

CCS X04



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

**GB/T 22971-2008**

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

牛奶和奶粉中安乃近代谢物残留量的测定 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 is drafted by: People's Republic of China Qinhuangdao Exit Inspection and Quarantine, Shenzhen People's Republic of China Entry-Exit Inspection Quarantine Bureau. The main drafters of this standard. Chen Jincan, Xieli Qi, Xiao Chengui, Zhu Yulan, Bear Babe, Lin Yankui, Pang Guofang. Milk and milk powder dipyrone metabolite Determination of residues of liquid chromatography - tandem mass spectrometry

### 1 Scope

GB/T 22971-2008 is the Chinese national standard on determination of metabolite residues of dipyrone 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 dipyrone metabolite 4-acylamino methamphetamine antipyrine (4-formylaminoantipyrine), 4- acetyl Amino antipyrine (4-acetylaminoantipyrine), 4- methyl-amino-antipyrine (4-methylaminoantipyrine) and 4-aminoantipyrine Antipyrine (4-aminoantipyrine) liquid chromatography - tandem mass spectrometry. This standard applies to milk and milk powder 4-acylamino methamphetamine antipyrine, 4-acetyl amino antipyrine, 4-methyl-amino-antipyrine and 4-aminoantipyrine determination than forest residues. The standard method detection limit is. For milk, 4 formamide methamphetamine antipyrine, 4-amino antipyrine,

4-methyl-amino-antipyrine And 4-acetyl-amino-antipyrine were 5.0µg/kg; for milk powder, 4 formamide methamphetamine antipyrine, 4-amino antipyrine, 4-methyl amino Antipyrine and 4-acetyl-amino antipyrine were 40.0µg/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 After milk and milk powder dipyrone metabolite residues were extracted with acetonitrile solution was added to Tris, defatted with hexane extract, liquid chromatography Spectrum - tandem mass spectrometer. 4-acetyl-amino-antipyrine, 4 formamide methamphetamine antipyrine and 4-amino-antipyrine using external standard, 4-methyl-amino-antipyrine quantified using internal standard method.

## 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 isopropanol.

4.3 acetonitrile. chromatography.

4.4 Tris (Tris). Ammonium acetate 4.5. HPLC grade. 4.6 1.0mol/LTris solution. Weigh accurately 12.1g Tris, water volume to 100mL.

4.7 sample volume solution. methanol - water (1 + 19).