ICS 71.100.40
CCS G 72

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 45000-2024

Surface active agents - Determination of components of sucrose fatty acid esters - Liquid chromatography

表面活性剂 蔗糖脂肪酸酯的组成分析 液相色谱法

Issued on: November 28, 2024 **Implemented on: June 1, 2025**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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1 Scope

This document describes the liquid chromatographic method for the compositional analysis of the surfactant sucrose fatty acid ester.

This document applies to the compositional analysis of synthetic sucrose fatty acid ester products.

4 Principle of the method

Sucrose fatty acid ester is made up of products of different degrees of substitution, that is monoester, diester, triester and tetraester, together with unreacted fatty acid. After the sample has been dissolved and filtered, a high performance liquid chromatograph is used to separate and detect the sucrose fatty acid esters of the different degrees of substitution and the fatty acid, and quantification is by area normalization.

5 Reagents and materials

5.1 Tetrahydrofuran: chromatographic grade.

5.2 Sucrose fatty acid ester reference substance, CAS 37318-3-3 as printed on the original: purity not less than 95 %.

5.3 Preparation of the sucrose fatty acid ester reference solution: weigh 0.125 g of the reference substance (5.2), to the nearest 0.001 g, into a 25 mL volumetric flask, add a suitable quantity of tetrahydrofuran (5.1) to dissolve it, sonicate for 1 min and make up to the mark with tetrahydrofuran (5.1). Filter through a 0.45 micrometre organic-phase microporous membrane and keep the filtrate for use.

6 Instruments and reference chromatographic conditions

6.1.1 High performance liquid chromatograph: fitted with a differential refractive index or evaporative light scattering detector and a column thermostat system.

6.1.2 Analytical balance: accuracy 0.1 mg.

6.1.3 Microsyringe: 10 microlitres.

6.1.4 Organic-phase microporous membrane: 0.45 micrometres.

6.1.5 Ultrasonic generator: ultrasonic power not less than 700 W, temperature control range 20 degrees Celsius to 80 degrees Celsius.

6.2.1 Chromatographic column: four high performance gel permeation chromatography columns (SuperH2500, 6.0 mm by 150 mm, 3 micrometres) in series, or equivalent.

6.2.2 Column temperature: 40 degrees Celsius.

6.2.3 Mobile phase: tetrahydrofuran.

6.2.4 Mobile phase flow rate: 0.4 mL/min.

6.2.5 Injection volume: 10 microlitres.

7 Test procedure

7.1 Test solution. Weigh 0.125 g of the sample, to the nearest 0.001 g, into a 25 mL volumetric flask, add tetrahydrofuran to dissolve it, sonicate for 1 min and make up to the mark. Filter through a 0.45 micrometre organic-phase microporous membrane and keep the filtrate for use.

7.2 Chromatographic analysis. Inject the prepared sucrose fatty acid ester reference solution (5.3) and the test solution (7.1) separately, and identify the chromatographic peaks of the components of the sample from the retention times of the reference substance. Identification is made by comparing the retention times of the peaks of the sample chromatogram with those of the peaks of the reference chromatogram. Figure 1 is a typical chromatogram of the sucrose fatty acid ester reference solution (5.3); the areas of the individual ester peaks vary with the degree of substitution of the sucrose fatty acid ester.

8 Calculation of the result

From the peak areas of the products of different degrees of substitution in the chromatogram, that is monoester, diester, triester and tetraester, and the peak area of the fatty acid, the content of sucrose fatty acid ester in the sample is calculated by Formula (1) and expressed as a percentage. In the formula X is the content of sucrose fatty acid ester; Ai is the peak area of the products of different degrees of substitution, that is monoester, diester, triester and tetraester; and B is the sum of the peak areas of the fatty acid. The content is the sum of the Ai divided by the sum of the Ai plus B, multiplied by 100 %.

9 Precision

The absolute difference between two independent results obtained under repeatability conditions shall not exceed 2 % of their arithmetic mean.