

# SN

## 中华人民共和国出入境检验检疫行业标准

SN/T 1769—2006

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### 进出口肉及肉制品中甲氧苄氨嘧啶 残留量测定方法 液相色谱法

Determination of trimethoprim residues in  
meat and meat products for import and export—  
Liquid chromatographic method

中华人民共和国出入境检验检疫  
行业标准  
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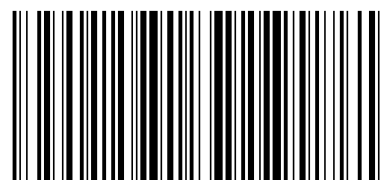
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## 4 Limit of determination and recovery

### 4.1 Limit of determination

The limit of determination of this method is 0.04 mg/kg.

### 4.2 Recovery

**4.2.1** According to the experimental data, the fortifying concentrations of trimethoprim in chicken meat and its corresponding recoveries are:

- 0.50 mg/kg, the recovery is 94.6% ;
- 0.20 mg/kg, the recovery is 85.5% ;
- 0.04 mg/kg, the recovery is 81.2% .

**4.2.2** According to the experimental data, the fortifying concentrations of trimethoprim in pork and its corresponding recoveries are:

- 0.50 mg/kg, the recovery is 92.6% ;
- 0.20 mg/kg, the recovery is 87.2% ;
- 0.04 mg/kg, the recovery is 82.5% .

## 前 言

本标准的附录 A 为资料性附录。

本标准由国家认证认可监督管理委员会提出并归口。

本标准由中华人民共和国黑龙江出入境检验检疫局负责起草。

本标准主要起草人:杨长志、康庆贺、马东升、张洪祥、张兰凤。

本标准系首次发布的出入境检验检疫行业标准。

**3.2.8** Potassium hydrate solution; 2 mol/L. Dissolve 112.2 g of GR potassium hydrate in 1 000 mL water.

**3.2.9** Trimethoprim standard; Purity $\geq$ 99%

**3.2.10** Trimethoprim standard solution: Accurately weigh an adequate amount of trimethoprim standard, dissolve in methanol and prepare a solution of 100  $\mu$ g/mL as the standard stock solution. According to the requirement, dilute a standard working solution of appropriate concentration with mobile phase.

### 3.3 Apparatus and equipment

**3.3.1** High performance liquid chromatography equipped with UV-detector.

**3.3.2** High speed blender.

**3.3.3** Centrifuge.

**3.3.4** Vortex mixer.

**3.3.5** Rotary evaporator.

**3.3.6** Nitrogen evaporator.

**3.3.7** Separator funnel; 250 mL.

**3.3.8** Centrifuge tubes with ground stopper; 15 mL, 50 mL.

**3.3.9** Column of anhydrous sodium sulfate; 8.0 cm $\times$ 1.5 cm(i. d), packed with 5 cm height of anhydrous sodium sulfate.

**3.3.10** Membrane filter; 0.45  $\mu$ m.

### 3.4 Procedure

#### 3.4.1 Extraction

Weigh ca 5 g (accurate to 0.1 g) of the test sample into a 50 mL centrifuge tube, add 15 mL of chloroform, and 20 mL of methanol-0.1 mol/L sulfuric acid solution (7+3) to the tube in sequence. Cap the tube and mix at high speed on vortex mixer for 2 min, then centrifuge at 3 000 r/min for 3 min. Transfer the supernatant into a 250 mL separator funnel. The remaining solution is extracted again with 20 mL of methanol - 0.1 mol/L sulfuric acid solution (7+3). Combine the supernatant into the same separator funnel. To the separator funnel, add 2 mL of 2 mol/L potassium hydrate solution and 2 $\times$ 80 mL of dichloromethane, then shake violently for 2 min and let stand to separate clearly. Pass the dichloromethane layer through an anhydrous sodium sulfate column to remove the water, let drain into a 250 mL evaporated flask. Evaporate the dehydrate dichloromethane to near dryness with rotary evaporator in a water-bath below 45 $^{\circ}$ C, then blow to dryness under a nitrogen flow. Dissolve the residues by adding accurately 2 mL of the mobile phase(3.4.3.1).

#### 3.4.2 Clean up

Transfer the above solution into 15 mL centrifuge tube, add 5 mL of chloroform and mix at high speed on vortex mixer for 2 min. Centrifuge at 3 000 r/min for 2 min. The clear supernatant is filtered through a 0.45  $\mu$ m membrane and ready for HPLC determination.

## 进出口肉及肉制品中甲氧苄氨嘧啶 残留量测定方法 液相色谱法

### 1 范围

本标准规定了进出口肉及肉制品中甲氧苄氨嘧啶残留量检验的抽样、制样和高效液相色谱测定方法。

本标准适用于进出口鸡肉和猪肉中甲氧苄氨嘧啶残留量的检验。

### 2 抽样和制样

#### 2.1 检验批

以不超过 2 500 件商品为一个检验批。

同一检验批的商品应具有相同的特征,如:包装、标记、产地、规格和等级等。

#### 2.2 抽样数量

抽样数量见表 1。

表 1

单位为件

批量	最低抽样数
1~25	1
26~100	5
101~250	10
251~500	15
501~1 000	17
1 001~2 500	20

#### 2.3 抽样方法

按 2.2 规定的抽样件数随机抽取,逐件开启。从每件中取一袋作为原始样品,其总量不得少于 2 kg。放入清洁容器内,加封后,标明标记,及时送实验室。

如每件中无小包装,或有小包装但质量超 2 kg 者,则可用灭菌刀在抽出的包件中,每件割取不少于 100 g。混合后置于清洁容器内,作为混合原始样。混合原始样的重量不少于 2 kg。加封后,标明标记,及时送交实验室。

#### 2.4 试样制备

从所取原始样品中缩分出 1 kg,经捣碎机充分捣碎均匀,均分成两份,分别装入洁净容器内作为原始样品。密封并标明标记。在抽样和制样的操作过程中,应防止样品受到污染或发生残留物含量的变化。

#### 2.5 试样保存

将试样于-18 $^{\circ}$ C 以下冷冻保存。

### 3 测定方法

#### 3.1 方法提要

试样中残留的甲氧苄氨嘧啶用三氯甲烷和酸性甲醇提取,提取液调至碱性后,用二氯甲烷萃取。