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National Food Safety Standards on Calcium

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FAIRS Subject Report

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Report Highlights:

On November 18, 2011, China notified the WTO of National Food Safety Standard: Food Additive Calcium Cyclamate as SPS/N/CHN/501. This standard applies to food additive calcium cyclamate, which is made from chemical combination of cyclohexylamine and sulfamic acid, and then reacts with calcium hydroxide. It specifies the technical requirements and testing methods for food additive calcium cyclamate. The date for submission of final comments to China is January 17, 2012. The proposed date of entry is to be determined. Comments can be sent to China's SPS Enquiry Point at sps@aqsiq.gov.cn. This report is an INFORMAL translation of this document.

General Information:
BEGIN TRANSLATION

GB National Food Safety Standard
GB XXXX—XXXX

National Food Safety Standard

Food Additive Calcium Cyclamate (Draft for Soliciting Opinions)

Date of Issue: XXXX-XX-XX

Date of Effectiveness: XXXX-XX-XX

Issued by the Ministry of Health of the People's Republic of China

National Food Safety Standard
Food Additive Calcium Cyclamate

1. Scope

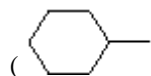
This standard applies to the food additive, Calcium Cyclamate, prepared from the cyclohexyl taurine, which is combined by and generated from the raw materials of cyclohexane and sulfamic acid, acted on calcium oxide.

2. Molecular formula, constitutional formula and relative molecular mass

2.1 Molecular formula

$C_{12}H_{24}CaN_2O_6S_2 \cdot nH_2O$ (dihydrate crystal $n=2$ and tetrahydrate crystal $n=4$)

2.2 Constitutional formula



$(NHSO_3)_2Ca \cdot nH_2O$

2.3 Relative molecular mass

Dihydrate crystal: 432.77 (according to international relative molecular mass in 2007);

Tetrahydrate crystal: 468.607 (according to international relative molecular mass in 2007).

3. Technical requirements

Organoleptic requirements: Appearance requirements: should conform to the requirements in table 1.

Table 1: Organoleptic Requirements

Item	Requirement	Method of inspection
Color	White	Apply adequate amount of sample onto a clean, dry and white porcelain dish and make a visual inspection under
Texture	Crystallizing particles or	

	crystallizing powders	natural lighting
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The physical and chemical indexes: shall conform to the requirements in Table 2.

Table 2. The physical and chemical indexes

Item	Index	Method of inspection
Calcium Cyclamate (in dry basis) w/%	98.0-101.0	A.4 in Appendix A
Loss on drying w/% ≤	Type A(2 crystal water): ≤9.5 Type B(4 crystal water): ≤16.5	A.5 in Appendix A
Cyclohexane (mg/kg) ≤	25	A.6 in Appendix A
Dicyclohexylamine	Test passed	A.7 in Appendix A
Lead (Pb) / (mg/kg) ≤	1	A.8 in Appendix A

Appendix A

Method of inspection

A.1 Cautions

Some of the testing processes as stipulated by the testing method may lead to dangerous situations, and adequate safety and protective measures shall be taken by the operator.

A.2 General Provision

Unless otherwise specified, only the reagents that have been identified as A.R and the Class III water as defined in GB/T6682-2008 can be used.

When no other requirement is noted, all the standard titration solution, the standard solution, preparation and products for determination of impurities that will be used by the testing method shall be prepared as prescribed in GB/T 601, GB/T 602

and GB/T 603; unless otherwise specified, all the solutions to be used shall be aqueous solution.

A.3 Identification test

A.3.1 Reagents and materials

A.3.1.1 Sodium nitrate: 100 g/L.

A.3.1.2 Hydrochloric acid solution: 1+3.

A.3.1.3 Barium chloride: 50 g/L.

A.3.2 Identification steps

A.3.2.1 About 1 g of sample is weighed to accurate to 0.0002 g, dissolved in 100 mL water as the sample solution.

A.3.2.2 Get 10 mL of such solution to be blended with 1 mL of Hydrochloric acid solution and 1 mL of Barium chloride solution, observe the solution, which shall be transparent; then add 1 mL of sodium nitrate to generate white deposition and escape of gases.

A.3.2.3 Take a piece of iron wire to dip the right amount of the sample to burn it in colorless fire, where the flame shall be brick red.

Determination is conducted as prescribed in GB/T614. The refractive index $n(20, D)$ shall be: 1.328 — 1.330.

A.4 Determination of Calcium Cyclamate content

A.4.1 Summary of method

When the reaction between Calcium Cyclamate and sodium nitrate is completed, the excessive sodium nitrate reacts with potassium iodide to generate the iodine under the acidity condition and the iodine becomes blue when touching the starch. The content of Calcium Cyclamate can be calculated according to the volume of the standard titration solution of sodium nitrate that has been consumed.

A.4.2 Reagents and materials

A.4.2.1 Hydrochloric acid solution: 1+1.

A.4.2.2 Potassium iodide-Amylum Test paper.

A.4.2.3 Standard titration solution of Sodium nitrate: $c(\text{NaNO}_2) = 0.1 \text{ mol/L}$.

A.4.3 Analysis steps

A.4.3.1 Weigh 0.4 g (accurate to 0.0002 g) of the sample that has been cooled just to the room temperature when it is dried for 2 hours at the temperature at 140°C into a 250 mL beaker.

A.4.3.2 Add into 50 mL water and 5 mL hydrochloric acid solution to dissolve the sample.

A.4.3.3 At the room temperature, use 0.1 mol/ L sodium nitrate for titration; when titrating to the last 1 mL volumetric solution, it is required to add the solution drop by drop until the blue color appears when using the glass bar to touch one drop of the solution onto the potassium iodide-amylum test paper; when titration is complete, the abovementioned end-point phenomenon can re-appear when the titrated solution remains for one minute, which indicates the titration end-point, then record the volume of sodium nitrate that has been consumed.

A.4.3.4 At the same time of the determination, a blank test is conducted by using the same amount of the reagent solution without sample according to the same determination steps, and then record the consumption volume of V_0 .

A.4.4 Result calculation

The mass fraction of Calcium Cyclamate (in dry basis) is w_1 , and the numerical value is indicated by %, which is calculated according to formula (A.1):

$$w_1 = \frac{(V - V_0) \cdot cM}{m} \times 100 \dots \dots \dots (A.1)$$

Where: V = the volume of the standard titration solution of Sodium nitrate that has been consumed by the sample, in the unit of milliliter (mL).

V_0 = the volume of the standard titration solution of Sodium nitrate that has been consumed by the blank test, in the unit of milliliter (mL).

C = the accurate numerical value of the concentration of the standard titration solution of Sodium nitrate, in the unit of mole per liter (mol/L).

m = the mass of the sample, in the unit of gram (g).

M = the numerical value of Calcium Cyclamate, in the unit of gram per mole (g/mol) ($M=198.27$).

The arithmetic mean of the result of two measurements in the same condition is taken as the report result, where the absolute difference between the results of two measurements in the same condition shall not be more than 0.40%.

A.5. Determination of the Loss on Drying

A.5.1 Analysis step

Weigh about 2.0. g of the sample, accurate to 0.0001 g, and place it into a weighing bottle that has been dried to invariable mass at (140+2) °C in advance; and it is dried for 2 hours in a thermostatic drying chamber with the temperature at (140+2) °C and then weigh it when it is placed into a desiccator being cooled for 30 min.

A.5.2 Result calculation

The mass fraction of **the Loss on drying** is w_2 and the numerical value is indicated by %, which is calculated according to formula (A.2):

$$W_2 = \frac{m_1 - m_2}{m} \times 100 \dots \dots \dots (A.2)$$

Where:

m_1 = the mass of the sample before drying, in the unit of gram (g);

m_2 = the mass of the weighing bottle and the sample before drying, in the unit of gram (g);

m = the mass of the weighing bottle and the sample after drying, in the unit of gram (g).

The arithmetic mean of the result of two measurements in the same condition is taken as the report result, where the absolute difference between the results of two measurements in the same condition shall not be more than 0.20%.

A.6 Determination of Cyclohexane

A.6.1 Reagents and materials

A.6.1.1 Alkaline solution of Ethylene diamine tetraacetic acid disodium salt: weigh 100 g of Ethylene diamine tetraacetic acid disodium salt and 34 g of sodium hydroxide to be dissolved into water, which is diluted into 1000 mL with water.

A.6.1.2 the solution of methyl orange and boracic acid: weigh 2 g of methyl orange and 3 g of boracic acid, add in 1000 mL water to be heated on the water bath to be dissolved and then stand for more than 24 hours. Filter the solution before use.

A.6.1.3 The mixed liquor of trichloromethane and n-butyl alcohol: 20+1.

A.6.1.4 The mixed liquor of methanol and sulphuric acid: 50 +1.

A.6.1.5 Anhydrous sodium sulfate: Na₂SO₄.

A.6.1.6 Standard of Cyclohexane: Each liter contains 0.0025 mg of cyclohexane. Weigh 0.1 g of cyclohexane (accurate to 0.0002 g), place it into a 100 mL volumetric flask, add into 50 mL water and 0.5 mL hydrochloric acid, and dilute both with water to the scale when dissolved, shaken up. Accurately measure 5.0 mL of such diluent and place it into another 100 mL volumetric flask and dilute it with water to the scale, shaken up.

A.6.2 Analysis step

Weigh 10.0 mL of laboratory sample, accurate to 0.01 g, dissolve it into about 60 mL water, place it into a 100 mL volumetric flask, and dilute it with water to the scale, shaken up. Accurately soak up 10.0 mL of this solution and the standard solution of cyclohexane respectively and place them into two 60 mL separate funnels respectively; add into 10.0 mL of Alkaline solution of Ethylene diamine tetraacetic acid disodium salt and 15.0 mL of the mixed liquor of trichloromethane and n-butyl alcohol, respectively in turn, shaking for 2 min, and leave them standing; obtain individually the layer of trichloromethane, from which 10 mL of the extracting solution of trichloromethane is measured and taken respectively and place them into two 60 mL separate funnels respectively and then add in 2.0 mL of solution of methyl orange and boracic acid, shaking for 2 min, and leave them standing; obtain individually the layer of trichloromethane, into which 1 g of anhydrous sodium sulfate is add, shaking up, and leave them standing, from which 5.0 mL of trichloromethane solution is measured and taken, then place it into a colorimetric cylinder, into which the mixed liquor of methanol and sulphuric acid is added respectively, shaking evenly. The color displayed from the sample solution shall not darken than that of the standard. The sample and the value of the standard absorbency are determined by spectrophotometer: with 520 nm wavelength, calibrate the zero by trichloromethane, and shift in the solution respectively into 1 cm cell, the sample absorbency of the test result shall not be more than that of the standard absorbency.

A.7 Determination of Dicyclohexylamine

A.7.1 Reagents and devices

A.7.1.1 Trichloromethane: it is washed for three times, with which the water amount is one third of the Trichloromethane each time to dissolve out the lay of Trichloromethane for determination.

A.7.1.2 Sodium hydroxide solution: 40 g/L.

A.7.1.3 Sodium bicarbonate solution: 8.4 g/L.

A.7.1.4 Hydrochloric acid solution: 1→12.

A.7.1.5 Reagent A: Weigh 75 mL of bromphenol blue and add in 60 mL water, add in 10 mL of sodium bicarbonate solution, stir them to dissovle; use Hydrochloric acid solution to adjust the pH value to 4.0 and dilute it with water into 100 mL, shaking evenly, store it in shade and cool place to be used in 48 hours.

A.7.1.6 Reagent B: Weigh 200 mL of Hydrochloric acid solution and 166 mL of Glacial acetic acid and put them into a 1000 mL volumetric flask, dilute them with water to the scale, shaking evenly.

A.7.2 Analysis steps

A.7.2.1 Weigh 10.0 g of the sample (accurate to 0.01g) into a 250 mL separatory funnel; add in 100 mL of Alkaline solution of Ethylene diamine tetraacetic acid disodium salt to dissolve them; take another 100 mL of Alkaline solution of Ethylene

diamine tetraacetic acid disodium salt into another separatory funnel for blank test.

A.7.2.2 10 mL of Sodium hydroxide solution is respectively added in, and then 10 mL, 10 mL and 5 mL of trichloromethane is used for extraction respectively in turn, from which the extracted layer of trichloromethane is combined into another two separate funnels.

A.7.2.3 100 mL of water, 3 mL of reagent B and 1 mL of reagent A is respectively added in, shaking for three min, place it in shade for 30 min, when standing and layering, the layer of trichloromethane is respectively obtained into a 25 mL test tube and add in trichloromethane to 25 mL.

A.7.2.4 With 410 nm wavelength, use 5 cm cell to calibrate the zero by trichloromethane to determine the absorbency of the blank and the sample solution.

A.7.2.5 When the difference of the absorbency between the sample solution and blank test is less than 0.15, it shall deemed as passed.

A.8 Determination of lead

A.8.1 Colorimetric method (Arbitration method)

It is conducted according to GB/T5009.75.

A.8.2 Atomic absorption spectrometry

The determination is undertaken according to GB 5009.12. The sample is treated by “dry digestion process” as defined in GB/T5009.75. When Graphite Furnace Atomic Absorption Spectrometry is adopted, the sample solution can be diluted properly according to the actual status of the sample.

END TRANSLATION