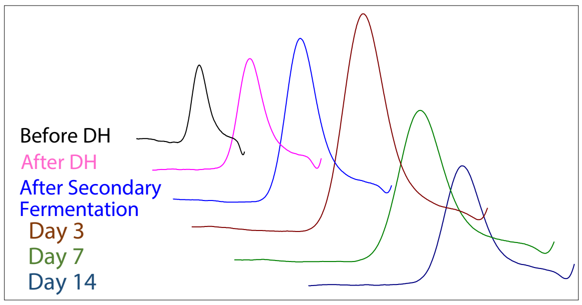

Fig. 5 Temporal Changes in Diacetyl Levels

■ Quantitative Results for VDK in Three Commercially Available Beer Samples

Table 2 shows the quantitative results for VDKs in the three brands of commercially available beers.

Table 2 Quantitative Results for VDK in Three Brands of Beer

Beer	Diacetyl	2,3-Pentanedione
A	46.1 ppb	10.8 ppb
B	233.0 ppb	19.9 ppb
C	215.4 ppb	19.2 ppb

■ Change in VDK Levels after Primary Fermentation

Fig. 4 shows the chromatograms before and after dry hopping (DH) and for each secondary fermentation period. Fig. 5 shows the chromatograms of the temporal changes in diacetyl. Table 3 shows quantitation results of VDKs in beer.

In this sample, it was found that the VDK peaks increased from immediately after dry hopping to three days of maturation, and then decreased thereafter. Based on these findings, it can be suggested that the increase in VDK levels observed from dry hopping is due to re-fermentation (hop creep). Additionally, it was observed that as the duration of secondary fermentation increased, the yeast consumed the diacetyl, indicating the occurrence of diacetyl rest.

Table 3 Quantitative Results for VDKs in Beer

	Diacetyl	2,3-pentanedione
Before DH	67.2 ppb	11.3 ppb
After DH	76.5 ppb	12.0 ppb
After Secondary Fermentation	92.3 ppb	16.8 ppb
Day 3	122.7 ppb	21.3 ppb
Day 7	88.1 ppb	16.3 ppb
Day 14	63.7 ppb	13.5 ppb

■ Conclusion

The VDK concentrations in the beer samples after dry hopping were analyzed using the Brevis GC-2050 (with an ECD detector) and HS-20 NX system. Nitrogen gas was used as the carrier gas for the analysis. It is possible to analyze the changes in VDK levels in beer samples, even at low concentrations, without the need for any special pre-treatment such as degassing or concentration.

<References>

Toru KISHIMOTO: The recent findings in the off-flavors of beer, J. Japan Association on Odor Environment Vol. 44 No. 1 2013
pdf (jst.go.jp)

<Related Applications>

1. Analysis of Diacetyl and 2,3-Pentanedine in Beer Using Nexis™ GC-2030, Application News No. G316B

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