

Application News

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Gas Chromatography

Aroma Component Analysis of Japanese Sake by GC-FID Using a Diacetyl Derivatizing Reagent

The food industry widely employs evaluation using analytical instruments for new product developments and quality assurance. In the brewing industry, aroma component analysis is highly important due to the strong effects on product quality. Regarding aroma components in brewed Japanese sake, the conventional analysis method uses a gas chromatograph combined with a headspace sampler. By combining this method with a high-sensitivity detector, detection of aroma components in low concentrations is possible. However, such a system is expensive and sample preparation calls for an experienced analyst.

In this article, we introduce an example analysis of aroma components in Japanese sake with a simple GC system configuration using a simplified pretreatment method which employs a derivatizing reagent manufactured by Shinwa Chemical Industries.

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Shinwa DS-DA (Diacetyl) Diacetyl Derivatization Kit Shinwa Chemical Industries

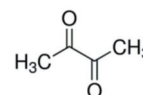
Shinwa DS-DA is a reagent kit for derivatizing diacetyl in non-alcoholic beverages and alcoholic beverages for measurement by gas chromatography.



Fig. 1 Shinwa DS-DA (Diacetyl)

Diacetyl in Japanese Sake

Diacetyl is a compound which has a characteristic aroma and greatly affects the quality of fermented beverage and food products. Although the aroma of diacetyl is indispensable for dairy products which are made by lactic acid fermentation such as cultured butter and some cheeses, it is considered undesirable for food and drinks which are made by alcoholic fermentation such as alcoholic beverages. In particular, sake breweries consider the aroma to be one of the most undesirable odors and therefore conduct quantitative evaluation at production.



Derivatization Reaction of Diacetyl Using Shinwa DS-DA

Diacetyl reacts with o-phenylenediamine and turns into 2,3-dimethyl quinoxaline (DMQX). Although FID sensitivity for diacetyl is low because diacetyl has two carbonyl groups, DMQX obtained by derivatization has a quinoxaline structure and therefore the FID sensitivity rises.

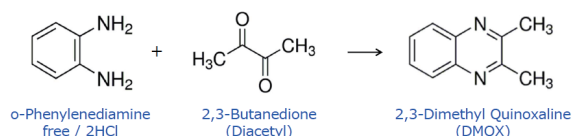


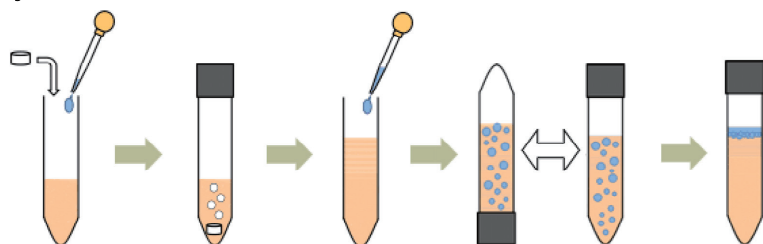
Fig. 2 Derivatization Reaction of Diacetyl Using Shinwa DS-DA

Procedure of Diacetyl Derivatization Using Shinwa DS-DA

Derivatization was performed using the Shinwa DS-DA (Diacetyl) reagent kit on a sample (Japanese sake). The diacetyl contained in the sample reacts and turns into DMQX. Methyl n-propyl quinoxaline (MPQX) was added as an internal standard substance.

Derivatizing Diacetyl Using Shinwa DS-DA

Sample	10 mL	Leave to stand for approx. 1 min until tablet dissolves	Reagent C	8 mL	Mix by inverting for 2 min	GC analysis of supernatant
Reagent A	100 μL		Reagent D	1 mL		
Reagent B	1 tablet					



- The tablet may take time to dissolve depending on the sample.
- The tablet may not completely dissolve.

Fig. 3 Procedure of Diacetyl Derivatization Using Shinwa DS-DA

Instrument Configuration and Analysis Conditions

Table 1 lists the configuration of the instrument used for analysis and the analysis conditions.

Table 1 Instrument Configuration and Analysis Conditions

Gas chromatograph	: GC-2010 Plus
Autosampler	: AOC-20i
Sample injection port	: SPL
Column	: ULBON HR-52 (25 m × 0.25 mm I.D. df = 0.25 mm)
Detector	: Flame ionization detector (FID)
Injection volume	: 2 µL
Carrier gas	: He, 30 cm/sec
Split ratio	: 5:1
Injection port temperature	: 200 °C
Detector temperature	: 280 °C
Column temperature	: 100 °C (2 min) - 10 °C/min - 270 °C (5 min)

Analysis of Diacetyl Derivatives Obtained Using Shinwa DS-DA

After derivatizing diacetyl, the peak from DMQX was detected in the vicinity of a retention time of 9.6 min (Fig. 4).

The dotted line in Fig. 5 indicates the calibration curve of a 15 % ethanol solution which was prepared in accordance with the composition of Japanese sake and then added diacetyl. The solid line is the calibration curve of a commercially available sake which was added diacetyl. From this we were able to determine that the diacetyl content in this commercially available sake is 125 ppb (µg/L).

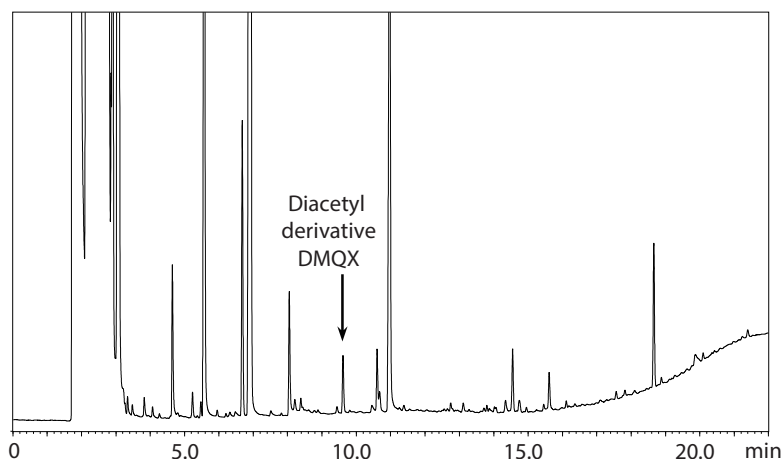


Fig. 4 Chromatogram of Japanese Sake Sample After Diacetyl Derivatization

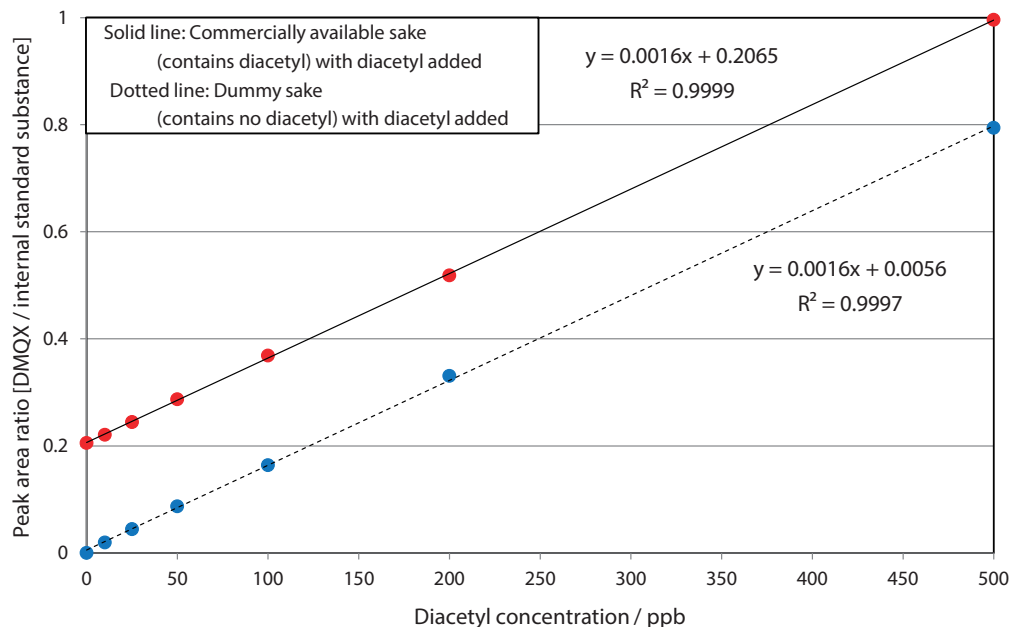


Fig. 5 Calibration Curve of Diacetyl Derivative Sample

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