

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

**SN/T 2226-2008**

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**Determination of urotropine residue in foodstuffs of animal  
origin for import and export. LC-MS/MS method**

进出口动物源性食品中乌洛托品残留量的检测方法 液相色谱-质谱/质谱法

**(English Translation)**

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### Foreword

The Standard Appendix A and Appendix B are informative.

This standard is proposed and managed by the National Certification and Accreditation Administration Committee.

This standard was drafted. Exit Inspection and Quarantine of Heilongjiang People's Republic of China, Chongqing, People's Republic of China Entry-Exit Inspection and Quarantine Bureau, People's Republic of China Liaoning Province Exit Inspection and Quarantine.

### 1 Scope

This standard specifies the import and export of food of animal origin methenamine residues LC - MS/MS detection method.

This standard applies to the import and export of chicken, liver, chicken and pork kidney methenamine residues detection and confirmation.

### 2 Normative references

The following documents contain provisions which, through reference in this standard and become the standard terms. For dated references, subsequently Some amendments (not including errata content) or revisions do not apply to this standard, however, encourage the various aspects of the agreement in accordance with this standard Research using the latest versions of these documents. For undated reference documents, the latest versions apply to this standard.

GB/T 6682 analytical laboratory use specifications and test methods (GB/T 6682-2008, ISO 3696. 1987, MOD)

### 3 Method summary

Extracting a sample of methenamine residue with acetonitrile and n-hexane to remove fat-soluble impurities, cation exchange solid phase extraction (SPE), the eluent After concentrating the residue with a basic set of acetonitrile solution, liquid chromatography - mass spectrometry/mass spectrometer, external standard.

### 4 Reagents and materials

All reagents unless otherwise specified, the reagents were of analytical grade water as a water GB/T 6682 regulations.

#### 4.1

acetonitrile. chromatography.

#### 4.2

hexane. chromatographically pure.

#### 4.3

Methanol. chromatographically pure.

#### 4.4

acetic acid (99%).

#### 4.5

Ammonia (25%).

#### 4.6

hydrochloric acid (36%).

#### 4.7

ammonium acetate.

4.8 0.1% acetic acid solution. amount of 0.5mL of acetic acid (4.4), diluted with water to 500mL.

4.9 0.1mol/L hydrochloric acid solution. amount of 4.2mL of hydrochloric acid (4.6), dilute with water to 500mL.

4.10 20mmol/L ammonium acetate buffer (pH = 4). Weigh accurately 0.77g of ammonium acetate were dissolved in 500mL water and neutralized with acetic acid solution (4.8) adjusted to pH 4.

4.11 5% ammonia - methanol (95 + 5, volume ratio) was. 5mL amount of aqueous ammonia (4.5), diluted to 100mL with methanol.

4.12 0.1% acetic acid - acetonitrile (8 + 2, volume ratio) solution. Measure 200mL acetic acid solution (4.8), diluted with acetonitrile to 1000mL.

**4.13**

acetonitrile saturated n-hexane solution. 20mL of acetonitrile was added 100mL of n-hexane, followed by sufficient shaking, standing layer, the upper layer n-hexyl Chloride layer alternate.

**4.14**

Anhydrous sodium sulfate. After burning 650 °C 4h, cooled storage backup in a desiccator.

**4.15**

methenamine standard substance (Urotropine, molecular formula.  $C_6H_{12}N_4$ ). purity of greater than 99.0%.

**4.16**

standard stock solution. Weigh accurately translated its purity was 100% by mass of hexamine standards 0.010g, dissolved in acetonitrile and Constant volume to 100mL, concentration equivalent to 100 $\mu$ g/mL. Stock solution at 0 °C ~ 4 °C refrigerator protected from light, can be used for six months.

**4.17**

intermediate standard solution. take the exact amount of standard stock solution (4.16) 1.0mL, acetic acid - is dissolved in acetonitrile (4.12) and set the volume to