

ICS 67.220.20
CCS X 40



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 29217-2012

Food additive cyclamic acid calcium

食品添加剂 环己基氨基磺酸钙

(English Translation)

Issued on: December 25, 2012

Implemented on: January 25, 2013

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

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1 Scope

This standard applies to cyclohexylamine and sulfamic acid as raw materials to produce cyclohexyl sulfamate and calcium hydroxide after the role of the resulting food Ingredients Cyclohexylaminosulfonate.

2.1 Molecular formula

$C_{12}H_{24}CaN_2O_6S_2 \cdot nH_2O$ ($n = 2$ or 4)

2.2 Structural formula

$[NHSO_3]_2Ca \cdot nH_2O$

2.3 Relative molecular mass

Dihydrate crystal products. 432.57 (according to the 2007 international relative atomic mass) Four water crystal products. 468.60 (according to the 2007 international relative atomic mass)

3.1 Sensory requirements

Shall comply with the provisions of Table 1.

Table 1 sensory requirements

The project requires a test method Color white State granular or powdered crystals Take appropriate sample, placed in a clean, dry white porcelain dish, in the self Under the light, the visual observation of color and state

3.2 Physical and chemical indicators

Shall comply with the provisions of Table 2.

Table 2 Physical and chemical indicators

project

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Dihydrate crystal products four crystal products Testing method (Based on dry basis) content, w /% 98.0 to 101.0 Appendix A, A.4 Dry reduction, w /% ≤ 9.5 16.5 Appendix A, A.5 Cyclohexylamine/(mg/kg) ≤ 25 Appendix A A.6 Dicyclohexylamine was tested in Appendix A, A.7 Lead (Pb)/(mg/kg) ≤ 1 Appendix A A.8 Appendix A Testing method A.1 Warning the test procedure specifies that some of the test procedures may lead to dangerous conditions and that the operator should take appropriate safety and protective measures.

A.2 General provisions Unless otherwise stated, only the reagents identified as analytical reagents and the tertiary water specified in GB/T 6682-2008 are used in the analysis.

Standard titration solution used in the test method, standard solution for the determination of impurities, preparations and articles, in the absence of other requirements, GB/T 601, GB/T 602 and GB/T 603. The solution used refers to the aqueous solution when no other requirements are indicated.

A.3 Identification test A.3.1 Reagents and materials A.3.1.1 Sodium nitrite solution. 100 g/L.

A.3.1.2 Hydrochloric acid solution. 1 3.

A.3.1.3 barium chloride solution. 50 g/L.

A.3.2 Identification method A.3.2.1 Weigh about 1 g of sample, dissolve in water and make it into 100 mL as sample solution. Take 10mL of the test solution, add 1mL of hydrochloric acid dissolved Liquid and 1mL barium chloride solution mixture, observe the solution should be transparent; then add 1mL sodium nitrite solution to generate white precipitation and escape gas.

A.3.2.2 Take the wire into the appropriate amount of sample, burn in a colorless flame, the flame was red.

A.4 Determination of calcium hexyl sulfamate (in dry basis) content A.4.1 Methodological Summary After the reaction of calcium cyclohexylsulfamate with sodium nitrite is complete, excess sodium nitrite reacts with potassium iodide under acidic conditions to form iodine, Case of starch blue. The content of calcium cyclohexyl sulfamate was calculated from the volume of the titration solution of sodium nitrite standard.

A.4.2 Reagents and materials A.4.2.1 Hydrochloric acid solution. 1 1.

A.4.2.2 Starch Potassium Iodide Test Paper.

A.4.2.3 standard titration solution of sodium nitrite. $c(\text{NaNO}_2) = 0.1\text{mol/L}$.

A.4.3 Analysis steps A.4.3.1 Weigh 0.4g for 2h after drying at 140°C and just 0.4g in the condenser just cooled to room temperature, accurate to 0.0002g in 250mL Beaker.

A.4.3.2 Add 50 mL of water and 5 mL of hydrochloric acid solution to dissolve.

A.4.3.3 titration with sodium nitrite standard titration, to the final 1mL titration solution should be added dropwise until the glass rod Dipped in 1 drop solution on the starch potassium iodide test paper immediately appeared blue, when the titration is complete, the drip maintained for 1min can still reproduce the end of the current as indicated by the titration end point, the volume of the standard titration solution of sodium nitrite consumed is recorded.

A.4.3.4 At the same time as the determination, the same amount of reagent solution was used as a blank test in the same procedure as in the measurement, Record the consumption volume V_0 .

A.4.4 Calculation of results the mass fraction w_1 of calcium (based on dry basis) of cyclohexylsulfamate is calculated according to formula (A.1) $w_1 = (V - V_0) c M m \times 1000 \times 100\%$ (A.1) where.

V - volume of sodium nitrite standard titration solution in milliliters (mL);

V_0 --- blank consumption of sodium nitrite standard titration solution volume, in milliliters (mL);

c - the exact value of the concentration of sodium nitrite standard titration solution in moles per liter (mol/L);

m - the quality of the sample, in grams (g);

1000 --- conversion factor;

M - the molar mass of calcium cyclohexylsulfamate in grams per mole (g/mol) ($M = 198.27$).

The arithmetic mean of the results of two parallel measurements is the result of the report. The absolute difference between the two parallel determinations is not more than 0.40%.

A.5 Determination of dry reduction A.5.1 Analysis steps Approximately 2.0 g of the sample was weighed to 0.0002 g and placed in a preheated flask at $140^\circ\text{C} \pm 2^\circ\text{C}$ to a constant mass. in $140^\circ\text{C} \pm 2^\circ\text{C}$ constant temperature drying oven for 2h, placed in a dryer for 30min cooling.

A.5.2 Calculation of results the mass fraction w_2 of the drying reduction is calculated according to the formula (A.2) $w_2 = m_1 - m_2 m \times 100\%$ (A.2) where.

m - the mass of the sample before drying, in grams (g);

m1 --- the quality of the flask and the sample before drying, in grams (g);

m2 --- Weigh the quality of the bottle and the sample after drying, in grams (g).

The arithmetic mean of the results of two parallel measurements is the result of the report. The absolute difference between the two parallel determinations is not more than 0.20%.

A.6 Determination of cyclohexylamine A.6.1 Reagents and materials A.6.1.1 anhydrous sodium sulfate.

A.6.1.2 Alkaline ethylenediaminetetraacetic acid disodium solution. Weigh 100g of ethylenediamine tetraacetic acid and 34g of sodium hydroxide, add water and dissolve it.

Water diluted to 1000 mL.

A.6.1.3 methyl orange boric acid solution. Weigh 2g of methyl orange and boric acid 35g, add water 1000mL, on the water bath to heat it to dissolve More than 24 hours. Filter before use.

A.6.1.4 Mixture of Trichloromethane and n-Butanol. 20 1.

A.6.1.5 Mixture of Methanol and Sulfuric Acid. 50 1.

A.6.1.6 Cyclohexylamine standard solution. 0.0025 mg of cyclohexylamine per milliliter. Weigh the cyclohexylamine 0.1g, accurate to 0.0002g, set 100mL volumetric flask, add 50mL of water and hydrochloric acid 0.5mL, dissolved and diluted with water to the mark, shake. Accurately measure 5.0mL, placed on Another 100mL volumetric flask diluted with water to the mark, shake. Accurately measure the dilution of 5.0mL set 100mL volumetric flask, with water Dilute to the scale, shake it.

A.6.2 Analysis steps Weigh 10.0g sample, accurate to 0.01g, dissolved in about 60mL water, placed in 100mL volumetric flask, diluted with water to the mark, shake uniform. Accurately absorb the solution and cyclohexylamine standard solution of 10.0mL, were placed in two 60mL separatory funnel, respectively, by adding Alkaline ethylenediamine tetraacetic acid disodium salt solution 10.0mL, chloroform and n-butanol mixture 15.0mL, shaking 2min, standing, take trichloride Methane layer, the amount of chloroform extraction of 10.0mL, placed in the other two 60mL separatory funnel, each adding methyl orange boric acid solution 2.0mL, shake 2min, standing, take the chloroform layer, add anhydrous sodium sulfate 1g, shaking, standing, the amount of chloroform solution 5.0mL, Placed in the color tube, the addition of methanol and sulfuric acid mixture solution 0.5mL, shake. The color of the sample solution should not be deeper than the standard. With spectrophotometry Measurements and standard absorbance. at 520nm wavelength, with chloroform correction of zero, the solution were moved into 1cm cuvette, Test results The sample absorbance should not be greater than the standard absorbance.

A.7 Determination of bicyclohexylamine A.7.1 Reagents and materials A.7.1.1

Trichloromethane. washed three times with water, each time the amount of water for