Determination of diacetyl (butanedione) & pentanedione in beer by HS-GC

Application Note

Author

Yunfei Bai[1] Hao Wang[2]
Shun Na[2]
[1] College of Food Science and Nutritional Engineering, China Agriculture University
[2] Agilent Technologies

Introduction

Diacetyl (butanedione) and 2,3-pentanedione are two natural productions of beer fermentation process. The improper controlling of fermentation process and microbial contamination would result in the increasing of 2,3-pentanedione as well as annoying “rancid rice flavor”. It would likely lead to the regeneration or pouring of beer, which couldn’t meet the national standard. Measuring the content of these two compounds could help us control the complete and appropriate fermentation. Headspace Gas Chromatography (HS-GC) technology has been used in this experiment. At the same time, electron capture detector (μECD) was used to detect diacetyl & pentanedione. The result shows that the method is sensitive, reproducible, and could achieve good quantitative linear results. Besides, wide adaptability matrix determination accuracy can be well guaranteed.

Instrument

Agilent 7697A Headspace Autosampler with 111 position tray; Agilent 7890A Gas-chromatography with µECD detector.

Reagents

Standard Reagent: (butanedione, 2,3-pentanedione) were taken from Technology Center of Yanjing Beer Coorperation; 4% ethanol (ethanol (HPLC), milli Q ultrapure water system) for diluting standard solutions.

Parameter of Agilent HS-GC

Headspace vials & Sample volume per vial:
20 ml Headspace vials; 5 ml liquid/vial

Oven temperature:	50 °C
Equilibration time:	30 min
Loop temperature:	100 °C
Extraction mode:	Single Extraction
Transmission line:	Silcotek fused silica capillary, Inner diameter 530 µm
Transmission line temperature:	110 °C
Pressure:	15 psi, 1 min
Gas injection volume:	1 ml
Gas injection time:	0.1 min

Parameter of Agilent 7890A GC

GC Column:	J&W 125-5065 DB-5 60m*0.53 mm*5 µm
Inlet site:	Split/splitless 1:1 120 °C
Carrier gas:	High purity nitrogen (>99.999%)
Liner description:	18740-80200 (HS)
Column flow:	Constant flow 10 ml/min
Temperature program:	45 °C 2 min→150 °C (10 °C/min)
Detector site:	µECD 150 °C
Make-up gas:	High purity nitrogen 30 ml/min
Sampling rate:	50 Hz/0.004 min

Chromatograms of butanedione & 2,3-pentanedione in standard solution & beer

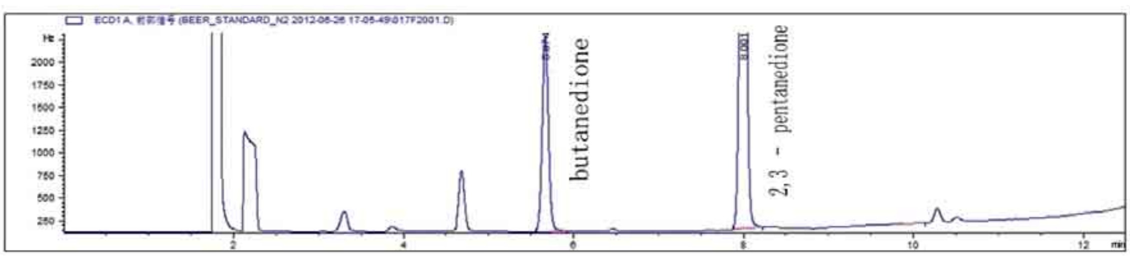

Figure 1. Chromatograms of butanedione & 2,3-pentanedione standard solution

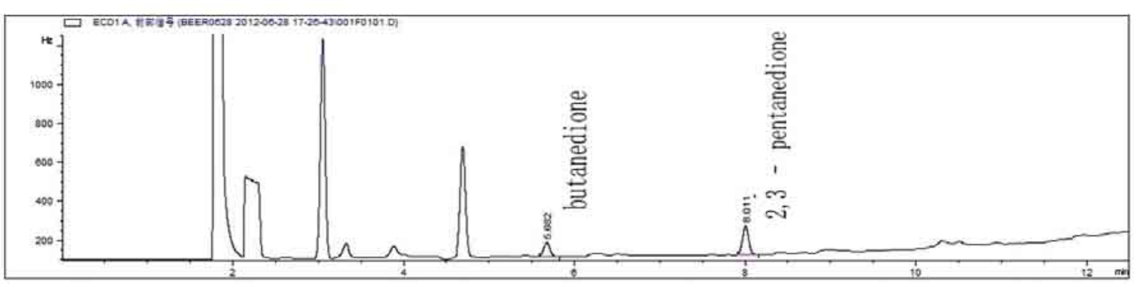

Figure 2. Chromatograms of butanedione & 2,3-pentanedione in beer

Precision measurement

Beer sample was used in analyzing the representative of precision (n=6), the repeated measurements chromatogram were as follows:

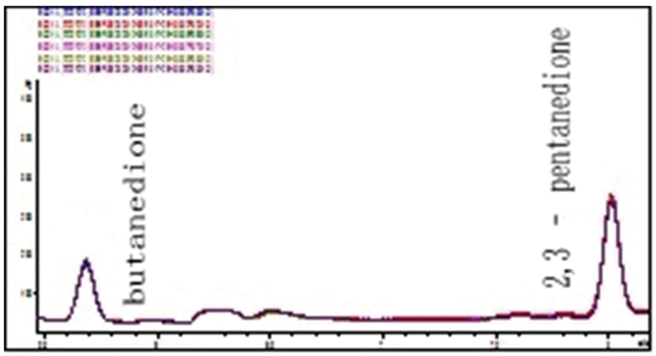

Figure 3. Repeating test chromatograms of butanedione & 2,3-pentanedione in beer

Tab 1. Repeatability of chromatograms peak area

	butanedione	2,3-pentanedione
1	595.4	879.8
2	578.6	858.2
3	583.3	869.7
4	569.9	869.5
5	556.9	850.3
6	563.6	857.7
RSD (%)	2.4	1.2

Tab 2. Repeatability of retention time

	butanedione (min)	2,3-pentanedione (min)
1	5.682	8.011
2	5.683	8.011
3	5.683	8.011
4	5.683	8.011
5	5.684	8.011
6	5.684	8.012
RSD (%)	0.01	<0.01

Linear

Standard mixture of butanedione and pentanedione was diluted to 0,25, 50,100, 200, 500 µg/L standard solutions. The external standard method has been taken based on the concentration of the standard series and the corresponding peak area. The standard curves of butanedione and pentanedione were drafted and achieved good linearity that could be able to quantification accurately, as shown below:

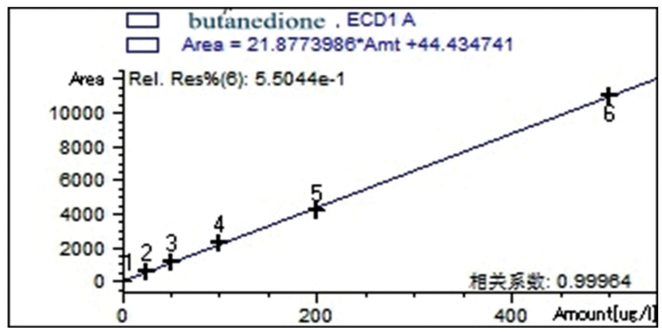

Figure 4. The correction curve of butanedione measurement.

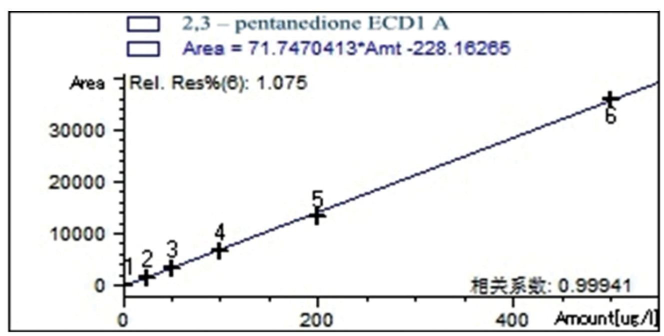

Figure 5. The correction curve of 2,3-pentanedione measurement

Diacetyl & 2,3-pentanedione contents in selected beer samples

Some commercially available beer samples were selected for butanedione and 2.3-pentanedione test. Each measurement were taken three times and the results are as follows:

Tab 3. Butanedione & 2,3-pentanedione content in commercially available beer samples

	butanedione (µg/L)	2.3-pentanedione (µg/L)
Beer 1	12.98	11.65
Beer 2	14.83	10.89
Beer 3	24.32	14.03
Beer 4	40.80	19.82
Beer 5	7.85	6.41
Beer 6	20.00	15.63
Beer 7	17.61	9.92

Diacetyl in different manufacturers and product types of beers were ranging from about 7 µg/L to 40 µg/L, while the 2,3-pentanedione content ranging from about 6 µg/L to 20 µg/L, which shows that diacetyl and 2,3-pentanedione contents could be closely related to the process conditions, product offerings and other factors. This method can be used for accurate quantification of diacetyl & 2,3-pentanedione in each specific type of beer.

Conclusion

The experiment method and sample test results proved Agilent 7697A Headspace Autosampler & 7890A Gas Chromatography system can simply, accurately, precisely completed the determination of diacetyl and 2,3-pentanedione in beer. The method achieves good linearity and is fully able to ensure the testing tasks for research institutions, as well as for product quality control in relevant factories.

Commercially available beer samples produced by different manufacturers, technologies and other conditions have different contents of diacetyl and 2,3-pentanedione. The present method can be used not only in accurate quantification of diacetyl and 2,3-pentanedione in each kind of beer sample, but also in research for what causes its difference content, how it changes in fermentation process and provide a basis on the product quality control.

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