

CCS X04



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

GB/T 22991-2008

**Determination of virginiamycin residue in bovine milk and milk
powder - LC-MS-MS method**

牛奶和奶粉中维吉尼霉素残留量的测定 液相色谱-串联质谱法

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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 was drafted. People's Republic of China Qinhuangdao Exit Inspection and Quarantine, People's Republic of China Liaoning Entry-Exit Inspection and Quarantine Bureau. The main drafters of this standard. Song Wenbin, Li Yichen, Dongzhen Lin, Mike Sui, Clue red, Xue Dafang, a Zhang, LI Zhen-Rong, Pang Guofang. Determination of virginiamycin residues in milk and milk powder Liquid chromatography - tandem mass spectrometry

1 Scope

GB/T 22991-2008 is the Chinese national standard on determination of virginiamycin residue in bovine 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 pure raw milk and milk powder virginiamycin M1 (virginiamycinM1) Determination of the amount of residual liquid chromatography - tandem Mass spectrometry. This standard applies to the determination of virginiamycin M1 residue in raw milk and pure milk powder. The detection limit of this standard. raw milk virginiamycin M1 is 0.25µg/L, pure milk powder virginiamycin M1 is 2.0µg/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) Principle

3 Sample remaining virginiamycin M1 + acetonitrile were mixed in methanol extract vortex extraction, centrifugation, and the supernatant with ammonium dihydrogen phosphate Buffer solution was diluted to C18 solid phase extraction column purification. The eluent of virginiamycin M1 separated and extracted with chloroform to remove Aqueous layer. Nitrogen blowing chloroform to dryness, treated with ammonium formate buffer solution and the mixture was dissolved in methanol and set the volume for liquid chromatography - tandem mass spectrometer.

4 Reagents and materials

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

4.1 acetic acid.

4.2 formic acid. Chromatographic pure.

4.3 Methanol. chromatographically pure.

4.4 acetonitrile. chromatography.

4.5 chloroform. chromatography.

4.6 ammonium dihydrogen phosphate. pure class distinctions.

4.7 Ammonium. pure class distinctions.

4.8 ammonium dihydrogen phosphate solution. 0.1mol/L, weighed 11.50g ammonium dihydrogen phosphate (4.6), dissolved in water and dilute to 1000mL.

4.9 ammonium dihydrogen phosphate solution. 0.01mol/L, amount of 0.1mol/L ammonium dihydrogen phosphate solution (4.8) 100mL, diluted with water to the 1000mL.

4.10 ammonium formate buffer solution. 0.12mol/L, pH = 3.2, weighed 7.56g ammonium

formate (4.7), dissolved in water, adjusted with formic acid (4.2) pH to 3.2, the volume to 1000mL.

4.11 ammonium formate buffer. 0.003mol/L, pH = 3.2, amount of 25.0mL ammonium formate buffer (4.10), diluted with water to the

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