

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

SN/T 5113-2019

**Determination of furan in foodstuffs and feeds for import and
export, liquid chromatography-mass spectrometry/mass
spectrometry and liquid chromatography**

进出口食用动物、饲料中呋喃测定 液相色谱-质谱/质谱法和液相色谱法

(English Translation)

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Foreword

This standard was drafted in accordance with the rules required by GB/T 1.1-2009.

This standard is proposed and managed by the General Administration of Customs of the People's Republic of China.

This standard was drafted. Tianjin Customs.

1 Scope

This standard specifies liquid chromatography-mass spectrometry/mass spectrometry method for determination of nitrofurantol metabolites in serum and urine of food animals for import and export.

Determination of furan in liquid by liquid chromatography.

This standard is applicable to the determination of nitrofurantol metabolites in serum (bovine, pig, chicken) and urine (bovine, pig) of imported and exported animals.

Feed, pre-mixed feed, concentrated feed).

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies to this article Pieces. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods (neq ISO 3696:1987, MOD)

3 Method principle

The pretreatment of nitrofurantoin metabolites in blood and urine samples is hydrolyzed under acidic conditions.

It was derivatized overnight, extracted with ethyl acetate solution, and then determined by liquid chromatography-mass spectrometry/mass spectrometry, and quantified by internal standard method.

Furan in feed samples was extracted with acetonitrile as the pretreatment. After concentration and volume, it was determined by liquid chromatography and quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, the water used is first-grade water specified in GB/T 6682.

4.1

Methanol, acetonitrile, ethyl acetate, n-hexane, formic acid. chromatographically pure.

4.2 2-Nitrobenzaldehyde, dipotassium hydrogen phosphate trihydrate. analytical grade.

4.3

Ammonium acetate. chromatographically pure.

4.4

Dimethyl sulfoxide. analytical grade.

4.5

Standard substances. Furancillin original drug (CAS code 59-87-0, molecular weight 198.4, molecular formula $C_6H_6N_4O_4$), furazolidone original drug (CAS code 67-45-8, molecular weight 225.16, molecular formula $C_8H_7O_5N_3$), furanone original drug (CAS code 139-91-3, molecular weight 324.33, molecular formula $C_{13}H_{16}N_4O_6$), furantoin original drug (CAS code 67-20-9, molecular weight 238.16, molecular formula $C_8H_6N_4O_5$) standard Quasi-substance, purity $\geq 98\%$.

4.6

Reference materials. Furacin metabolites (CAS code 57-56-7, molecular weight 75.07, molecular formula CH_5N_3O), furazolidone metabolites (CAS code 80-65-9, molecular weight 102.09, molecular formula $C_3H_6N_2O_2$), furanone metabolites (CAS code 43056-63-9, molecule the amount is.201.22, molecular formula $C_8H_{15}N_3O_3$), furantoin metabolites (CAS code 2827-56-7, molecular weight 151.55, molecular formula

C₃H₆CIN₃O₂) standard material, purity ≥98%.

4.7

Preparation of 1mol/L hydrochloric acid solution. Transfer 16.9mL of concentrated hydrochloric acid and make up to 200mL with water.

4.8 0.1mol/LK₂HPO₄ solution preparation. Weigh K₂HPO₄ · 3H₂O 11.4g, dissolve it in water, and make it to 500mL.

4.9

Preparation of Namol solution at 5mol/L. Weigh 100g of NaOH, dissolve it in water, and make up to 500mL.

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4.10

Preparation of derivatization reagent. 37.8 mg of 2-nitrobenzaldehyde was dissolved in 5 ml of dimethyl sulfoxide.

4.11 10% acetonitrile-water solution. Measure 10mL of acetonitrile and 90mL of water and mix.

4.12 2% formic acid. Measure 2mL of formic acid and 98mL of water and mix.

4.13

Methanol-0.5 mmol/L ammonium acetate solution (20.80, V/V). Weigh 38.5 mg of ammonium acetate, dilute with water and make up to volume.

800mL, then 200mL of methanol was added and mixed.

4.14. 5% ammonium acetate solution; weigh 500mg of ammonium acetate, dilute with water and make up to 1L.

4.15

Standard furan stock solutions. 0.1 mg/mL. Accurately weigh the appropriate amount of standard material (4.5), and prepare each with acetonitrile A standard stock solution of 0.1 mg/mL was stored at -18 ° C, protected from light.

4.16

Standard furan metabolite stock solution. 0.1 mg/mL. Accurately weigh an appropriate amount of standard material (4.6), and prepare each with methanol A standard stock solution of 0.1 mg/mL was stored at -18 ° C, protected from light.

4.17

Furan original standard working solution. Pipette the standard stock solution (4.15) as needed, and dilute to volume with acetonitrile.

4.18

Furan metabolite standard working solution. Pipette the standard stock solution (4.16) as needed, and dilute to volume with methanol.

4.19

Filter. 0.22 μm , organic phase filter.

5 Instrument

5.1

Liquid chromatography-tandem mass spectrometer. equipped with an electrospray ion source.

5.2

Liquid chromatograph. equipped with UV detector.

5.3

Analytical balance. Sensitivity. 0.01g, 0.0001g.

5.4

Ultrasonic cleaner.

5.5

Solid-phase extraction device.

5.6

Nitrogen Concentrator.

5.7

Centrifuge. The speed is 4,000r/min or more.

5.8

Water bath constant temperature oscillator.

6.1 Preparation of sample

For blood and urine samples, take the supernatant after centrifugation, mix well, and aliquot. For feed samples, take a representative sample of 1kg, quartered Method to reduce, take about 200g, pulverize, pass through a 0.45mm hole sieve, mix and distribute into a grind bottle. During sample preparation, prevent samples from being affected.

To pollution or changes in the content of the target.