



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

GB 1903.20-2016

**Food safety national standard food nutrition enhancers --
Thiamine nitrate**

食品安全国家标准 食品营养强化剂 硝酸硫胺素

(English Translation)

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1 Scope

This standard applies to acrylonitrile or the like as a main raw material malononitrile food fortifier by chemical synthesis of thiamine nitrate.

2.1 Chemical Name

4-methyl-3 - [(2-methyl-4-amino-5-pyrimidinyl) methyl] -5- (2-hydroxyethyl) thiazole nitrate bromide Formula 2.2 $C_{12}H_{17}N_5O_4S$

2.4 Molecular Weight

327.37 (according to 2013 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall comply with the requirements in Table 1.

Table 1 Sensory requirements

Project requires test methods White or white color Odor odorless or slight specific odor
State of powder or crystalline powder Take about 2g sample is placed in a clean, dry white dish, under natural light, Observe the color and state, smell the smell

3.2 Physical and Chemical Indicators

Physical and chemical indicators should be consistent with the provisions of Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Thiamine nitrate ($C_{12}H_{17}N_5O_4S$, dry basis) content, w /% ≥ 98.0

Appendix A A.4 pH (20g/L) 6.0 ~ 7.5 GB/T 23769 Loss on drying, w /% ≤ 1.0 A.5
 Appendix A Residue on ignition, w /% ≤ 0.1 A.6 Appendix A Chloride (based on Cl), w /%
 ≤ 0.06 A.7 Appendix A Sulfate (in terms of SO₄), w /% ≤ 0.011 A.8 Appendix A Lead
 (Pb)/(mg/kg) ≤ 1.0 GB 5009.12 Total arsenic (As)/(mg/kg) ≤ 1.0 GB 5009.11 Appendix A
 Testing method A.1 Safety Tips Reagents of this standard test methods used toxic or
 corrosive, operating according to the relevant regulations, required careful operation. If
 splash to the skin immediately wash with water, severe cases should be treated
 immediately. , Be carried out in a fume hood when using a volatile acid.

A.2 General Provisions the reagents used in this standard, unless otherwise specified, only
 confirmed in the analysis of analytical grade reagents and GB/T 6682 in a predetermined
 three water. Standard Solution, product formulation and testing methods required, in the
 absence of other requirements specified, are by GB/T 601, GB/T 603 Preparation of
 regulations.

A.3 Identification Test A.3.1 solubility test Slightly soluble in water, slightly soluble in
 methanol.

A.3.2 color ferrous sulfate A.3.2.1 Reagents and materials A.3.2.1.1 sulfuric acid.

A.3.2.1.2 ferrous sulfate solution. Take 8g ferrous sulfate (FeSO₄ · 7H₂O), was added
 100mL boiling over cold water and dissolved, shake, Using now.

Procedure A.3.2.2

Take a sample of approximately 10mg, test tube, add water to dissolve 1mL, the same
 amount of sulfuric acid, mixed and allowed to cool, dissolved ferrous sulfate was added
 along the wall Liquid, so that liquid into two layers, then the interface was brown liquid.

A.3.3 chromogenic lead acetate A.3.3.1 Reagents and materials A.3.3.1.1 lead acetate.

A.3.3.1.2 lead acetate solution. Take lead acetate 10g, add boiling over cold water was
 dissolved, the solution was added dropwise acetic clarified, plus boiling over cold Water
 volume to 100mL, that is, too.

A.3.3.1.3 sodium hydroxide solution. 100g/L.

Procedure A.3.3.2

Sample taken about 5mg, which was dissolved in a mixed solution of a solution of lead
 acetate and 1mL 1mL sodium hydroxide solution, the solution was yellow, water bath the
 mixed solution was heated for several minutes, the solution turned brown, black precipitate
 on standing due.

A.3.4 Fluorescence

A.3.4.1 Reagents and materials A.3.4.1.1 sodium hydroxide solution. 40g/L.

A.3.4.1.2 potassium ferricyanide solution. 100g/L.

A.3.4.1.3 hydrochloric acid solution. 36.5g/L.

A.3.4.1.4 n-butanol.

Procedure A.3.4.2

Sample taken about 5mg, after adding sodium hydroxide solution to dissolve 2.5mL, 0.5mL was added potassium ferricyanide solution and 5mL n-butanol, Qiang Lie Shaking 2min, placed allowed to separate, the upper layer was a strong blue fluorescence. After about 1mL hydrochloric acid solution was added, the fluorescence disappeared; then about After 3mL of sodium hydroxide solution, and show fluorescence.

A.4 Determination of thiamine nitrate ($C_{12}H_{17}N_5O_4S$, dry basis) content A.4.1 Reagents and materials A.4.1.1 anhydrous formic acid.

A.4.1.2 acetic anhydride.

A.4.1.3 perchloric acid standard titration solution. $c(HClO_4) = 0.1mol/L$.

A.4.2 Analysis step

Weigh about 0.14g sample, accurate to 0.0001g, placed in a conical flask, add anhydrous formic acid dissolved 5mL, 70mL acetic anhydride was added, According to potentiometric titration, the titration standard solution was titrated with perchloric acid, and titration with blank correction.

A.4.3 calculation results Thiamine nitrate ($C_{12}H_{17}N_5O_4S$, dry basis) content of the mass fraction w_1 , calculated according to formula (A.1).

$(V-V_0) \times c \times M$

$m \times (1-w_2) \times 1000 \times$

100% (A.1)

Where.

V --- standard sample consumption perchloric acid titration solution volume in milliliters (mL);

--- consumption volume V_0 blank perchloric acid standard titration solution in milliliters (mL);

The actual concentration of standard C --- perchloric acid titration solution in units of moles per liter (mol/L);

--- M thiamine nitrate ($2C_{12}H_{17}N_5O_4S$) Molar mass in grams per mole (g/mol) [$M(2C_{12}H_{17}N_5O_4S) = 163.7g/mol$];

--- m sample mass in grams (G);

w_2 --- A.5 measured by loss on drying,%;

1000 --- conversion factor.

The arithmetic average of the measurement results parallel the measurement results, the measurement results of two parallel allows absolute difference not more than 0.3%.

Determination of loss on drying A.5 A.5.1 Analysis step Weigh about 1g sample, accurate to 0.0001g, was placed on the dried to constant weight weighing bottle 105 °C ± 2 °C, at 105 °C ± 2 °C Oven dried to constant weight.

A.5.2 calculation results Loss on drying mass fraction w_2 , calculated according to equation (A.2).

$m_1 - m_2$

$m \times 100\%$

Where.

The total mass of the sample before drying and M_1 --- weighing bottles in grams (G);

--- M_2 After drying and weighing the total mass of the sample bottles in grams (G);

--- m sample mass, in grams (g).

The arithmetic average of the measurement results parallel the measurement results, the measurement results of two parallel allows the absolute difference is not greater than 0.1%.

Determination of residue on ignition A.6 A.6.1 Analysis step Weigh about 1g sample, accurate to 0.0001g, was placed on ignition to constant weight in a porcelain crucible at 750 °C ± 50 °C, low heat and slowly add Heat to completely carbonized, After cooling, add 0.5mL of sulfuric acid from about the sample to completely wet, and heated to a low temperature after the sulfuric acid vapor divisible, into a high temperature furnace in to burning at 750 °C ± 50 °C constant weight.

A.6.2 calculation results Ignition residue content of w_3 , calculated according to formula (A.3).

$m_3 - m_4$

$m \times 100\%$

Where.

--- M_3 total mass of the crucible and the residue, in grams (G);

--- M_4 crucible mass in grams (G);

--- m sample mass, in grams (g).

Parallel to the arithmetic average of the measurement results of the measurement result, the absolute difference determination results parallel to 0.02%.