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National Food Additive Standard- Sucralose

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Report Highlights:

On May 5, 2010, China notified the WTO of "National Food Safety Standard: Food Additives - Sucralose" as SPS/N/CHN/208. This measure "applies to the technical requirements and testing methods for sucralose." The date for submission of final comments to the WTO is May 20, 2010. The proposed date of entry is May 30, 2010. This report is an INFORMAL translation of this document.

Executive Summary:

On May 5, 2010, China notified the WTO of "National Food Safety Standard: Food Additives - Sucralose" as SPS/N/CHN/208. This measure “applies to the technical requirements and testing methods for sucralose.” The date for submission of final comments to the WTO is May 20, 2010. The proposed date of entry is May 30, 2010.

Thanks go to the Keller and Heckman LLP Shanghai Representative Office for their assistance in translating this document.

This report contains an UNOFFICIAL translation of National Food Safety Standard: Food Additives - Sucralose.

General Information:

BEGIN TRANSLATION

GB National Food Safety Standard
GB/T 5009.11—xxxx
To replace GB/T 5009.11—2003

National food safety standard

Food Additive Sucralose

(Draft for soliciting opinions)

Issued on XX-XX, 2010 Implemented from XX-XX, 2010

Issued by the Ministry of the People’s Republic of China

Preface

The revision of this Standard is based on “Sucralose” at the sixth version of Food Chemicals Codex (FCC 6).

The main technical difference of this Standard from “Sucralose” in the sixth version of Food Chemicals Codex (FCC 6) is,

--Revision on the identification test.

Appendix A of this Standard is a normative appendix.

National Food Safety Standard

Food Additive Sucralose

1. **Scope**

This Standard is applicable to sucralose products made with cane sugar as a raw material, whose three hydroxyl radicals are substituted by chlorine atom.

2.**Normative reference documents**

Reference documents in this Standard are indispensable for the application of this Standard. For the reference documents with indicated dates, only their version with date indicated shall be applied to this Standard. For the reference documents without indicated dates, the latest version shall be applied to this Standard.

3. **Chemical name, molecular formula, structural formula and molecular weight**

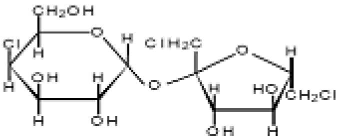
1.1 Chemical name

1,6-Dichloro-1,6-dideoxy-β-D-fructofuranosyl-4-chloro-4-deoxy-α-D-galactopyranoside

1.2 Molecular formula

$C_{12}H_{19}Cl_3O_8$

1.3 Structural formula



1.4 Relative molecular weight

397.64 (according to international relative atomic mass table in 2001)

4. **Technical requirements**

4.1 Sensory requirements: Compliance with the regulations in Table 1 below.

Table 1 Sensory requirements

Item	Requirements	Test method
Color	White to near white.	Take proper amount of sample, and place it in the clean and dry white ceramic plate. Then observe its color and state under natural light, and smell its odor.
Odor	No smell.	
State	Crystallized powder, no visible foreign substance to normal eyesight.	

4.2 Physical and chemical indexes: Compliance with regulations in Table 2.

Table 2 Physical and chemical indexes

Item	Index	Test method
Content (based on the dry product), w/%	98.0~102.0	A.3 in Appendix A
Specific optical rotation, αm (20℃,D)/[(º)·dm2·kg-1]	+84.0~+87.5	A.4 in Appendix A
Water content, *w*/% ≤	2.0	GB/T 6283
Ignition residue , *w*/% ≤	0.7	GB/T 9741[a]
Hydrolysis products	After the test	A.5 in Appendix A
Related substances	After the test	A.6 in Appendix A
Methanol, *w*/% ≤	0.1	A.7 in Appendix A
Lead (Pb)/ (mg/kg) ≤	1	GB 5009.12
[a] Sample weight 1 g ~2g.		

Appendix A

(Normative appendix)

Test method

A.1 General rules

Unless otherwise specified, only the reagents confirmed as analytically pure and the water specified by the GB/T 6682 standard shall be used during the analysis. The standard titration solutions, the standard solutions, preparations and products for the determination of impurities, shall be prepared according to the regulations in GB/T 601, GB/T 602 and GB/T 603 standards. All the solutions in this test shall be water solution unless specific solvents are indicated for the preparation of solutions.

A.2 Identification test

A.2.1 In the test of sucralose content, the retention time of main peak for test solution in the liquid phase chromatogram shall be the same as that for the sucralose in standard solution.

A.2.2 In the test for related substances, the Rf value of main color spot in the test solution shall be same as that of main color spot for standard solution on the thin chromatogram.

A.3 Determination of contents

A.3.1 Reagents and solutions

α. Acetonitrile: chromatography grade.

β. Standard product of sucralose: mass fraction ≥98.0% (known mass fraction).

A.3.2 Instruments and devices

High performance liquid chromatography, with refractive index detector

A.3.3 Reference chromatographic conditions

α. Chromatographic column: RadPakC18 reverse-phase chromatographic column, 8mm×10cm, particle size 5μm, or equivalent chromatographic column.

β. Mobile phase: Mix 150mL chromatography-grade acetonitrile with 850mL water, and after it is even, pass it with 0.45μm filter membrane and ultrasonic degassing for spare use.

χ. Column temperature: room temperature.

δ. Flow rate: 1.5 mL/min.

ε. Sample injection amount: 20 μL.

ϕ. Retention time of sucralose: about 9 minutes. To ensure that the required retention time is achieved, it is possible to adjust the ratio of mobile phase when necessary.

Note: The applicability of this system shall be such that after injection of standard solution for two times, the relative tolerance for the response area shall be less than 2.0%.

A.3.4 Analysis steps

A.3.4.1 Preparation of standard solution

Weigh about 0.025g standard sucralose product (with accuracy to 0.0001g) and dissolve it with mobile phase, then transfer it to 25mL volumetric flask, add mobile phase to the required graduation with constant volume. The resulted solution is filtered by the 0.45μm filter membrane and ready for use.

A.3.4.2 Preparation of test solution

Weigh about 0.025g test specimen (with accuracy to 0.0001g) and dissolve it with mobile phase, then transfer it to 25mL volumetric flask, add mobile phase to the required graduation with constant volume. The resulted solution is filtered by the 0.45μm filter membrane and ready for use.

A.3.5 Determination

Measure the standard solution and test solution under the above chromatographic conditions with sample feeding amount of 20μL. Repeat the sample feeding for one time. Then calculate the average value of peak area.

A.3.6 Result calculation

Content X_1 of sucralose is calculated by formula (A.1):

$$X_1 = \frac{A_U \times M_s \times P}{A_S \times M_U \times (1 - X_0)} \times 100 \qquad \text{(A.1)}$$

Where,

X_1——Content of sucralose in test solution, %;

A_U——Average value of main chromatographic peak area in test solution;

M_S——Mass of measured standard sucralose product, unit: gram (g);

P——Indicated content of sucralose in standard sucralose product, %;

A_S——Average value of main chromatographic peak area in standard solution;

M_U——Mass of measured test sample, unit: gram (g);

X_0——Water content of test sample, %.

A.3.7 Allowable deviation

Test results shall be based on arithmetic mean value of the results from parallel determination. The absolute deviation between two independent test results under the repeated conditions shall be no more than 2% of the arithmetic mean value.

A.4 Specific optical rotation

A.4.1 Weigh about 1g sucralose specimen (with accuracy to 0.001g) and dissolve it with water, then transfer it to 100mL volumetric flask, add water to the graduation with constant volume, and shake it up well. Measurement temperature is 20℃±0.5℃.

Specific optical rotation α_m (20℃, D) is expressed with "(°) ·dm^2·kg^{-1}", and calculated in formula (A.2):

$$\alpha_m \left(20\ ^\circ C\ , D\right) = \frac{\alpha}{l \rho_\alpha} \qquad \text{(A.2)}$$

Where,

α —— Measured angle of optical rotation, unit: degree (°);

l —— Length of polarimeter tube, unit: decimeter (dm);

ρ_α ——Mass concentration of active constituents in solution, unit: gram per milliliter (g/mL).

A.4.2 Other items shall be based on the regulations in GB/T 613-2007.

A.5 Hydrolysis products

A.5.1 Reagents and solutions

α. P-Anisidine.

β. Phthalic acid.

χ. Methanol.

δ. Mannitol.

ε. Fructose.

A.5.2 Instruments and devices

TLC plate: applied with 0.25mm thick Merk silica gel 60 or equivalent substances.

A.5.3 Analysis steps

A.5.3.1 Preparation of color developing reagent

Dissolve 1.23g p-Anisidine and 1.66 g phthalic acid into 100 mL methanol. Store the solution at the dark and cool place. The solution will have lost its effect in case of any discoloration.

A.5.3.2 Preparation of standard solution A

Weigh about 10g mannitol (with accuracy to 0.001g) and dissolve it with water, then transfer it to 100mL volumetric flask, add water to the required graduation with constant volume.

A.5.3.3 Preparation of standard solution B

Weigh 10g mannitol (with accuracy to 0.001g) and 0.04g fructose (with accuracy to 0.0001g), then transfer them to 100mL volumetric flask, and add water to the required graduation with constant volume.

A.5.3.4 Preparation of test solution

Weigh 2.5g test sample (with accuracy to 0.001g) and dissolve it with 5mL methanol, then transfer it to 10mL volumetric flask, add methanol to the required graduation with constant volume.

A.5.4 Determination

Take 5μL for each of standard solution A, standard solution B and test solution, and make the sample application at different positions on the same TLC plate respectively, where each solution is applied for five times (1μl per time), and each sample point shall be dry before the next sample application. The three sampling points shall have basically the same area. After the sample application, spray the color developing agent on it, and heat it in the drying oven at 100℃±2℃ for 15 minutes. Then immediately observe the TLC plate under the dark background, when the generated color of color spot in test solution shall not be darker than that of standard solution B.

Note: If the sampling point of standard solution A becomes black, it indicates that the heating time is too long for TLC plate, which shall be made again.

A.6 Related substances

A.6.1 Reagents and solutions

- α. Sucralose Standard: Mass fraction ≥ 98.0%.
- β. Acetonitrile.
- χ. Concentrated sulphuric acid.
- δ. Methanol.
- ε. Sodium chloride (NaCl) water solution: 5.0% (w/v).

A.6.2 Instruments and devices

TLC plate: applied with 0.2mm thickness C18-alkyl-modified silica gel (for example, Whatman LKC18) or other equivalent substances.

A.6.3 Analysis steps

A.6.3.1 Preparation of developing agent

Developing agent: Sodium chloride (NaCl) water solution: acetonitrile =70:30 (volume ratio).

A.6.3.2 Preparation of color developing reagent

Preparation of methanol solution with 15% concentrated sulphuric acid (volume fraction).

A.6.3.3 Preparation of standard solution

Weigh about 0.5g standard sucralose product (with accuracy to 0.001g) and dissolve it into 5.0mL methanol. This is called solution A. Absorb 0.5mL solution A, and add methanol to the required graduation with constant volume. The resulted solution is called solution B.

A.6.3.4 Preparation of test solution

Weigh 1.0g sucralose sample (with accuracy to 0.001g) and dissolve it into 10.0mL methanol.

A.6.4 Determination

Take 5μL for each of solution A and solution B, apply them on the bottom of TLC plate, and place the TLC plate into the chromatographic tank with developing reagent. When the developing reagent moves forward to 15cm, take out the TLC plate and let it be. After the developing reagent completely disappears by volatilization, spray the color developing agent on it, and place it into the drying oven for heating at the temperature of 125℃±2℃ with the duration of 10 minutes. The R_f value of main color spot in test solution shall be the same as that for solution A, and the color of other color spots in test solution shall not be darker than that in solution B.

R_f value——Ratio of the distance from the color spot to origin with the distance from front edge of solvent to origin after the development of test specimen.

A.7 Methanol

A.7.1 Reagents and solutions

α. Standard product of methanol: chromatography grade.

β. N-propanol: chromatography grade.

χ. Pyridine: chromatography grade.

A.7.2 Instruments and devices

Gas chromatograph: equipped with hydrogen flame ionization detector.

A.7.3 Reference chromatographic conditions

α. Chromatographic column: 2.1m×4mm (id) glass column, filled with 80~100 meshes polystyrene chromatographic stationary phase; or equivalent chromatographic column.

β. Carrier gas: nitrogen.

χ. Column temperature: 150℃.

δ. Sample inlet temperature: 200℃.

ε. Detector temperature: 250℃.

ϕ. Flow rate: 20mL/min.

γ. Sample injection amount: 1μL.

Note: The applicability of this system shall be such that after injection of standard solution for two times, the relative tolerance for the response area shall be less than 2.0%.

A.7.4 Analysis steps

A.7.4.1 Preparation of internal standard solution

Accurately absorb 1.0mL n-propanol and place it into the 100mL volumetric flask. Then add pyridine to the required graduation with constant volume. Shake it up well. Take 5mL from this solution, and place it into 500mL volumetric flask. Add pyridine to the required graduation with constant volume. Shake it up well.

A.7.4.2 Preparation of standard solution

Accurately absorb 2.0mL methanol and place it into the 100mL volumetric flask. Then add internal standard solution to the required graduation with constant volume. Shake it up well. Take 1.0mL from this solution, and place it into 100mL volumetric flask. Add internal standard solution to the required graduation with constant volume. Shake it up well.

A.7.4.3 Preparation of test solution

Weigh about 2g sucralose test sample (with accuracy to 0.001g), dissolve it with internal standard solution, transfer it into 10mL volumetric flask, and add internal standard solution to the required graduation to the constant volume. Then shake it up well.

A.7.5 Determination

Measure the standard solution and test solution under the chromatographic conditions in the above A.7.3 with sample injection amount of 1μL. Repeat the sample injection for one time. Then calculate the average value of peak area for methanol every time.

A.7.6 Result calculation

Content X_2 of methanol is calculated in formula (A.3):

$$X_2 = \frac{R_U \times 0.00158}{R_S \times M_U} \times 100 \quad \text{……………………………………} \quad (A.3)$$

Where,

X_2——Content of methanol in sucralose test solution, %;

R_U——Average value of peak area ratio between methanol and internal standard substance (n-propanol) in the test solution at two times of sample feeding;

0.00158——Methanol concentration in standard solution× specific weight of methanol × volume of test solution (2× 10^{-4} ×0.79×10);

R_S——Average value of peak area ratio between methanol and internal standard substance (n-propanol) in the standard solution at two times of sample feeding;

M_U——Mass of weighed sample, unit: gram (g).

A.7.7 Allowable deviation

Test results shall be based on arithmetic mean value of the results from parallel determination. The absolute deviation between two independent test results under the repeated conditions shall be no more than 10% of the arithmetic mean value.

END TRANSLATION