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

GB 29685-2013

**Determination of Lincomycin, Clindamycin and Spectinomycin
residues in animal derived food by Gas Chromatography Mass
Spectrometric method**

食品安全国家标准 动物性食品中林可霉素、克林霉素和大观霉素多残留的测定
气相色谱—质谱法

(English Translation)

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1 Scope

This standard specifies the animal food lincomycin, clindamycin and spectinomycin Residues sample preparation and gas chromatography - mass spectroscopy Given method.

This standard applies to pigs, cattle and chicken muscles and kidneys as well as milk and eggs lincomycin, clindamycin and spectinomycin single or multiple Detecting a residual amount of the drug.

2 Normative references

The following documents for the application of this document is essential. For dated references, only applies to the version dated paper Pieces. For undated references, the latest edition (including any amendments) applies to this document.

GB/T 6682 Water for analytical laboratory specifications and test methods Principle 3 the specimens remaining lincomycin, clindamycin and spectinomycin, extracted with phosphate buffer, 3% trichloroacetic acid precipitated protein, ion pairs Reversed-phase SPE column purification principle, N, O- bis (trimethylsilyl) trifluoroacetamide derivative, gas chromatography - mass spectrometry, external standard.

4 Reagents and materials

The following reagents used, unless otherwise stated are analytical reagents, water as a water line with GB/T 6682 provisions.

Lincomycin hydrochloride 4.1 Standard. content $\geq 99\%$; clindamycin hydrochloride and spectinomycin hydrochloride standard. content $\geq 95\%$.

4.2

N, O- bis (trimethylsilyl) trifluoroacetamide.

Acetate 4.3. excellent pure.

4.4

Methanol. HPLC grade.

4.5

Acetonitrile. chromatographically pure.

4.6

hexane. chromatography.

4.7

potassium dihydrogen phosphate. pure class distinctions.

4.8

phosphoric acid. pure class.

4.9

Potassium hydroxide. pure class.

4.10

TCA. pure class distinctions.

4.11

sodium dodecyl sulfate. HPLC grade.

4.12

HLB solid phase extraction column. 60mg/3mL, or equivalent person.

The extract was 4.13. Take 1.36 g of potassium dihydrogen phosphate, dissolved and diluted to 1000mL with water, adjusted to pH 4.0 with phosphoric acid.

4.14 3% trichloroacetic acid solution. Take trichloroacetic 3.0g, dissolve and dilute to 100mL with water.

4.15 2mol/L potassium hydroxide solution. Take potassium hydroxide 11.6g, dissolve and

dilute to 100mL with water.

4.16

sodium dodecyl sulfate solution. Take sodium dodecyl sulfate 2.78g, dissolved and diluted to 50mL with water.

4.17

sodium dodecyl sulphate buffer. Take 2.78 g sodium dodecyl sulfate, dissolved in water, adding acetic acid 1mL, diluted with water 500mL.

4.18 1mg/mL lincomycin, clindamycin and spectinomycin standard stock solution. Weigh accurately lincomycin, clindamycin and spectinomycin each standard 10mg, respectively 10mL volumetric flask, dissolved in methanol and diluted to the mark, formulated at a concentration of 1mg/mL of Lincomycin Su, clindamycin and spectinomycin standard stock solution. 2 °C ~ 8 °C from light, valid for 6 months.

4.19 100µg/mL standard working solution. precise amount of 1mg/mL lincomycin, clindamycin and spectinomycin standard stock solution of each 1.0 mL, respectively 10mL volumetric flask, dilute to the mark with methanol, formulated at a concentration of 100µg/mL lincomycin, clindamycin, and Spectinomycin standard working solution. 2 °C ~ 8 °C from light, one week period.

5 Apparatus

5.1

Gas chromatography - mass spectrometry. with the EI source.

5.2

Analytical balance. a sense of volume 0.00001g.

5.3

Balance. a sense of the amount of 0.01g.

5.4

freezing high-speed centrifuge.

5.5

electric heated water bath.

5.6

vortex mixer.

5.7

homogenizer.

5.8

PTFE tube. 50mL.

Preparation and Storage of sample 6

6.1.1 Pork, beef and chicken muscle, liver and kidney tissue

Fresh or thawed take appropriate blank or test tissue, minced, and homogenized.

--- the test sample taken after homogenization, as the feed try.

--- blank sample taken after homogenization, as a blank sample.

--- blank sample taken after homogenization, adding a suitable concentration of the standard working solution, is added as a blank sample.

6.1.2 Milk and eggs

An appropriate amount of fresh or frozen test blank or milk and eggs, mixed, and homogenized.

--- the test sample taken after homogenization, as the feed try.

--- blank sample taken after homogenization, as a blank sample.

--- blank sample taken after homogenization, adding a suitable concentration of the standard working solution, is added as a blank sample.

Save 6.2 sample

The following tissue samples stored at -20 °C. Eggs and milk sample or less 2 °C ~ 8 °C stored.

Determination Step 7

7.1 Preparation of matrix-matched standard curve

Were the precise amount of 100µg/mL lincomycin, clindamycin and spectinomycin appropriate amount of working standard solution, diluted with methanol and formulated into concentrated 50,100,200,400,800,1600 degrees and 3200µg/L of a mixed series of standard solution from each 1.0 mL, extracted separately dissolved, Purifying and drying the residue of the sample blank, thoroughly mixed, dried at room temperature, was added N, O- bis (trimethylsilyl) trifluoroacetamide and 500µL

100 L acetonitrile, vortex mixed for 1min, sealed at 75 deg.] C incubator IH derivatization reaction, dry nitrogen at room temperature, was added n-hexane solution 0.5mL the residue solution was vortexed, gas chromatography - mass spectrometry. To measure peak area for the vertical axis, corresponding to the concentration of standard solution as abscissa, the standard curve. Seeking regression equation and correlation coefficient.