

ICS 67.220.20
CCS X 42



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

GB 14757-2010

Replacing GB 14757-1993

**National food safety standards -- Food additives -- Niacin
(English Translation)**

Issued on: December 21, 2010

Implemented on: February 21, 2011

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

Table of Contents

Foreword

1 Scope

2 Normative references

3 Chemical name, molecular formula, molecular mass and structural formula

3.1 Chemical Name

3.3 Formula

3.4 Relative molecular mass

4 Technical Requirements

Foreword

This standard replaces GB 14757-1993 "Food Additives niacin" this standard compared with GB 14757-1993 main changes are as follows.

- Loss on drying index from 1% revised to 0.5%;
- Added Infrared Spectroscopy;
- Increased arsenic indicators.

The Standard Annexes A and B are normative appendix.

This standard replaces the standards previously issued as follows.

--GB 14757-1993.

National Food Safety Standard Food Additives niacin

1 Scope

This standard applies to chemical synthesis of food additives niacin.

2 Normative references

The standard file referenced in the application of this standard is essential. For cited documents with dates, only the date of Version applies to this standard. For undated references, the latest edition (including any amendments) applies to this standard.

3.1 Chemical Name

Pyridine-3-carboxylic acid Formula 3.2 C₆H₅NO₂

3.3 Formula

COOH

3.4 Relative molecular mass

123.11 (according to 2007 international relative atomic weight)

4 Technical Requirements

4.1

Sensory requirements. comply with the requirements of Table 1.

Table 1 Sensory requirements

Project requires test methods White or white color proper amount of sample is placed in 50mL beaker, observe the color in natural light and texture. Smell it. Odour odorless or has a slight odor Organizational status crystalline powder

4.2

Physical indicators. in accordance with Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Niacin (dry basis), w /% 99.5 ~ 101.0 Appendix A A.4 Loss on drying, w /% ≤ 0.5 A.5 in Appendix A

Table 2 (Continued) physical and chemical indicators

Chloride (Cl dollars), w /% ≤ 0.02 A.6 in Appendix A Residue on ignition, w /% ≤ 0.1 Appendix A A.7 Arsenic (As)/(mg/kg) ≤ 2 Appendix A A.8 Melting point/°C 234 ~ 238 Appendix A, A.9 Heavy metals (Pb)/(mg/kg) ≤ 20 Appendix A A.10 Appendix A (Normative) Testing method A.1 Safety Tips Reagents The standard test methods used for toxic or corrosive, according to the relevant provisions of the operation, the operation need to be careful!

If splashed on the skin should immediately wash with water, severe cases should be treated immediately. When using a volatile acid, to be carried out in a fume hood.

A.2 General Provisions the reagents used in this standard, unless otherwise noted, only in the analysis confirmed analytically pure reagents and GB/T 6682-2008 in Regulation Given three water.

Standard Solution, Test Method for preparation and products required, did not indicate

when the other requirements, according to GB/T 601, GB/T 603 Provisions prepared.

A.3 Identification Test A.3.1 Reagents and materials A.3.1.1 95% ethanol.

A.3.1.2 2,4- dinitro chlorobenzene.

A.3.1.3 Potassium hydroxide solution. take 3.5 g of potassium hydroxide, add 100 mL ethanol to dissolve, after resting the supernatant.

A.3.1.4 sodium hydroxide solution. 4 g/L.

A.3.1.5 copper sulfate solution. 125 g/L.

A.3.2 Identification method A.3.2.1 Identification of the pyridine ring A.3.2.1.1 Method summary Niacin plus 2,4-dinitro-chlorobenzene melted to form a quaternary ammonium compound, and then potassium hydroxide solution, which was purple (Vongerichten reaction).

A.3.2.1.2 analysis step Laboratory samples taken from about 4 mg, 8 mg 2,4- plus dinitro chlorobenzene, Research uniform, a test tube, slowly heated to melt, and then the number of heating Seconds, cooled to room temperature, 3 mL ethanol solution of potassium hydroxide. That was purple.

A.3.2.2 Identification precipitation reaction with copper sulfate A.3.2.2.1 Method summary Carboxyl group-containing nicotinic acid, neutralized with sodium hydroxide, the case of copper sulfate to generate light blue nicotinic acid precipitation.

A.3.2.2.2 analysis step Take about 50 mg laboratory samples, add 20 mL of water dissolved a solution of sodium hydroxide solution to neutral reaction in case of litmus paper, add 3 mL Copper sulfate solution, that is slowly precipitated pale blue precipitate.

A.3.2.3 UV spectrophotometry A.3.2.3.1 Method summary Nicotinic acid and nicotinamide aqueous solution by spectrophotometry, absorption maximum at 262 nm wavelength are, nicotinic acid in 237 nm Wavelength of minimum absorption; nicotinamide at 245 nm wavelength of minimum absorption, the absorption of niacin ratio A_{237nm}/A_{262nm} 0.35 to 0.39;

Nicotinamide absorbance ratio A_{245nm}/A_{262nm} 0.63 to 0.67. Therefore, this method can be used to distinguish between niacin and niacinamide.

A.3.2.3.2 analysis step Take laboratory sample amount, add water produced per 1 mL of 20 μ g of the solution, according to spectrophotometric method (Chinese Pharmacopoeia 2005 edition of two Appendix IV A) Determination of maximum absorption at a wavelength of 262 nm $\pm$ 1 nm, the minimum absorption at 237 nm $\pm$ 1 nm in wavelength; 237 nm $\pm$ 1 nm absorbance wavelength and absorbance ratio of 262 nm $\pm$ 1 nm wavelength should be 0.35 to 0.39.

A.3.2.4 infrared absorption spectroscopy Using potassium bromide tablet method according

to GB/T 6040 test, the laboratory sample infrared light absorption spectrum with a control of the map Induced (control profile see Appendix B).

A.4 Determination of niacin A.4.1 Method summary Phenolphthalein as an indicator, titration with aqueous sodium hydroxide standard solution titration sample, according to standard titration of sodium hydroxide solution consumed the amount was calculated content of C₆H₅NO₂ meter niacin. The results and titration with blank test correction.

A.4.2 Reagents and materials A.4.2.1 sodium hydroxide standard titration solution. c (NaOH) = 0.1 mol/L.

A.4.2.2 phenolphthalein indicator solution. 10 g/L ethanol solution.

A.4.3 Analysis step

Weigh about 0.3 g laboratory samples, accurate to 0.000 1 g, add 50 mL freshly boiled and cooled water dissolved, add 3 drops of phenol Phthalate indicator solution, titration with sodium hydroxide standard solution titration to pink.

A.4.4 Calculation Results Niacin (in C₆H₅NO₂ meter) mass fraction 1w, expressed in%, according to formula (A.1) Calculated.

() 100

(1) 1000

VV c Mw

- x x = xx - x% (A.1) where.

V - consumption of sodium hydroxide standard titration solution volume value in milliliters (mL);

OV - Numerical volume consumed sodium hydroxide standard titration blank test solution, in milliliters (mL);

c - sodium hydroxide standard titration solution actual concentration values, units of moles per liter (mol/L);

m - the quality of the laboratory sample value in units of grams (g);

2w --A.5 measured value loss on drying,%;

M - molar mass values of nicotinic acid in grams per mole (g/mol) [M (C₆H₅NO₂) = 123.1].

The absolute difference between two parallel determination of not more than 0.3%.

A.5 Determination of loss on drying A.5.1 Analysis step Weigh about 1.0 g laboratory samples, accurate to 0.000 1 g, has been placed at 105 °C ± 2 °C drying to constant weight flat weighing bottle, at 105 °C ± 2 °C drying 1h, into the cooled to room temperature in a desiccator and weighed.