

CCS X04



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

GB/T 22974-2008

**Determination of isometamidium residues in milk and milk
powder - LC-MS-MS method**

牛奶和奶粉中氮氨菲啶残留量的测定 液相色谱-串联质谱法

Issued on: December 31, 2008

Implemented on: May 1, 2009

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;
Standardization Administration of China**

Table of Contents

- 1 Scope
- 2 Normative references
- 3 PRINCIPLE OF THE METHOD
- 4 Reagents and materials

Foreword

The Standard Appendix A, Appendix B is an informative annex. This standard is proposed and managed by the State Administration of Quality Supervision Inspection and Quarantine of People's Republic of China. This standard is mainly drafted by: People's Republic of China Qinhuangdao Exit Inspection and Quarantine, Inner Mongolia, People's Republic of China Entry-Exit Inspection and Quarantine bureau, Inner Mongolia Mengniu Dairy (Group) Co., Ltd. The main drafters of this standard. Pan Guoqing, ZHANG Chun-yan, Bai Guotao, to Liu Jun, Zheng Book Fair, Li, Zhang Wei, Chang Jianjun, Li, Pang Guofang. Milk and milk powder Determination of ammonia nitrogen phenanthridine Residues Liquid chromatography - tandem mass spectrometry

1 Scope

GB/T 22974-2008 is the Chinese national standard on determination of isometamidium residues in milk and milk powder - lc-ms-ms method. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 31 December 2008 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 May 2009. Classification: ICS (ICS) , CCS X04. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This standard specifies the milk and milk powder phenanthridine ammonia nitrogen residues in liquid chromatography - tandem mass spectrometry. This standard applies to phenanthridine residues in milk and milk powder - Determination of ammonia nitrogen. The standard detection limit. milk 0.010mg/kg, milk was 0.080mg/kg.

2 Normative references

The following documents contain provisions which, through reference in this standard and become the standard terms. For dated references, subsequent Amendments (not including errata content) or revisions do not apply to this standard, however, encourage the parties to the agreement are based on research Whether the latest versions of these documents. For undated reference documents, the latest versions apply to this standard.

GB/T 6379.1 measurement methods and results of Accuracy (trueness and precision) - Part 1. General principles and definitions (GB/T 6379.1- 2004, ISO 5725-1.1994, IDT)

GB/T 6379.2 measurement methods and results of Accuracy (trueness and precision) - Part 2. Determine the standard measurement method to repeat Of the basic methods of reproduction (GB/T 6379.2-2004, ISO 5725-2.1994, IDT)

GB/T 6682 analytical laboratory use specifications and test methods (GB/T 6682-2008, ISO 3696. 1987, MOD)

3 PRINCIPLE OF THE METHOD

Ammonia nitrogen in the sample phenanthridine with acetonitrile and methanol mixed solution of ammonium formate extract, the extract was concentrated and defatted by liquid chromatography - tandem mass Spectrometer, external standard, selected ion qualitative.

4 Reagents and materials

Unless otherwise indicated, the reagents used were of analytical grade water as a water GB/T 6682 regulations.

4.1 Methanol. chromatographically pure.

4.2 formic acid. Chromatographic pure.

4.3 acetonitrile. chromatography.

4.4 hexane. chromatographically pure.

4.5 ammonium formate. 4.6 0.25mol/L ammonium formate in methanol solution. Weigh 15.76g ammonium formate (4.5) was dissolved in 1000mL methanol (4.1) in. 80% aqueous methanol 4.7. Measure 80mL methanol (4.1) was added 20mL of water.

4.8 saturated with 80% aqueous methanol solution of hexane. n-hexane was added 300mL (4.4) in a separating funnel, the appropriate amount of 80% aqueous methanol Solution (4.7) oscillation, standing layer, discarding the lower aqueous methanol, n-hexane solution top alternate. Extract 4.9. amount of acetonitrile (4.3) and/L ammonium formate in methanol 0.25mol (4.6) in accordance with 1.1 (volume ratio) were mixed.

4.10 ammonia nitrogen phenanthridine hydrochloride standard. purity of greater than or equal to 97%.

4.11 ammonia nitrogen phenanthridine standard stock solution. accurately weighed amount of ammonia nitrogen standard phenanthridine hydrochloride with methanol (4.1) dubbed the concentration of 100 μ g/mL of the standard stock solution. This solution is 0 °C ~ 4 °C refrigerator stored.

4.12 ammonia nitrogen phenanthridine intermediate standard solution. Imbibe 5mL of ammonia nitrogen phenanthridine standard stock solution (4.11) in 50mL brown volumetric flask, With methanol (4.1) to the mark, formulated at a concentration of 10 μ g/mL of ammonia nitrogen phenanthridine intermediate standard solution. The solution at 0 °C ~ 4 °C refrigerator