SN

# 中华人民共和国出入境检验检疫行业标准

SN/T 0684—2011
代替 SN 0684—1997

# 出口肉及肉制品中奥芬哒唑、芬苯哒唑、苯硫胍及奥芬哒唑砜残留量检测方法 液相色谱-质谱/质谱法

**Determiantion of oxfendazole, fenbendazole, febantel and oxfendazole sulphone residues in meat and meat products for export—HPLC-MS/MS method**

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中华人民共和国
国家质量监督检验检疫总局　发布

# 前　言

本标准按照 GB/T 1.1—2009 给出的规则起草。

本标准代替 SN 0684—1997《出口肉及肉制品中奥芬哒唑残留量检验方法》。与 SN 0684—1997 相比主要修改如下：

——对标准的名称进行了修改；

——增加了检测项目，除奥芬哒唑外，还包括了芬苯哒唑、苯硫胍及奥芬哒唑砜；

——检测方法由液相色谱法改为液相色谱-质谱/质谱法；

——修改了奥芬哒唑检验结果的表述，奥芬哒唑、芬苯哒唑、苯硫胍的检验结果均以可能被氧化成奥芬哒唑砜的可萃取残留总量计。

本标准由国家认证认可监督管理委员会提出并归口。

本标准起草单位：中华人民共和国上海出入境检验检疫局。

本标准主要起草人：郭德华、邓晓军、韩丽、盛永刚、伊雄海、赵善贞。

本标准于 1997 年首次发布，本次为第一次修订。

# 出口肉及肉制品中奥芬哒唑、芬苯哒唑、苯硫胍及奥芬哒唑砜残留量检测方法 液相色谱-质谱/质谱法

## 1 范围

本标准规定了出口肉及肉制品中奥芬哒唑、芬苯哒唑、苯硫胍和奥芬哒唑砜残留量的测定方法。

本标准适用于牛肉、猪肝、午餐肉中奥芬哒唑、芬苯哒唑、苯硫胍和奥芬哒唑砜残留量的检测。

## 2 规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件，仅注日期的版本适用于本文件。凡是不注日期的引用文件，其最新版本(包括所有的修改单)适用于本文件。

GB/T 6682 分析实验室用水规格和试验方法

## 3 原理

试样以盐酸乙腈混合溶液提取、正己烷脱脂离心，吸取部分下层溶液并调节 pH 值到 8～9，再用乙酸乙酯提取，旋转蒸干后，用液相色谱-串联质谱仪测定，内标法定量。

## 4 试剂和材料

除另有规定外，试剂均为分析纯，水为 GB/T 6682 规定的一级水。

4.1 正己烷。

4.2 异丙醇。

4.3 甲醇：色谱纯。

4.4 乙腈：色谱纯。

4.5 乙酸乙酯：色谱纯。

4.6 25%氨水。

4.7 *N*,*N*-二甲基甲酰胺：色谱纯。

4.8 *N*,*N*-二甲基甲酰胺-甲醇混合溶液：水-*N*,*N*-二甲基甲酰胺-甲醇溶液(7+2+1，体积比)。

4.9 浓盐酸：36%，密度为 1.18 g/L。

4.10 1 moL/L 盐酸：量取浓盐酸 83 mL，加水稀释至 1 000 mL。

4.11 0.75 mol/L 盐酸乙腈溶液：乙腈-1 mol/L 盐酸溶液(1+3，体积比)。

4.12 标准品：奥芬哒唑(oxfendazole，CAS 号 53716-50-0)、芬苯哒唑(fenbendazole，CAS 号 43210-67-9)、奥芬哒唑砜(oxfendazole sulphone，CAS 号 54029-20-8)、苯硫胍(febantel，CAS 号 58306-30-2)，纯度均大于 99%。

4.13 芬苯哒唑-d3 标准品：甲酯位的 3 个 H 被 D 取代，纯度大于 99%。

4.14 标准储备液：分别称取适量奥芬哒唑、芬苯哒唑、奥芬哒唑砜、苯硫胍、芬苯哒唑-d3，用 10 mL

*N*,*N*-二甲基甲酰胺溶解后，再用甲醇定为 100 μg/mL，0 ℃～4 ℃保存，有效期 6 个月。

4.15 标准工作溶液：根据需要，用乙腈-水溶液(2＋8，体积比)将奥芬哒唑、芬苯哒唑、奥芬哒唑砜、苯硫胍标准储备液配成供液相色谱-串联质谱测定用的浓度为 5 μg/L、10 μg/L、50 μg/L、100 μg/L、200 μg/L 的混合标准工作溶液。其中同位素内标芬苯哒唑-d3 浓度均为 10.0 μg/L，混合标准工作溶液使用前配制。

4.16 滤膜：0.2 μm，有机相。

## 5 仪器和设备

5.1 液相色谱-串联质谱仪，配有电喷雾离子源。

5.2 均质器。

5.3 pH 计。

5.4 涡旋振荡器。

5.5 离心机：4 000 r/min。

5.6 旋转蒸发仪。

5.7 超声波水浴。

5.8 分析天平：感量 0.1 mg，0.01 g。

## 6 试样制备与保存

制样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。

从所取全部样品中取出有代表性样品可食部分约 500 g，用组织捣碎机充分捣碎均匀，装入洁净容器中，密封，并标明标记，于－18 ℃以下冷冻存放。

## 7 测定步骤

### 7.1 提取和净化

称取 5 g 样品(准确至 0.01 g)于 50 mL 离心管中，加入 10 mL 乙腈盐酸溶液(4.11)、3 mL 异丙醇和 10 mL 正己烷，50 ng 芬苯哒唑-d3，涡旋混匀 2 min，14 000 r/min 速度高速匀质 30 s，以 4 000 r/min 离心 5 min，吸取下清液至 50 mL 离心管中；再用 10 mL 乙腈盐酸溶液按上述方法重复提取一次，合并下清液，并用水稀释到 25 mL，混匀。

吸取上述溶液 5 mL 到 50 mL 离心管，用氨水调节 pH 值到 8～9，再加入 10 mL 乙酸乙酯，涡旋混匀 3 min，以 4 000 r/min 离心 3 min，收集上层溶液。再向离心管中加入 1 mL 乙腈、5 mL 乙酸乙酯，重复上述操作。合并上层溶液，于 40 ℃减压旋转蒸发至干。

用 1.0 mL *N*,*N*-二甲基甲酰胺-甲醇混合溶液(4.8)溶解残渣，超声 5 min 后过 0.2 μm 滤膜，滤液供液相色谱-串联质谱测定。

### 7.2 测定

#### 7.2.1 液相色谱-质谱/质谱条件

7.2.1.1 色谱柱：SB $C_{18}$ 柱，100 mm×2.1 mm(内径)，1.8 μm 或相当者。

7.2.1.2 流动相：A：0.1%甲酸水溶液，B：0.1%甲酸乙腈溶液；流速：0.3 mL/min，梯度洗脱程序见表 1。

表 1　梯度洗脱程序

| 梯度时间<br>min | 流动相 A<br>% | 流动相 B<br>% |
|---|---|---|
| 0 | 90 | 10 |
| 1.0 | 50 | 50 |
| 3.0 | 25 | 75 |
| 5.0 | 25 | 75 |
| 7.0 | 10 | 90 |
| 7.5 | 90 | 10 |
| 10.0 | 90 | 10 |

7.2.1.3　柱温：35 ℃。

7.2.1.4　进样量：10 μL。

7.2.1.5　离子源：电喷雾 ESI，正离子。

7.2.1.6　扫描方式：多反应监测 MRM。

7.2.1.7　其他参数条件参见附录 A。

### 7.2.2　定量测定

根据试样中被测物的药物含量，选取响应值相近的标准工作液进行分析。标准工作液和待测液中药物的响应值均应在仪器线性响应范围内。如果含量超过标准曲线范围，应用乙腈-水(2+8)稀释到合适浓度后分析。在上述色谱条件下的待测药物的参考保留时间分别为：奥芬哒唑 4.4 min、奥芬哒唑砜 4.9 min、芬苯哒唑 5.5 min、苯硫胍 7.0 min，标准溶液的多反应监测色谱图参见附录 C。

### 7.2.3　定性测定

按照液相色谱-质谱/质谱条件测定样品和标准工作溶液，如果检测的质量色谱峰保留时间与标准品一致，定性离子对的相对丰度是用相对于最强离子丰度的强度百分比表示，应当与浓度相当标准工作溶液的相对丰度一致，相对丰度允许偏差不超过表 2 规定的范围，则可判断样品中存在对应的被测物。

表 2　定性确证时相对离子丰度的最大允许偏差

| 相对离子丰度 | >50% | >20%～50% | >10%～20% | ≤10% |
|---|---|---|---|---|
| 允许的相对偏差 | ±20% | ±25% | ±30% | ±50% |

## 7.3　空白试验

除不加试样外，均按上述操作步骤进行。

# 8　计算结果与表述

## 8.1　结果计算

用色谱数据处理机或按式(1)计算样品中待测药物残留量，计算结果需扣除空白值。

$$X=\frac{c_s\times A\times A_{si}}{A_i\times A_s\times m\times 1\,000} \qquad \cdots\cdots(1)$$

式中：

$X$ ——样品中待测组分残留量，单位为毫克每千克(mg/kg)；

$c_s$ ——标准工作溶液中药物的浓度，单位为微克每升(μg/L)；

$A$ ——样液中药物的峰面积；

$A_{si}$——标准工作溶液中内标物的峰面积；

$A_i$——样液中内标物的峰面积；

$A_s$——标准工作溶液中药物的峰面积；

$m$ ——试样质量，单位为克(g)。

### 8.2 表述

样品中奥芬哒唑、芬苯哒唑和苯硫胍的残留量均以可能被氧化成奥芬哒唑砜的可萃取残留总量计，可提取的奥芬哒唑砜残留量＝样品中苯硫胍残留量×0.74＋样品中奥芬哒唑残留量×1.05＋样品中芬苯哒唑残留量×1.11＋样品中奥芬哒唑砜残留量。

## 9 测定低限、回收率

### 9.1 测定低限(LOQ)

奥芬哒唑、芬苯哒唑、奥芬哒唑砜、苯硫胍测定低限均为 10 μg/kg。

### 9.2 回收率

添加回收率实验数据参见附录 B。

# 附 录 A
## （资料性附录）
## Agilent 6410B 液相色谱-四级杆串联质谱仪参数[1)]

Agilent 6410B 液相色谱-四级杆串联质谱仪参数如下：

a) 电离源：电喷雾正离子(ESI+)；

b) 离子源喷雾电源(IS)：3 500 V；

c) 离子源温度：350 ℃；

d) 气流速(Gas flow)：11 L/min；

e) 雾化气(Nebulizer)：276 kPa(40 psi)；

f) 电子倍增器电压(Delta EMV)：600 V；

g) 检测方法：多反应监测(MRM)。

**表 A.1 多反应监测参数表**

| 组分名称 | 母离子 m/z | 子离子 m/z | 去簇电压 V | 碰撞能量 V | 色谱保留时间 $t_r$ min |
|---|---|---|---|---|---|
| 奥芬哒唑 | 316 | 159* | 140 | 35 | 4.4 |
| | | 191 | | 15 | |
| 芬苯哒唑 | 300 | 159 | 120 | 35 | 5.5 |
| | | 268* | | 10 | |
| 奥芬哒唑砜 | 332 | 159 | 140 | 45 | 4.9 |
| | | 300* | | 20 | |
| 苯硫胍 | 447 | 415 | 140 | 1 | 7.0 |
| | | 383* | | 10 | |
| 芬苯哒唑-d3(内标) | 303 | 268 | 140 | 10 | 5.5 |
| 注：带“*”为定量离子对。 | | | | | |

1) 非商业性声明：附录 A 所列参数是在 Agilent 6410B 质谱仪完成的，此处列出试验用仪器型号仅是为了提供参考，并不涉及商业目的，鼓励标准使用者尝试不同厂家和型号的仪器。

# 附 录 B
（资料性附录）
添加回收率实验数据

表 B.1 添加回收率实验数据

| 基 质 | 药 物 | 添加浓度 | 回收率范围 |
|---|---|---|---|
| 牛肉 | 苯硫脲 | 10 μg/kg | 79.0%～96.0% |
| | | 50 μg/kg | 91.4%～101.6% |
| | | 100 μg/kg | 81.7%～102.5% |
| | 奥芬哒唑砜 | 10 μg/kg | 81.9%～107.0% |
| | | 50 μg/kg | 74.8%～98.2% |
| | | 100 μg/kg | 78.5%～102.5% |
| | 芬苯哒唑 | 10 μg/kg | 75.6%～106.0% |
| | | 50 μg/kg | 75.0%～99.0% |
| | | 100 μg/kg | 77.2%～94.5% |
| | 奥芬哒唑 | 10 μg/kg | 78.4%～102.0% |
| | | 50 μg/kg | 74.8%～99.2% |
| | | 100 μg/kg | 77.3%～98.7% |
| 猪肝 | 苯硫脲 | 10 μg/kg | 82.3%～102.0% |
| | | 50 μg/kg | 87.8%～105.2% |
| | | 100 μg/kg | 88.6%～103.6% |
| | 奥芬哒唑砜 | 10 μg/kg | 76.7～104.0% |
| | | 50 μg/kg | 76.8%～98.6% |
| | | 100 μg/kg | 77.5%～95.8% |
| | 芬苯哒唑 | 10 μg/kg | 78.8%～106.0% |
| | | 50 μg/kg | 73.2%～100.6% |
| | | 100 μg/kg | 75.7%～101.6% |
| | 奥芬哒唑 | 10 μg/kg | 75.4%～97.8% |
| | | 50 μg/kg | 75.0%～95.0% |
| | | 100 μg/kg | 76.3%～101.4% |
| 午餐肉 | 苯硫脲 | 10 μg/kg | 81.9%～107.0% |
| | | 50 μg/kg | 85.8%～104.6% |
| | | 100 μg/kg | 83.7%～106.5% |
| | 奥芬哒唑砜 | 10 μg/kg | 78.8%～102.0% |
| | | 50 μg/kg | 76.8%～99.2% |
| | | 100 μg/kg | 83.6%～108.57% |

表 B.1（续）

| 基　质 | 药　物 | 添加浓度 | 回收率范围 |
|---|---|---|---|
| 午餐肉 | 芬苯哒唑 | 10 μg/kg | 76.2%～98.6% |
| | | 50 μg/kg | 80.6%～99.2% |
| | | 100 μg/kg | 77.7%～101.4% |
| | 奥芬哒唑 | 10 μg/kg | 75.8%～105.0% |
| | | 50 μg/kg | 75.0%～100.6% |
| | | 100 μg/kg | 78.4%～104.2% |

# Foreword

This standard was drafted by GB/T 1.1—2009.

The method of determination of this standard was an amendment of SN 0684—1997《Method for determination of oxfendazole residues in meats and meat products for export》.

Difference between this standard and SN 0684—1997 are listed as following:

—The name of the standard was changed;

—The scope of the standard was expand, include oxfendazole, fenbenzazole, febantel and oxfendazole sulphone;

—The determination method was changed to HPLC-MS/MS;

—The expression of result was changed to the extractable residue content of oxfendazole sulphone, which is the oxidization product of the oxfendazole, fenbendazole, febantel.

This standard was proposed by and is under the charged of the Certification and Accreditation Administration of the People's Republic of China.

This standard was drafted by Shanghai Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

The standard was mainly drafted by Guo Dehua, Deng Xiaojun, Han Li, Sheng Yonggang, Yi Xionghai, Zhao Shanzhen.

This standard was a professional standard for entry-exit inspection and quarantine promulgated for the first time in 1997, and this version is the first amendment.

# Determination of oxfendazole, fenbenzazole, febantel and oxfendazole sulphone residues in meat and meat products for export—HPLC-MS/MS method

## 1 Scope

The standard specifies the methods of sample preparation and determination of oxfendazole, fenbendazole, febantel and oxfendazole sulphone in foodstuffs of meat and meat products.

This standard is applicable to determination of oxfendazole, fenbendazole, febantel and oxfendazole sulphone residues in beef, pork liver and luncheon meat.

## 2 Normative references

The following documents are necessary for this standard. For dated references, only dated editions shall apply to this standard. For undated references, the latest edition of the normative document (including subsequent amendments) referred to applies.

GB/T 6682 Water for analytical laboratory use—Specification and test methods

## 3 Principle

The oxfendazole, fenbendazole, febantel and oxfendazole sulphone residues derived from samples are extracted by the mixture of HCl and acetonitrile, and degreased by *n*-hexane. After centrifuging, adjust the pH value of lower supernatant to 8～9, and then extract with ethyl acetate. The anyalytes are determined by HPLC-MS/MS, and quantified by isotope internal standard method.

## 4 Reagents and materials

Unless otherwise specified, all the reagent used should be analytical grade, water is deionized water specified by the standard GB/T 6682.

**4.1** *n*-hexane: HPLC grade.

**4.2** Isopropanol: HPLC grade.

4.3 Methanol: HPLC grade.

4.4 Acetonitrile: HPLC grade.

4.5 Ethyl acetate: HPLC grade.

4.6 25% ammonia.

4.7 *N*, *N'*-dimethylformamide: HPLC grade.

4.8 $H_2O$-*N*, *N'*-dimethylformamide-methanol (7+2+1, *V*/*V*).

4.9 HCl: 36% density of 1.18 g/L.

4.10 1 mol/L HCl: Mix83 mL 36% HCl with water to final volume of 1 000 mL.

4.11 0.75 mol/L HCl-acetonitrile: Acetonitrile-1 mol/L HCl(1+3, *V*/*V*).

4.12 Standards: Oxfendazole, (CAS No.: 53716-50-0), Fenbendazole (CAS No.: 43210-67-9), Oxfendazole sulphone, (CAS No.: 54029-20-8), Febantel (CAS No.: 58306-30-2). Purity>99%.

4.13 Fenbendazole-d3: Purity>99%.

4.14 Stock standard solution: Accurately weigh appropriate standard oxfendazole, fenbendazole, oxfendazole sulphone and febantel, and fenbendazole-d3, then dissolve them in 10 mL *N*, *N'*-dimethylformamide individually, and then dilute to 100 mL with methanol and the concerntration of these solutions is 100 μg/mL. Store them refrigerated at 0 ℃～4 ℃, valid for 6 months.

4.15 Working solutions: To require as useful, mix and dilute the stock standard solutions of oxfendazole, fenbendazole, oxfendazole sulphone and febantel with acetonitrile-water (2+8) to five concentration levels 5 μg/L, 10 μg/L, 50 μg/L, 100 μg/L, 200 μg/L and the concentration of internal standard of fenbendazole-d3 is 10.0 μg/L. And the mixed standard working solutions are prepared before use.

4.16 Organic phase film: 0.2 μm.

## 5 Apparatus and equipment

5.1 Liquid chromatograhy combined with electrospray ionization mass spectrometry.

5.2 Homogenizer.

5.3 pH meter.

5.4 Vortex mixer.

5.5 Centrifuger:4 000r/min.

5.6 Rotary vacuum evaporator.

5.7 Ultrasonic water bath apparatus.

5.8 Balances (0.1 mg,0.01 g).

## 6 Preparation and storage of test sample

In the course of sample preparation, precautions shall be taken to avoid contamination or any factors that may cause the change of residue content.

Take the representative portions from the whole primary sample. It is about 500 g and ground in a blender. Keep the sample into a clean vessel, seal and label. The test sample is stored below −18 ℃.

## 7 Test procedures

### 7.1 Extraction and clean up

Weigh 5 g (accurate to 0.01 g) sample in a 50 mL centrifuge tube, add 10 mL acetonitrile-HCl (4.11), 3 mL isopropanol, 10 mL *n*-hexane, and 50 ng fenbendazole-d3, mix them for 2 min and followed by homogenization at 14 000 r/min for 30 s. The mixture was centrifuged at 4 000 r/min for 5 min. Transfer the lower supernatant to another centrifuge tube and repeat the extraction for one time. Combine the lower supernatant and dilute to 25 mL by deionized water.

Take 5 mL of the solution mentioned above to a 50 mL centrifuge tube, adjust the pH to 8～9, add 10 mL of ethyl acetate, mix for 3 min and centrifuge at 4 000 r/min for 3 min, collect the upper supernatant and add 1 mL acetonitrile and 5 mL ethyl acetate and repeat the porcedures above. Combine the organic phase and evaporate to nearly dryness below 40 ℃.

Dissolve the residue by 1.0 mL of $H_2O$-*N*, *N*′-dimethylformamide-methanol (4.8), mix them in ultrasonic water bath for 5 min and filter by 0.2 μm film for HPLC-MS/MS analysis.

### 7.2 Determination

#### 7.2.1 HPLC-MS/MS operating conditions

**7.2.1.1** Column: SB $C_{18}$, 1.8 μm, 100 mm × 2.1 mm (i.d.), or the equivalent.

**7.2.1.2** Mobile phase: A: 0.1% formic acid, B: Acetonitrile + 0.1% formic acid. Flow rate: 0.3 mL/min. The gradient program is listed in the table 1.

Table 1—Gradient program of mobile phase

| Time<br>min | Mobile phase A<br>% | Mobile phase B<br>% |
|---|---|---|
| 0 | 90 | 10 |
| 1.0 | 50 | 50 |
| 3.0 | 25 | 75 |
| 5.0 | 25 | 75 |
| 7.0 | 10 | 90 |
| 7.5 | 90 | 10 |
| 10.0 | 90 | 10 |

**7.2.1.3** Column temperature: 35 ℃.

**7.2.1.4** Injection volume: 10 μL.

**7.2.1.5** Ion source: ESI, positive mode.

**7.2.1.6** Scan mode: multiple reaction monitoring (MRM).

**7.2.1.7** Other related parameters are listed in the annex A.

#### 7.2.2 Quantitation of HPLC-MS/MS

According to the above HPLC-MS/MS operating condition, determine the sample solution and the standard working solution simultaneously. The responses of the analytes in the standard working solution and the sample solution should be within the linear range of the instrument detection. Quantified by internal standard. Under the above HPLC-MS/MS operating condition, the retention time of benzodiazepines are listed as following, oxfendazole(4.4 min), fenbendazole (5.5 min), oxfendazole sulphone(4.9 min) and febantel (7.0 min). The MRM chromatograms of the standards are presented in the annex C.

### 7.2.3 Confirmation of HPLC-MS/MS

Determine the sample under the established LC/MS-MS conditions, and calculate the intensity ratio of two selected ion pairs of the sample solution and the standard working solutions. If the retention time of sample chromatogram peaks are consistent with that of working solution, and the relative abundance ratio tolerance meets the requirements listed in the table 2, it can be concluded that this compound do exist in the sample.

Table 2—Maximum permitted tolerances for relative ion intensities while confirmation

| Relative ion intensities | >50% | >20%～50% | >10%～20% | ≤10% |
|---|---|---|---|---|
| Permitted relative tolerances | ±20% | ±25% | ±30% | ±50% |

## 7.3 Blank test

The operation of the blank test is the same as the described in the method of determination, but without addition of the sample.

# 8 Calculation and expression of result

## 8.1 Calculation

Calculate the content of benzodiazepine residues in the test sample by HPLC-MS/MS data processor or according to the formula (1). The blank value should be subtracted from the above result of calculation.

$$X = \frac{c_s \times A \times A_{si}}{A_i \times A_s \times m \times 1000} \qquad \cdots\cdots(1)$$

Where:

$X$ —the residue content in the test sample, mg/kg;

$c_s$ —the concentration in standard working solution, μg/L;

$A$ —the peak area of residue in sample solution;

$A_{si}$—the peak area of internal standard in standard working solution;

$A_i$ —the peak area of internal standard in sample solution;

$A_s$ —the peak area of residue in standard working solution;

$m$ —the mass of test sample of final sample solution, g.

### 8.2 Expression of result

The residues of oxfendazole, fenbendazole and febantel in the sample are calculated as the extractable residue content of oxfendazole sulphone, which is the oxidization product of the fomer 3 compounds.

The extractable residue content of oxfendazole sulphone = the residue content of febantel in the test sample × 0.74 + the residue content of oxfendazole in the test sample × 1.05 + the residue content of fenbendazole in the test sample × 1.11 + the residue content of oxfendazole sulphone in the test sample.

## 9 Limit of quantitation (LOQ) and recovery

### 9.1 Limit of quantification (LOQ)

The values of LOQ for oxfendazole, fenbendazole, oxfendazole sulphone and febantel are all 10 μg/kg.

### 9.2 Recovery

According to the experimental data, the corresponding recovery of each fortifing level is listed in the annex B.

# Annex A
# (Informative annex)
# Main mass parameters of Aglient 6410B[1)]

Main mass parameters of Aglient 6410B are as follows:

a) Ion source:ESI,Positive (ESI+);

b) Electrospray capillary voltage:3 500 V;

c) Ion source tempeture:350 ℃;

d) Gas flow:11 L/min;

e) Nebulizer:276 kPa (40 psi);

f) Delta EMV:600 V;

g) Monitor mode:Multiple reaction monitoring,MRM.

**Table A.1—Related parameters in MRM**

| Compounds | Parention m/z | Daughter ion m/z | Cone Voltage V | Collision energy V | Retention time $t_r$ min |
|---|---|---|---|---|---|
| Oxfendazole | 316 | 159* | 140 | 35 | 4.4 |
| | | 191 | | 15 | |
| Fenbendazole | 300 | 159 | 120 | 35 | 5.5 |
| | | 268* | | 10 | |
| Oxfendazole sulphone | 332 | 159 | 140 | 45 | 4.9 |
| | | 300* | | 20 | |
| Febantel | 447 | 415 | 140 | 1 | 7.0 |
| | | 383* | | 10 | |
| Fenbendazole-d3 | 303 | 268 | 140 | 10 | 5.5 |
| Note: The ions signed" * "is quantification ions. | | | | | |

1) Non-commercial statement:the equipments and their types involved in the standard method are not related to commercial aims,and it is encouraged to use equipments of different corporation of different type.

# Annex B
(Informative annex)
## The recovery ranges of febantel, oxfendazole, fenbendazole and oxfendazole sulfone residues

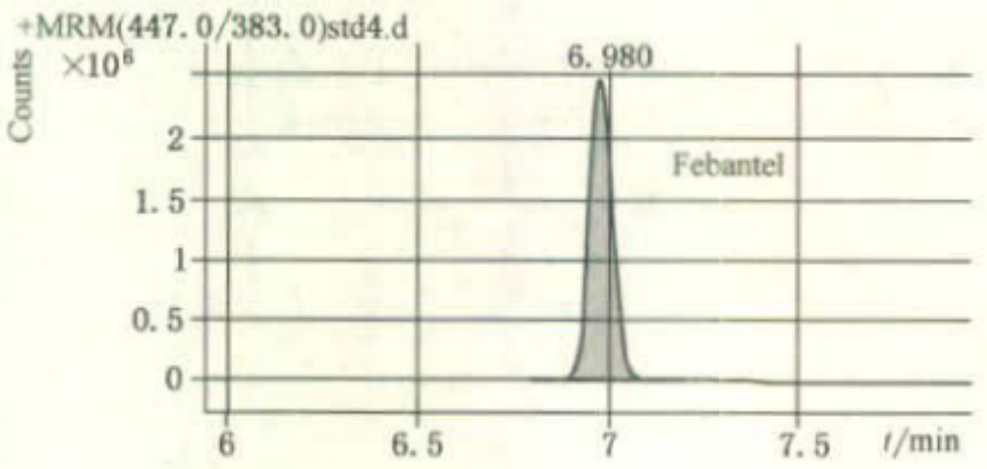
Table B. 1—The recovery ranges of febantel, oxfendazole, fenbendazole and oxfendazole sulfone residues

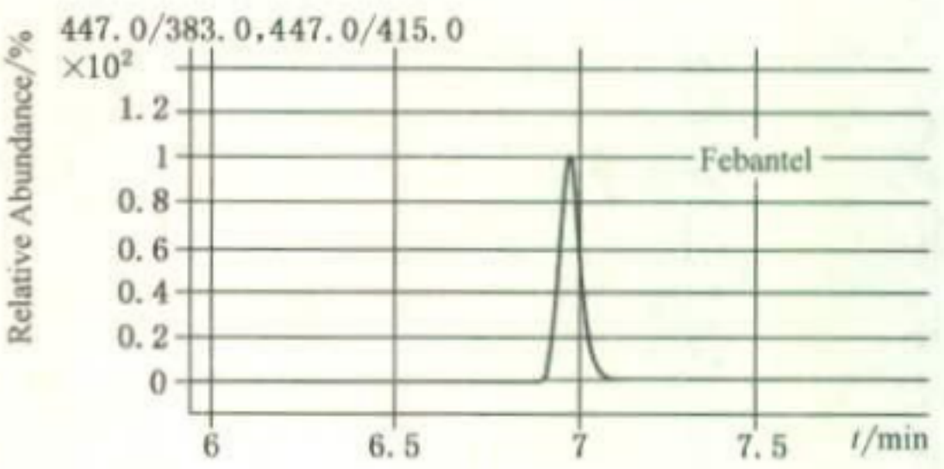
| Matrix | Residue | Fortified level | Recovery range |
|---|---|---|---|
| Beef | Febantel | 10 μg/kg | 79.0%～96.0% |
| | | 50 μg/kg | 91.4%～101.6% |
| | | 100 μg/kg | 81.7%～102.5% |
| | Oxfendazole sulphone | 10 μg/kg | 81.9%～107.0% |
| | | 50 μg/kg | 74.8%～98.2% |
| | | 100 μg/kg | 78.5%～102.5% |
| | Fenbendazole | 10 μg/kg | 75.6%～106.0% |
| | | 50 μg/kg | 75.0%～99.0% |
| | | 100 μg/kg | 77.2%～94.5% |
| | Oxfendazole | 10 μg/kg | 78.4%～102.0% |
| | | 50 μg/kg | 74.8%～99.2% |
| | | 100 μg/kg | 77.3%～98.7% |
| Pork liver | Febantel | 10 μg/kg | 82.3%～102.0% |
| | | 50 μg/kg | 87.8%～105.2% |
| | | 100 μg/kg | 88.6%～103.6% |
| | Oxfendazole sulphone | 10 μg/kg | 76.7～104.0% |
| | | 50 μg/kg | 76.8%～98.6% |
| | | 100 μg/kg | 77.5%～95.8% |
| | Fenbendazole | 10 μg/kg | 78.8%～106.0% |
| | | 50 μg/kg | 73.2%～100.6% |
| | | 100 μg/kg | 75.7%～101.6% |
| | Oxfendazole | 10 μg/kg | 75.4%～97.8% |
| | | 50 μg/kg | 75.0%～95.0% |
| | | 100 μg/kg | 76.3%～101.4% |
| Luncheon meat | Febantel | 10 μg/kg | 81.9%～107.0% |
| | | 50 μg/kg | 85.8%～104.6% |
| | | 100 μg/kg | 83.7%～106.5% |
| | Oxfendazole sulphone | 10 μg/kg | 78.8%～102.0% |
| | | 50 μg/kg | 76.8%～99.2% |
| | | 100 μg/kg | 83.6%～108.57% |

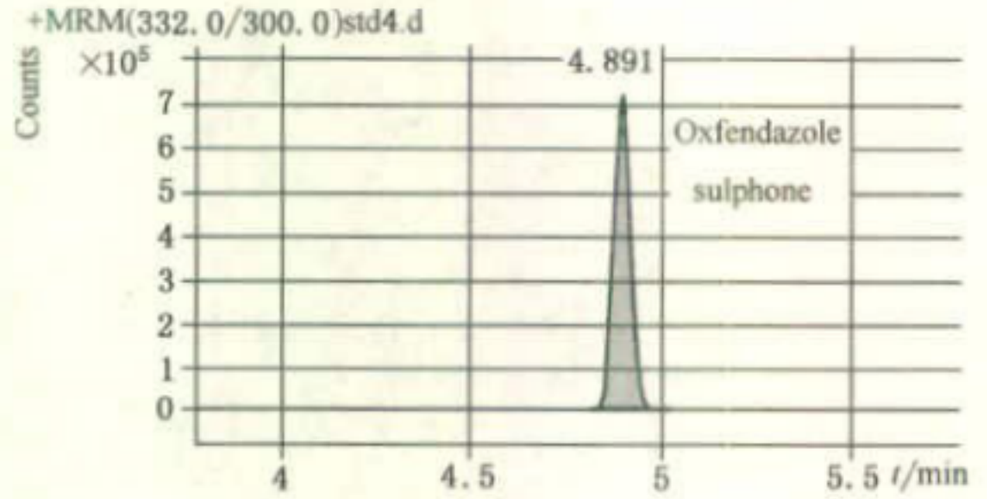
Table B.1 (continued)

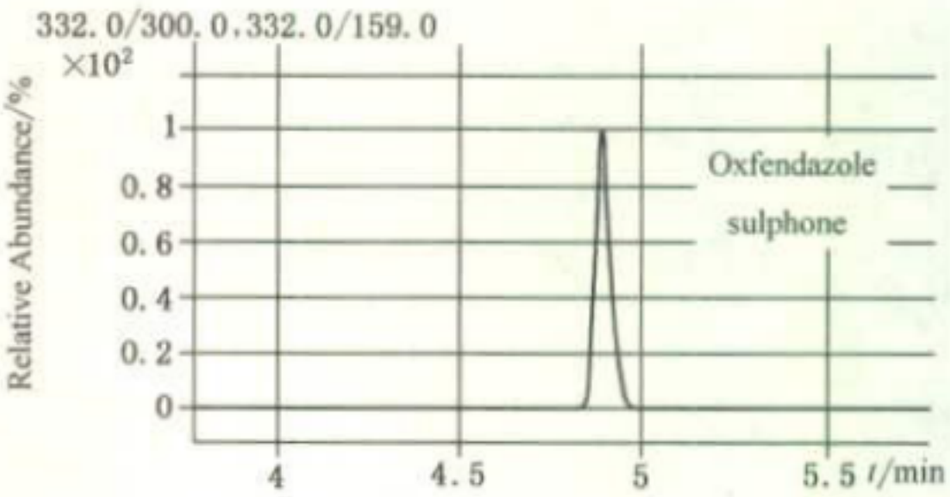
| Matrix | Residue | Fortified level | Recovery range |
|---|---|---|---|
| Luncheon meat | Fenbendazole | 10 μg/kg | 76.2%～98.6% |
| | | 50 μg/kg | 80.6%～99.2% |
| | | 100 μg/kg | 77.7%～101.4% |
| | Oxfendazole | 10 μg/kg | 75.8%～105.0% |
| | | 50 μg/kg | 75.0%～100.6% |
| | | 100 μg/kg | 78.4%～104.2% |

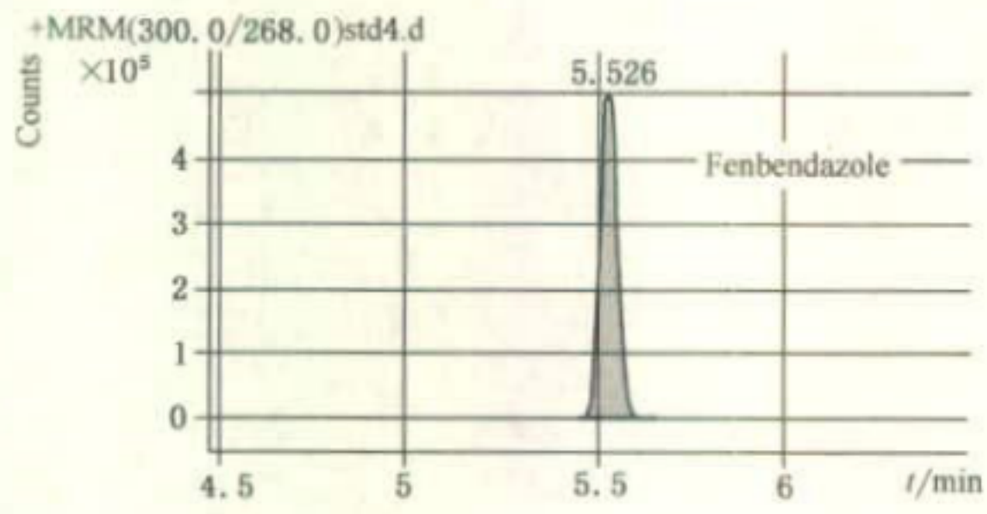
# Annex C
(Informative annex)
Selected ion chromatograms of oxfendazole, fenbendazole, oxfendazole sulphone, febantel and fenbendazole-d3 (MRM)

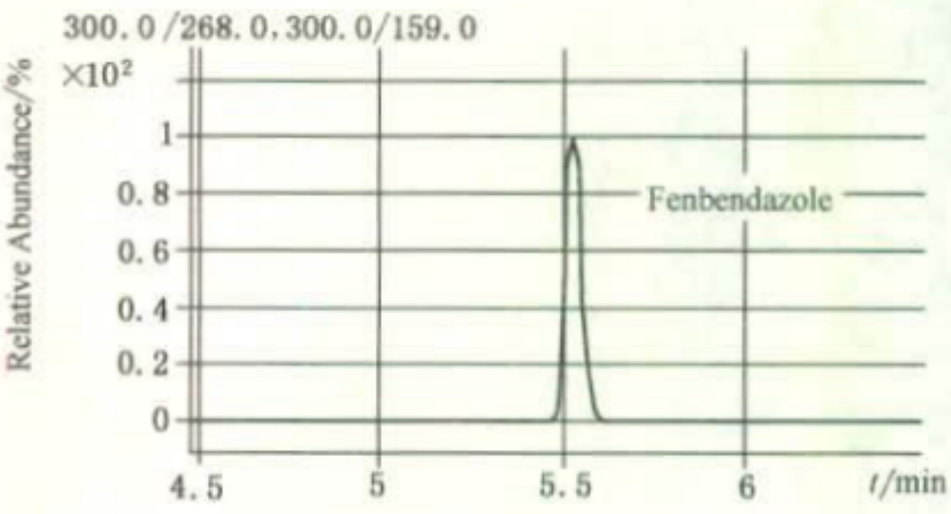


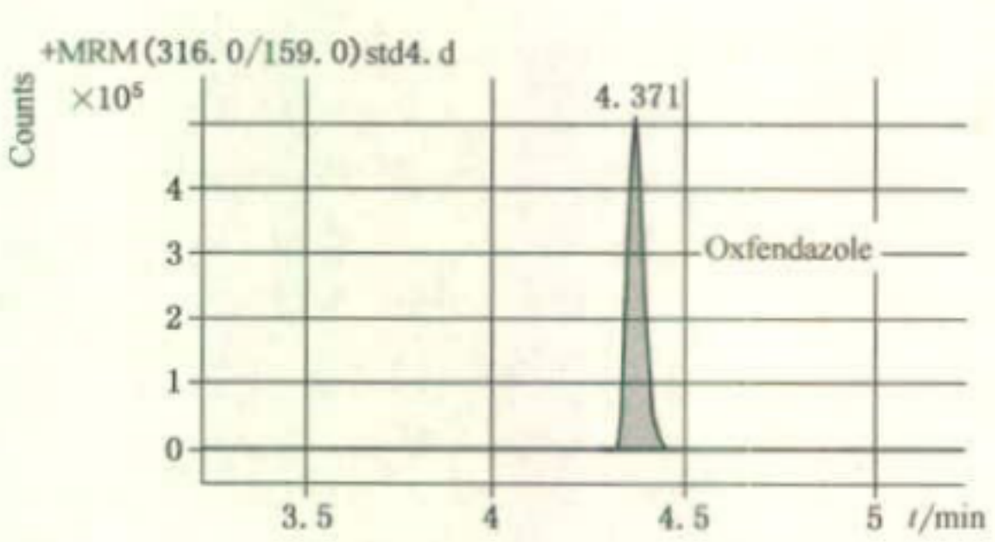



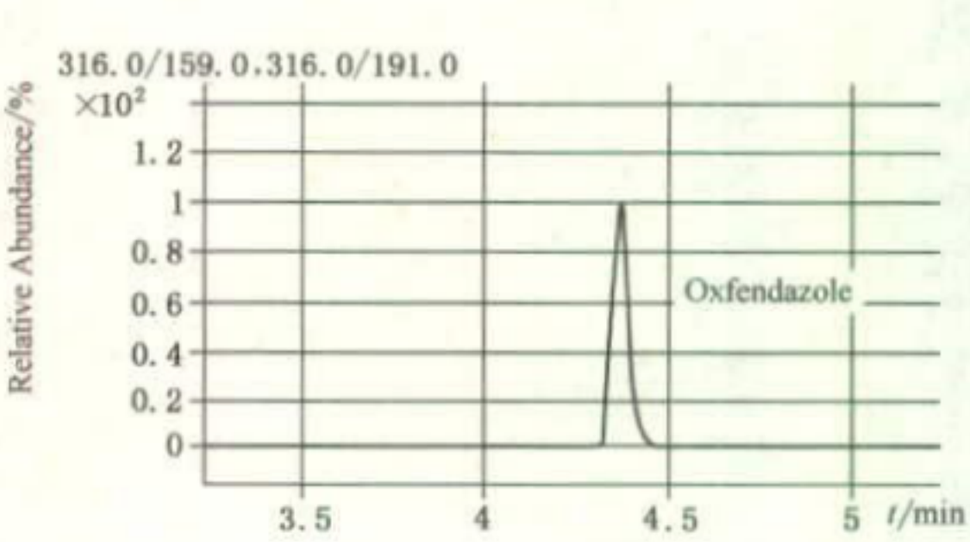




Figure C.1—MRM chromatogram of oxfendazole, fenbendazole, oxfendazole sulphone, febantel and fenbendazole-d3 standards

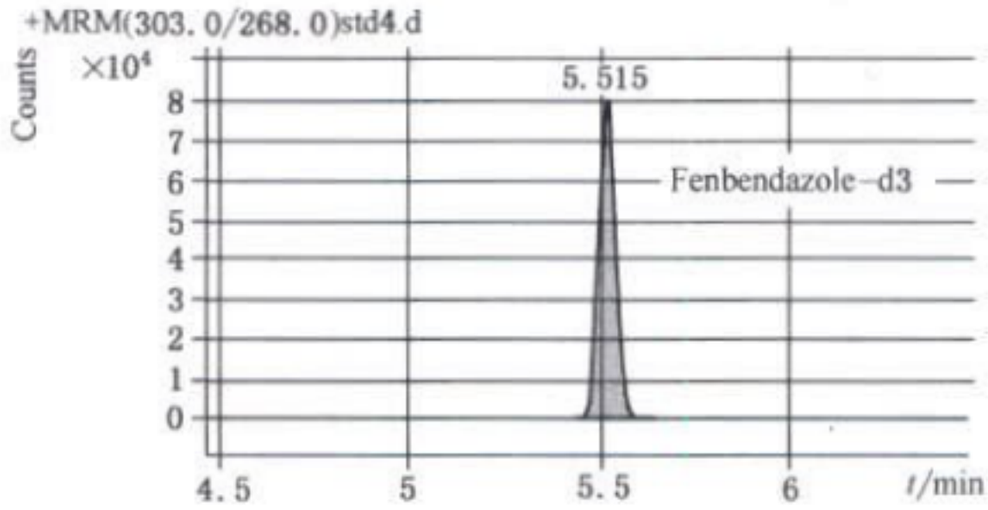



**Figure C.1** (continued)

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