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Application News

Nexis™ GC-2030 Gas Chromatograph

Analyzing Residual Solvents in Sucrose Fatty Acid Esters by HS-GC-FID Analysis (Japan's Specifications and Standards for Food Additives)

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User Benefits

- Five types of sucrose fatty acid ester impurities can be analyzed by HS-GC-FID analysis.
- Target substances can be quantitated easily using the standard addition method.
- Using a headspace (HS) sampler avoids the need for tedious pretreatment.

■ Introduction

Sucrose, the main compound in table sugar, consists of a glucose molecule with fructose attached. A sucrose fatty acid ester is formed by attaching a plant-based fatty acid to one of the 8 locations on sucrose that have an affinity to water.

Since sucrose loses its sweetness when bonded to a fatty acid, sucrose fatty acid esters are tasteless and odorless molecules. That makes sucrose fatty acid esters a non-ionic surfactant with a hydrophilic sucrose end and a lipophilic fatty acid end. Therefore, they are widely used in foods and pharmaceuticals as emulsifiers, dispersants, additives for adjusting viscosity, foaming agents, and antioxidants.

Test methods for analyzing residual solvents in sucrose fatty acid esters are specified in Japan's Specifications and Standards for Food Additives and the U.S. Food Chemicals Codex (FCC 11). (For more information about FCC 11, refer to Shimadzu Application News 01-00495.)

This article describes an example of separating, analyzing, and quantitating residual solvents in a commercial sucrose fatty acid ester product based on analysis conditions specified in Japan's Specifications and Standards for Food Additives.

■ Preparing Standard and Measurement Samples

Standard samples for calibration curve were prepared as follows.

First, 0.2 g each of methanol, 2-propanol, 2-butanone, ethyl acetate, and isobutanol were weighed and mixed. Then water was added to make exactly 50 mL of solution (Solution A).

Next, 5 mL and 10 mL quantities of Solution A were accurately measured and diluted with water to accurately make 20 mL of Solutions B and C, respectively. For this example, Solution B was also diluted 10-fold to make Solution D.

1 g quantities of an unknown sample (a commercial sucrose fatty acid ester product was used for this example) were weighed and placed in headspace vials. Then 5 µL each of water and the standard sample prepared above were accurately added to each vial and used as the sample solutions for analysis using the conditions indicated in the next section.

5 µL of either water or the standard sample was added to 1 g of the unknown sample to obtain the quantity (µg) of each component per 1 g of the unknown samples indicated in Table 1.

Table 1 Content of Each Component in 1 g of Sample

µg/g	0	0.5	5	10	20
Standard Sample	Water	D	B	C	A

■ Analysis Conditions

The analysis conditions are shown in Table 2. Japan's Specifications and Standards for Food Additives specifies using splitless conditions for analyzing sucrose fatty acid esters, but the flow was split for this example because it is the optimal best condition setting for the headspace sampler (HS-20 NX).

Table 2 Analysis Conditions

GC Analysis Conditions	
Model:	Nexis GC-2030
Detector:	FID-2030
Column:	SH-1 (0.53 mm I.D. × 30 m, d.f. = 1.5 µm) (P/N: 221-75732-30)
Inj. Mode:	Split 1:10
Carrier Gas:	Constant Linear Velocity Mode (N_2)
Linear Velocity:	30 cm/sec
Column Temp.:	40 °C (7 min)
FID Temp.:	200 °C
Makeup Gas (N_2):	24 mL/min
H_2 Flow:	32 mL/min
Air Flow:	200 mL/min
HS Analysis Conditions	
Model:	HS-20 NX (Loop)
Oven Temperature:	80 °C
Sampling Line Temp.:	85 °C
Transfer Line Temp.:	110 °C
Vial Pressure:	80.0 kPa (N_2)
Vial Holding Time:	40 min.
Vial Pressurization Time:	1.0 min.
Pressure Equili. Time:	0.1 min.
Loading Time:	0.5 min.
Injection Time:	0.5 min.
Needle Flush Time:	5 min.

■ Calibration Curves (Standard Addition Method) and Quantitation Results

Fig. 1 shows an example of overlaid chromatograms from various concentrations of a standard sample added to commercial sucrose fatty acid ester. The calibration curves for respective components are shown in Fig. 2. That resulted in calibration curves with an excellent coefficient of correlation R of 0.998 or greater for all components. The content of five types of compounds in the samples used for this example are indicated in Table 3. Japan's Specifications and Standards for Food Additives specifies that samples contain no more than 10 µg/g of methanol, 2-butanone, and isobutanol and no more than 0.035 % of 2-propanol, ethyl acetate, and propylene glycol (omitted in this example) in combined total. In this example, the results satisfy those values for all five types of compounds tested.

Table 3 Content of 5 Components in Actual Samples

Compound	Content in 1 g
Methanol	1.954 µg
2-Propanol	N.D.
2-Butanone	N.D.
Ethyl acetate	N.D.
Isobutanol	N.D.

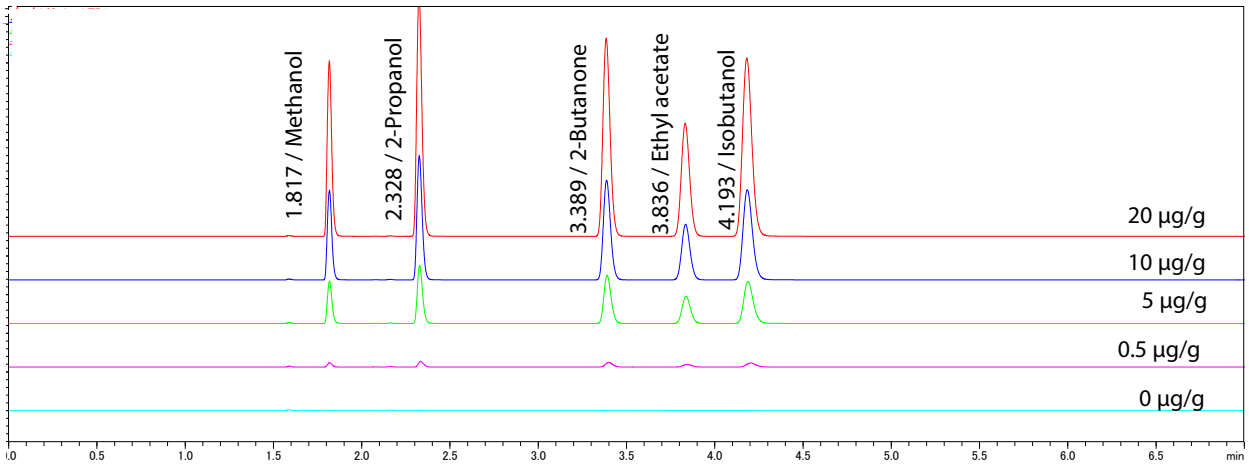

Fig. 1 Chromatograms from Samples with Standard Sample Added to Commercial Sucrose Fatty Acid Ester

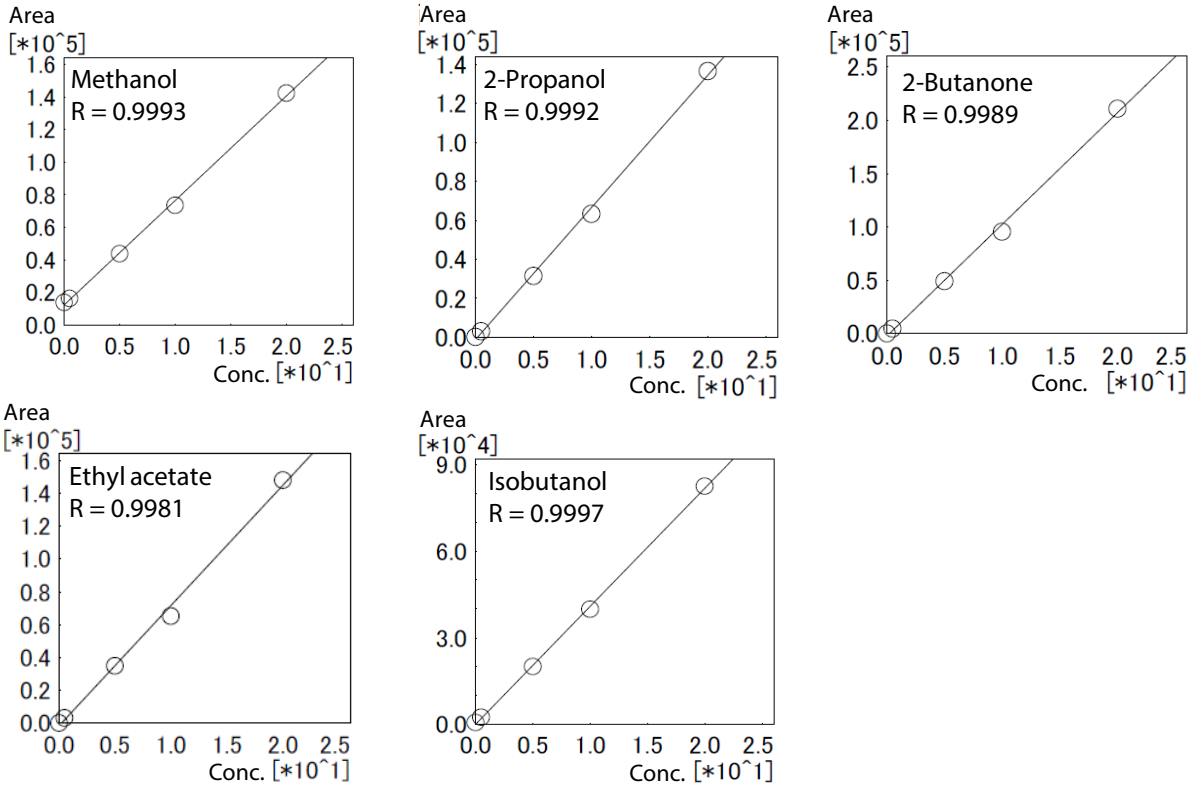

Fig. 2 Calibration Curve for Each Component

■ Summary

The Analysis conditions specified in Japan's Specifications and Standards for Food Additives were used as a reference to analyze residual solvents in commercial sucrose fatty acid ester, with the split mode, which is the optimal condition for the headspace sampler (HS-20 NX). The results showed good peak separation, of course, and calibration curves with good correlation and good quantitation values.

An external view of the system used in this example is shown in Fig. 3 for reference purposes.

Fig. 3 Nexis™ GC-2030 + HS-20 NX (Loop Model)

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Reference Documents

2018 Specifications and Standards for Food Additives (9th Edition), Consumer Affairs Agency, Ministry of Health, Labour and Welfare

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