

CCS X04



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

GB/T 22986-2008

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

牛奶和奶粉中氢化泼尼松残留量的测定 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, 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, Baiguo Tao, ZHANG Chun-yan, Liu Lai Jun, Zheng Book Fair, Li, Zhang, Li, Chang army, Pang Guofang. Determination of milk and milk powder prednisolone Residues Liquid chromatography - tandem mass spectrometry

1 Scope

GB/T 22986-2008 is the Chinese national standard on determination of prednisolone 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 prednisolone residues liquid chromatography - tandem mass spectrometry. This standard applies to the determination of milk and milk powder prednisolone residues. The standard detection limit. milk 0.5µg/kg, milk was 4.0µg/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)

3 PRINCIPLE OF THE METHOD

Sample of prednisolone with methanol - acetic acid buffer solution extracted with n-hexane in addition to fat, solid-phase extraction (SPE), 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 hexane. chromatographically pure.

4.4 glacial acetic acid.

4.5 sodium acetate trihydrate. 4.6 0.2mol/L acetic acid solution. lessons 5.72mL glacial acetic acid (4.4) to 500mL volumetric flask with water to volume. 4.7 0.2mol/L sodium acetate solution. Weigh 27.22g sodium acetate trihydrate (4.5), transfer dissolved in water to 1000mL volumetric flask, Water to volume.

4.8 acetic acid - sodium acetate buffer solution (pH = 5.2). Measure 21mL of acetic acid solution (4.6) and the 79mL solution of sodium acetate (4.7) were mixed, Adjusting the solution pH = 5.2. 4.9 30% aqueous solution of methanol. Measure 30mL of methanol (4.1), diluted with water volume to 100mL.

4.10 methanol saturated with n-hexane. amount of methanol (4.1) 10mL placed in a separatory funnel, add n-hexane (4.3) 90mL, and mixed by shaking 1min, standing

stratification, the upper liquid reserve.

4.11 prednisolone standards. purity of greater than 99%.

4.12 prednisolone standard stock solution. Weigh accurately prednisolone standard, dubbed 0.10mg/mL standard with methanol (4.1)

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