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Application News

Nexis™ GC-2030 Gas Chromatograph

Analyzing Residual Solvents in Sucrose Fatty Acid Esters by HS-GC-FID Analysis (FCC 11)

Akara Hashimoto

User Benefits

- Four impurity types of sucrose fatty acid ester can be analyzed by HS-GC-FID analysis based on FCC 11 requirements.
- Target substances can be quantitated easily using the standard addition method.
- Using a headspace sampler (HS) eliminates the need for tedious pretreatment processes.

■ 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 gives them a hydrophilic sucrose end and a lipophilic fatty acid end, which makes sucrose fatty acid esters an excellent non-ionic surfactant. Therefore, they are widely used in foods, samples, 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 Japan's Specifications and Standards for Food Additives, refer to Shimadzu Application News 01-00486.)

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 FCC 11.

■ Measurement Sample Preparation

Measurement samples were prepared as follows for quantitative analysis by the standard addition method. First, 500 mg each of methanol, methyl ethyl ketone, ethyl acetate, and isobutanol were precisely weighed and mixed. Then water was added to make exactly 50 mL of solution (Solution A). Solution A was diluted with water to concentrations of 200 µg/mL (Solution B), 300 µg/mL (Solution C), and 400 µg/mL (Solution D).

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

The quantities of each component contained in each standard sample are indicated in Table 1.

Table 1 Content of Each Component in 1 g of Sample

Solvent Concentrations in Solutions (µg/mL)	0	200	300	400
Solvent Concentrations in Samples (mg/kg)	0	1	1.5	2
Standard Sample	Water	B	C	D

■ Analysis Conditions

The analysis conditions are shown in Table 2. The headspace sampler (HS-20 NX) loop mode was used to inject and quantitate 1 mL quantities.

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 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 maximum concentration levels permitted by the regulation and the quantitated values for the four types of compounds in the samples used for this example are indicated in Table 3. The results show that the concentrations of all solvents were below the regulation maximums.

Table 3 Regulation Maximum and Actual Quantitation Values for 4 Components in Actual Samples

Compound	Quantitation Value	Regulation Maximum Value
Methanol	2.37 mg/kg	10 mg/kg
Methyl ethyl ketone	N.D.	10 mg/kg
Ethyl acetate	N.D.	350 mg/kg
Isobutanol	0.16 mg/kg	10 mg/kg

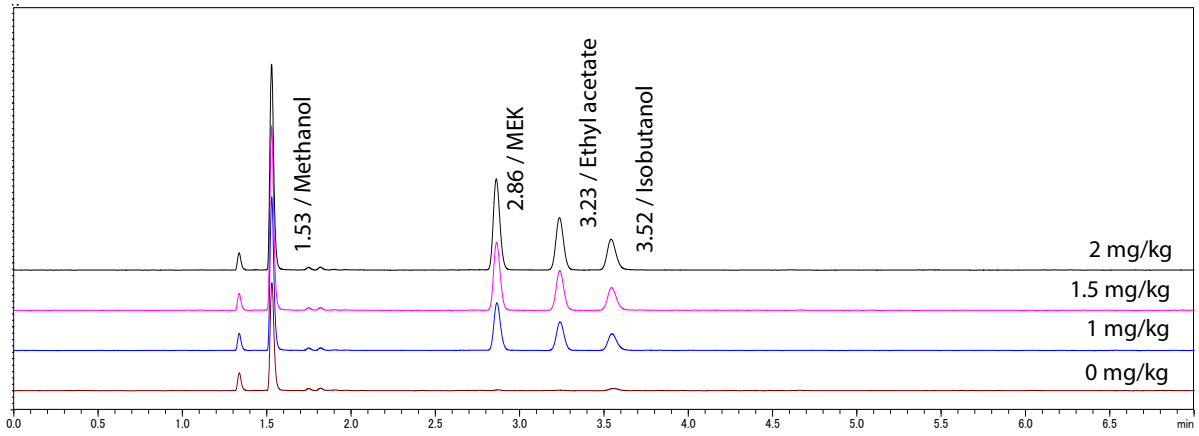

Fig. 1 Chromatograms from Samples with Standard Sample Added to Commercial Sucrose Fatty Acid Ester

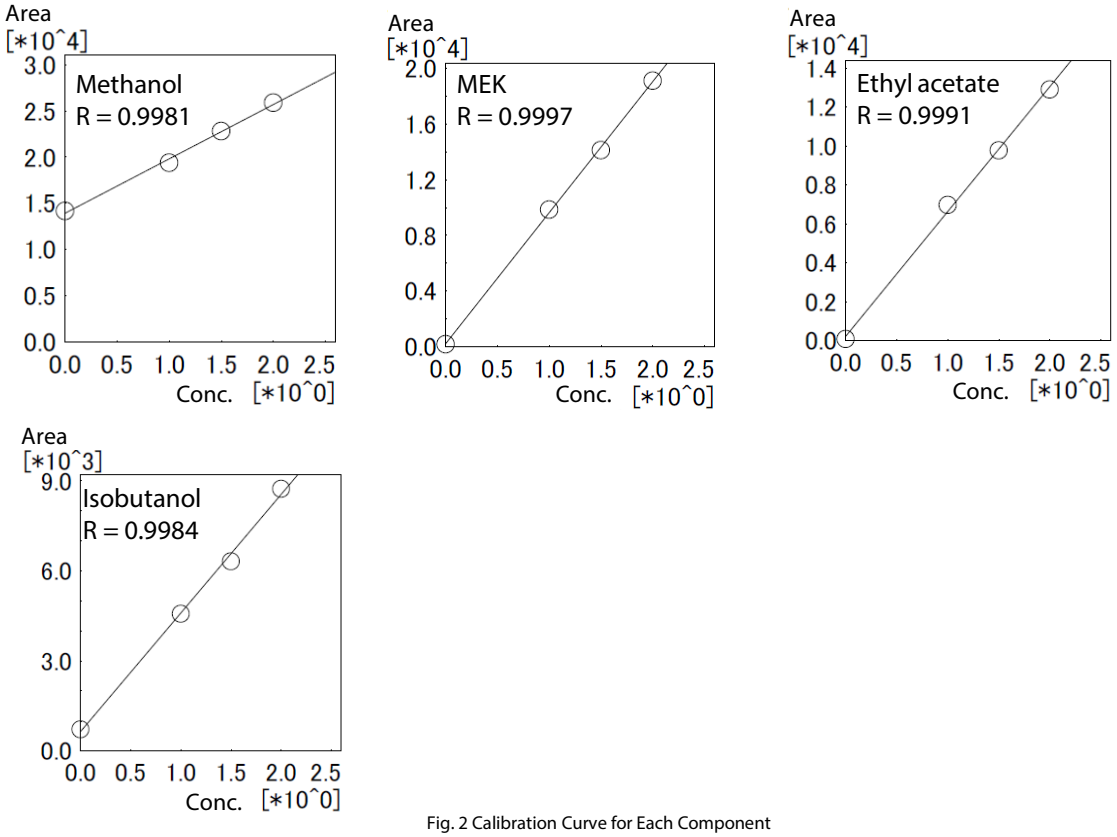

Fig. 2 Calibration Curve for Each Component

■ Summary

Condition settings specified in U.S. Food Chemicals Codex (FCC 11) were used as a reference for using a headspace sampler (HS-20 NX) to analyze residual solvents in commercial sucrose fatty acid ester. The results showed good peak separation, of course, calibration curves with good correlation, and good quantitation values.

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

Reference Documents
United States Food Chemical Codex 11 (FCC 11)

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

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