

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

SN/T 5118-2019

**Determination of melamine residues in foodstuffs and feeds for
import and export by liquid chromatography-mass
spectrometry/mass spectrometry**

进出口食用动物、饲料中三聚氰胺残留测定 液相色谱-质谱/质谱法

(English Translation)

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Table of Contents

Foreword	
1 Scope	
2 Normative references	
3 Method summary	
4 Reagent materials	
4.4 Ammonium acetate	
5 Instruments and equipment	
6 Sample preparation and storage	
6.1 Sample collection and preparation	
6.2 Sample preservation	
7 Analysis steps	
7.1 Extraction	
7.2 Purification	
7.3 Measurement and confirmation	
7.4 Blank test	
8 Results calculation and expression	
9.1 Low limit of determination (LOQ)	
9.2 Recovery rate	
10 Precision	

Foreword

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

Please note that some content of this document may involve patents, and the issuing authority of this document is not responsible for identifying these patents.

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

This standard was drafted. Chongqing Customs of the People's Republic of China, Nanjing Customs of the People's Republic of China.

1 Scope

This standard specifies the liquid chromatography-mass spectrometry/mass spectrometry method for melamine residues in imported and exported food animals and feed.

This standard applies to the determination of melamine in the blood, urine and animal feed of imported and exported food animals (such as pigs, cattle, sheep, rabbits, chickens, ducks).

And confirmation.

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies to this document.

file. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods GB/T 14699.1 Feed sampling GB/T.20195 Preparation of animal feed samples

3 Method summary

After the sample was ultrasonically extracted with an acidic methanol aqueous solution, the sample was purified by a mixed cationic solid-phase extraction column, and detected by liquid chromatography-mass spectrometry/mass spectrometry.

Confirmed and quantified by internal standard method.

4 Reagent materials

Unless otherwise specified, the reagents used are of analytical grade, and the water is first-grade water specified in GB/T 6682.

4.1

Methanol. HPLC grade.

4.2

Acetonitrile. HPLC grade.

4.3

Acetic acid. HPLC grade.

4.4 Ammonium acetate

4.5 50% methanol aqueous solution (5 + 5, V/V). Measure 500mL of methanol and 500mL of water and mix.

4.6 95% acetonitrile aqueous solution. Take 50mL of water and 950mL of acetonitrile and mix well.

4.7 5% acetic acid solution. Take 5mL of acetic acid and dissolve in 95mL of water.

4.8 5% ammonia in methanol. Take 5mL of ammonia to dissolve in 95mL of methanol.

4.9 Acidic methanol extraction solution. Add 90 mL of acetic acid to 90 mL of 50% methanol aqueous solution (4.5) and mix.

4.10 5mol/L ammonium acetate solution. Weigh 0.385g of ammonium acetate and dissolve it in 1,000mL of water.

4.11 Reference materials. Melamine (CAS No .. 108-78-1), Melamine internal standard (15N3-Melamine, CAS) no. 2877476-11-3), the purity is greater than 98%.

4.12 Standard stock solution. Accurately weigh 0.1g of melamine standard (accurate to 0.0001g), and fully dissolve it in 50% methanol aqueous solution (4.5).

Dissolve, dilute to a 100mL volumetric flask, prepare a standard stock solution with a concentration of 1,000mg/mL, and dissolve the melamine in the isotope.

Standard 1mg, fully dissolved with 50% methanol aqueous solution (4.5), diluted and fixed in a 10mL volumetric flask, formulated into a concentration of SN/T 5181-2019 ?????, ?????, ?????, ??

For 0.1100 mg/mL standard stock solution, pour the volume of the standard stock solution into brown reagent bottles, and store them in the dark below -18 ° C.

4.13 Standard intermediate stock solution. Accurately transfer 100 µL of melamine standard stock solution (4.12) and 100 µL of melamine internal standard stock solution (4.12) in two 100 mL volumetric flasks, dilute to volume with acetonitrile, and separately prepare Melamine standard solution of 1,000 µg/mL and 0.1100 µg/mL melamine isotope internal standard solution were prepared. The solution was placed in a brown reagent bottle and stored under -18 ° C in the dark. 4.14 Standard working solution. Dilute the standard intermediate stock solution (4.13) to a suitable concentration with a 95% acetonitrile aqueous solution (4.6) as required.

Standard working curve solution, the reference linear concentration range is 5ng/mL ~ 100ng/mL, the internal standard concentration of melamine isotope is 2ng/mL Ready for immediate use.

4.15 MCX solid phase extraction column (60mg, 3mL) 1) or equivalent. 3mL methanol, 3mL

water and 3mL 5% acetic acid in order before use Solution (4.7) was activated.

4.16 Microporous membrane. organic, 0.22 μm .

5 Instruments and equipment

5.1

Liquid chromatography-mass spectrometry/mass spectrometer. Power distribution spray ion source (ESI).

5.2

Balance. Sensitivity is 0.0001g and 0.01mg.

5.3

Centrifuge. The speed should not be lower than 8000r/min.

5.4

Vortex mixer.

5.5

Ultrasonic cleaner.

5.6

Solid-phase extraction device.

5.7

Nitrogen blowing instrument.

6.1 Sample collection and preparation

6.1.1

Feed. Collect representative samples in accordance with GB/T 14699.1 and prepare according to GB/T 2095.

6.1.2

Blood. Animal blood is collected with heparin sodium anticoagulant blood collection tubes. The collected amount shall not be less than 10mL, sealed, and marked.

6.1.3

Urine. Collect it with a clean centrifuge tube. The collected amount should not be less than 10mL. After stirring well, seal it and mark it.