



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

GB/T 22954-2008

**Determination of streptomycin, dihydrostreptomycin and
kanamycin residues in fugu and eel - LC-MS-MS method**

河豚鱼和鳗鱼中链霉素、双氢链霉素和卡那霉素残留量的测定 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. This standard was drafted. People's Republic of China Qinhuangdao Exit Inspection and Quarantine. The main drafters of this standard. FEDERATION Zhangshou Jun, Zhang Jinjie, Caoyan Zhong, Yang Zhiwei, Cao Yaping, Pang Guofang. Puffer fish and eels in streptomycin, dihydrostreptomycin And kanamycin residues Determination Liquid chromatography - tandem mass spectrometry

1 Scope

GB/T 22954-2008 is the Chinese national standard on determination of streptomycin, dihydrostreptomycin and kanamycin residues in fugu and eel - 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 puffer fish and eel in streptomycin, dihydrostreptomycin and kanamycin residues in liquid chromatography - tandem mass spectrometry method. This standard applies to streptomycin, dihydrostreptomycin and determination of kanamycin residues in eels and puffer fish. The detection limit of the standard methods. streptomycin, dihydrostreptomycin and kanamycin detection limits were 10.0µg/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 Puffer fish or eel sample antibiotic use phosphoric acid extraction, TCA protein precipitation with carboxylic acid type and solid phase extraction Column purification. Liquid chromatography - tandem mass spectrometry (ESI +) detection, external standard.

4 Reagents and materials

Unless otherwise indicated, the reagents are pure class distinctions.

4.1 Water. GB/T 6682, level.

4.2 Methanol. chromatographically pure.

4.3 acetonitrile. chromatography.

4.4 formic acid.

4.5 concentrated phosphoric acid.

4.6 dipotassium hydrogen phosphate (K_2HPO_4).

4.7 TCA ($C_2HCl_3O_2$).

4.8 heptane sulfonate ($C_7H_{15}NaO_3S \cdot H_2O$). 4.9 5% phosphoric acid solution (1 + 19).

Take 50mL concentrated phosphoric acid (4.5), water volume to 1L. 4.10 0.2mol/L phosphate buffer solution. pH = 8.5. He said dissolved in water taken 34.8g potassium phosphate dibasic (4.6), set the volume to 1L, with Sodium hydroxide solution to adjust pH = 8.5.

4.11 trichloroacetic acid solution. 50%, mass fraction. Take 20g trichloroacetic acid (4.7), water volume to 20mL. 4.12 0.01mol/L sodium heptane solution. Weigh 2.20g of heptane sulfonate (4.8), dissolved in water, dilute to 1L.