SN

中华人民共和国出入境检验检疫行业标准

SN/T 2318—2009

动物源食品中地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜残留量的检测 高效液相色谱-质谱/质谱法

Determination of diclazuril, toltrazuril, toltrazuril sulfoxide and toltrazuril sulfone residues in foodstuffs of animal origin—HPLC-MS/MS method

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前　言

本标准的附录A、附录B和附录C均为资料性附录。

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河北出入境检验检疫局负责起草。

本标准主要起草人：王凤池、郭春海、艾连峰、陈瑞春、邢军。

本标准系首次发布的出入境检验检疫行业标准。

动物源食品中地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜残留量的检测 高效液相色谱-质谱/质谱法

1 范围

本标准规定了动物源食品中地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜残留量的高效液相色谱-质谱/质谱检测方法。

本标准适用于鸡肉、鸡肝、鸡肾、猪肉、猪肝、猪肾、兔肉、兔肝、兔肾和鸡蛋中地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜残留量的检测。

2 规范性引用文件

下列文件中的条款通过本标准的引用而成为本标准的条款。凡是注日期的引用文件，其随后所有的修改单(不包括勘误的内容)或修订版均不适用于本标准，然而，鼓励根据本标准达成协议的各方研究是否可使用这些文件的最新版本。凡是不注日期的引用文件，其最新版本适用于本标准。

GB/T 6682 分析实验室用水规格和试验方法

3 方法提要

试样中的地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜残留用乙酸乙酯提取，通过凝胶渗透色谱(GPC)净化后，用高效液相色谱-串联质谱(HPLC-MS/MS)测定，内标法定量。

4 试剂和材料

除另有规定外，所用试剂均为色谱纯，水为 GB/T 6682 规定的一级水。

4.1 乙腈。

4.2 乙酸。

4.3 环己烷。

4.4 乙酸乙酯。

4.5 甲醇。

4.6 无水硫酸钠：分析纯，经 650 ℃灼烧 4 h，置于干燥器内备用。

4.7 乙酸乙酯-环己烷(50+50)：相同体积的乙酸乙酯与环己烷互溶。

4.8 甲醇-水(80+20)：80 体积的甲醇和 20 体积的水互溶。

4.9 标准品：地克珠利(CAS 号：101831-37-2)、妥曲珠利(CAS 号：69004-03-1)、妥曲珠利亚砜(CAS 号：69004-15-5)、妥曲珠利砜(CAS 号：69004-04-2)和氘代妥曲珠利，纯度均大于等于 98.0%。

4.10 地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜标准储备液：分别称取每种标准品各 10 mg(准确至 0.1 mg)，用甲醇溶解，并分别定容到 50 mL 棕色容量瓶中，混匀，该溶液的浓度为 200 μg/mL，可在－18 ℃以下避光保存 12 个月。

4.11 地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜混合标准中间液：分别取标准储备液(4.10)各 0.5 mL～10 mL 容量瓶中，用甲醇定容至刻度，配制成混合标准中间液，浓度为 10 μg/mL，可在－18 ℃以下避光保存 6 个月。

4.12 地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜标准工作液：根据需要，吸取一定量的混合标准中间液(4.11)，用甲醇稀释至所需浓度，现用现配。

4.13 内标标准储备溶液：称取氘代妥曲珠利标准品 10 mg(准确至 0.1 mg)，用甲醇溶解，并分别定容到 50 mL 棕色容量瓶中，混匀，该溶液的浓度为 200 μg/mL，可在－18 ℃以下避光保存 12 个月。

4.14 内标标准工作液：量取标准储备液(4.13)0.5 mL～10 mL 容量瓶中，用甲醇定容至刻度，配制成内标工作液，浓度为 10 μg/mL，可在－18 ℃以下避光保存 6 个月。

4.15 0.45 μm 有机滤膜。

5 仪器和设备

5.1 高效液相色谱-串联质谱仪：配有电喷雾离子源(ESI)。

5.2 凝胶渗透色谱仪：配有紫外检测器(固定波长：254 nm)。

5.3 涡旋振荡器。

5.4 旋转蒸发器。

5.5 均质器。

5.6 离心机，最大转速大于等于 3 000 r/min。

6 试样制备与保存

6.1 动物肌肉、肝脏和肾脏样品

从所取全部样品中取出有代表性样品可食部分约 0.5 kg，用组织捣碎机充分捣碎均匀，均分成两份，分别装入洁净容器中，密封，并标明标记，于－18 ℃以下冷冻避光保存。

6.2 禽蛋样品

从所取全部样品中取出约 0.5 kg，去壳，匀浆，均分成两份，分别装入洁净容器中，密封，并标明标记，于－18 ℃以下冷冻避光保存。

7 测定步骤

7.1 提取

肌肉样品、禽蛋样品称取 5 g(精确至 0.01 g)，肾脏、肝脏样品称取 2.5 g(精确至 0.01 g)于 50 mL 离心管中，加入 50 μL 内标工作液(4.14)，涡旋混合 30 s 后，加入 25 mL 乙酸乙酯和 5 g 无水硫酸钠，以 10 000 r/min 均质提取 1 min，3 000 r/min 转速离心 10 min。然后禽蛋样品准确分取 10 mL 提取液，肌肉、肾脏、肝脏样品分取 5 mL 提取液于 15 mL 的玻璃管中，45 ℃下氮气吹干。用乙酸乙酯-环己烷(4.7)溶解残渣并定容至 10 mL，待 GPC 净化。

7.2 净化

7.2.1 凝胶渗透色谱条件

a) 净化柱：22 g S-X3 Bio-Beads 填料，200 mm×20 mm(内径)，或相当者；

b) 流动相：乙酸乙酯-环己烷(50＋50，体积比)，流速：5.0 mL/min；

c) 进样量：5 mL；

d) 净化程序：弃去 7.5 mL 以前的洗脱液，收集 7.5 min～12.5 min 的洗脱液。

7.2.2 净化过程

按上述的条件进行 GPC 净化。净化后将收集的洗脱液蒸干，用 1 mL 甲醇-水(4.8)溶解残渣，涡漩振荡，过 0.45 μm 滤膜，供 HPLC-MS/MS 分析。

7.3 高效液相色谱-串联质谱测定

7.3.1 液相色谱条件

a) 色谱柱：C_{18}，150 mm×2.1 mm(内径)，5 μm，或相当者；

b) 流动相:0.1%乙酸水溶液-乙腈(45+55,体积比);

c) 流速:200 μL/min;

d) 色谱柱温度:30 ℃;

e) 进样量:10 μL。

7.3.2 质谱条件

a) 离子化模式:电喷雾负离子扫描模式(ESI-);

b) 质谱扫描方式:多反应监测(MRM);

c) 其他参考质谱条件参见附录 A。

7.3.3 测定

7.3.3.1 定量测定

根据样液中被测物的含量情况,选定响应值相近的混合标准工作液。标准工作溶液和样液中分析物的响应值均应在仪器的检测线性范围内。对标准工作溶液和样液等体积参差进样测定。在上述仪器条件下,各分析物的参考保留时间为:妥曲珠利亚砜 4.73 min、妥曲珠利砜 7.81 min、地克珠利 11.02 min、氘代妥曲珠利 13.53 min、妥曲珠利 13.61 min;标准品色谱图参见附录 B。

7.3.3.2 定性测定

地克珠利和妥曲珠利亚砜有两组离子对,能够定性分析。在相同实验条件下,在扣除背景后的样品质量色谱图中,所选择的离子对均出现。经对比所选离子对的丰度比与标准品离子对的丰度比,其值在允差范围内(允差范围值参见表 1),则可判断样品中存在对应的被测物。

表 1 定性确证时相对离子丰度的最大允许误差

相对离子丰度/%	>50	>20~50	>10~20	≤10
允许的相对误差/%	±20	±25	±30	±50

7.4 空白实验

除不称取样品外,均按上述测定条件和步骤进行。

8 结果计算和表述

试样中的残留含量,按式(1)计算:

$$X = \frac{c_s \times A \times c_i \times A_{si} \times V}{A_s \times c_{si} \times A_i \times m} \quad \cdots\cdots (1)$$

式中:

X——试样中待测组分残留量,单位为微克每千克(μg/kg);

c_s——标准工作溶液中被测物的浓度,单位为纳克每毫升(ng/mL);

A——样品中被测组分峰面积;

A_s——混合标准工作溶液中被测组分的峰面积;

c_i——样液中内标物的浓度,单位为纳克每毫升(ng/mL);

c_{si}——标准工作溶液中内标物的浓度,单位为纳克每毫升(ng/mL);

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;

V——样液最终定容体积,单位为毫升(mL);

m——样品的重量,单位为克(g)。

9 测定低限、回收率

9.1 测定低限

本方法的测定低限：肾脏、肝脏为 20 μg/kg；肌肉为 10 μg/kg；禽蛋为 1 μg/kg。

9.2 回收率

地克珠利、妥曲珠利、妥曲珠利亚砜、妥曲珠利砜在不同基质，三个不同添加水平的回收率范围参见附录 C。

附 录 A
（资料性附录）
质谱条件[1)]

参考质谱条件：

a） 鞘气压力：30 unit；

b） 辅助气压力：8 unit；

c） 负离子模式电喷雾电压(IS)：－3 500 V；

d） 毛细管温度：320 ℃；

e） 源内诱导解离电压：10 V；

f） Q1，Q3 分辨率：0.7；

g） 碰撞气：高纯氩气；

h） 碰撞气压力：1.5 mTorr；

i） 其他质谱参数见表 A.1。

表 A.1 被测物的采集时间段、离子对和碰撞能量

化合物	保留时间	离子对	碰撞能量/eV
妥曲珠利亚砜	4.73	440.1/371.1[a] 440.1/383.1	18 14
妥曲珠利砜	7.81	456.0/456.0[a]	0
地克珠利	11.02	404.1/334.0[a] 404.1/335.0	19 19
妥曲珠利	13.53	424.0/424.0[a]	0
氘代妥曲珠利砜	13.61	427.0/427.0[a]	0
注：对于不同质谱仪器，仪器参数可能存在差异，测定前应将质谱参数优化到最佳。			
[a] 为定量离子对。			

1） 非商业性声明：附录 A 所列参考质谱条件是在 Thermo TSQ Quantum Ultra AM 型液质联用仪上完成的，此处多列出试验用仪器型号仅为提供参考，并不涉及商业目的，鼓励标准使用者尝试不同厂家或型号的仪器。

附　录　B
（资料性附录）
标准物质多反应监测质谱图

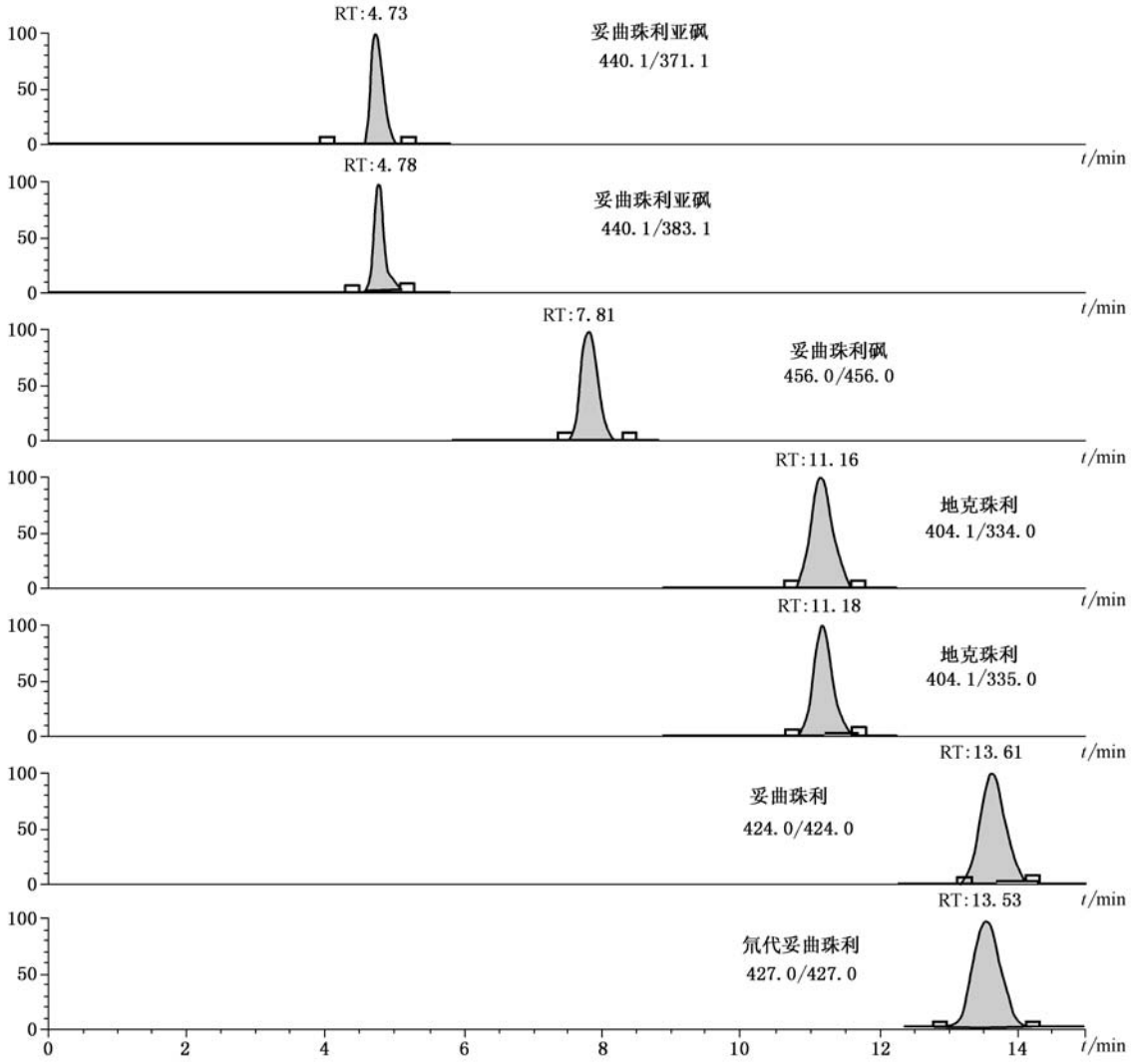

图 B.1　地克珠利、妥曲珠利、妥曲珠利亚砜和妥曲珠利砜标准多反应监测质谱图

附 录 C
（资料性附录）
回收率范围

表 C.1 添加回收率范围数据

基质	添加浓度/(μg/kg)	回收率/%			
		妥曲珠利亚砜	妥曲珠利砜	地克珠利	妥曲珠利
鸡蛋	1	87.0～101	82.0～102	84.0～105	87.0～106
	10	88.8～114	85.2～105	90.1～108	86.5～109
	50	86.6～110	87.2～107	89.0～108	86.0～105
鸡肉	10	78.9～91.3	90.0～104	96.0～109	91.0～106
	100	81.4～103	88.2～101	90.8～110	96.1～108
	500	80.5～104	87.0～109	85.0～98.5	88.5～102
鸡肝	20	80.5～93.5	92.0～107	92.5～106	93.5～108
	100	81.8～104	88.6～102	91.2～104	96.5～108
	600(3 000)[a]	80.2～101	85.0～107	83.2～96.4	86.6～99.6
鸡肾	20	82.5～95.5	90.5～104	93.0～104	91.0～107
	100	83.4～108	86.2～103	92.6～105	92.5～105
	400(1 000)[a]	86.4～99.6	82.8～106	80.8～94.0	84.4～96.8
猪肉	10	81.0～94.1	93.1～105	95.7～105	98.7～109
	100	84.0～106	90.2～103	92.9～103	98.3～108
	500	85.0～104	91.5～107	89.0～102	92.5～105
猪肝	20	88.5～107	83.5～102	83.5～101	84.5～102
	100	86.1～99.7	85.7～99.3	85.0～103	82.0～103
	500(3 000)[a]	85.0～107	90.8～107	83.6～107	86.0～108
猪肾	20	93.0～102	88.0～105	87.5～105	92.5～105
	100	92.4～106	94.2～104	94.3～109	91.7～106
	250(1 000)[a]	91.2～104	88.4～106	86.8～107	91.2～107
兔肉	10	84.3～98.0	91.4～104	94.2～105	86.3～102
	100	84.1～102	91.1～103	93.0～104	98.5～108
	500	82.5～108	86.0～108	88.5～102	91.5～105
兔肝	20	91.0～105	86.5～104	86.0～104	86.5～104
	100	89.7～103	89.8～109	87.4～105	90.1～109
	500(3 000)[a]	96.4～105	92.8～104	90.6～103	90.8～106
兔肾	20	94.0～105	91.0～108	88.5～108	91.5～105
	100	89.8～106	96.7～107	89.4～105	88.2～107
	250(1 000)[a]	91.2～101	88.8～107	87.2～107	92.0～107

[a] 括号中为地克珠利的添加水平。

Foreword

Annex A, Annex B and Annex C of this standard is informative.

This standard was proposed by and is under the charge of National Regulatory Commission for Certification and Accreditation.

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

The main drafters of this standard are Wang Fengchi, Guo Chunhai, Ai Lianfeng, Chen Ruichun and Xing Jun.

This standard is an Entry-Exit inspection and quarantine professional standard promulgated for the first time.

Determination of diclazuril, toltrazuril, toltrazuril sulfoxide and toltrazuril sulfone residues in foodstuffs of animal origin —HPLC-MS/MS method

1 Slope

The standard specifies the methods of determination of diclazuril, toltrazuril, toltrazuril sulfoxide and toltrazuril sulfone residues in animal origin foods by liquid chromatography-mass spectrometry.

This standard is applicable to the determination of diclazuril, toltrazuril, toltrazuril sulfoxide and toltrazuril sulfone residues in poultry muscle, poultry liver poultry kidney, swine muscle, swine liver, swine kidney, rabbit muscle, rabbit liver, rabbit kidney and egg.

2 References

The following normative documents contain provisions which, through references in this text, constitute provisions of this standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

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

3 Abstract of this method

The residues in the test sample are extracted with ethyl acetate, cleaned up with gel permeation chromatography (GPC). Determination is made by LC-MS/MS using the internal standard method.

4 Reagents and materials

Unless specifically noted, all reagents used should be of HPLC grade; "water" is fhe first grade water prescribed by GB/T 6682.

4.1 Acetonitrile.

4.2 Acetic acid.

4.3 Cyclohexane.

4.4 Ethyl acetate.

4.5 Methanol.

4.6 Anhydrous sodium sulfate: Analytical grade, Ignite at 650 ℃ for 4 h, and keep in a tightly closed container after cool.

4.7 Cyclohexane-ethyl acetate(50+50): Mix the same volume ethyl acetate and cyclohexane.

4.8 Methanol-water(80+20): Mix 80 volume units methanol and 20 volume units water.

4.9 Standards of diclazuril(CAS No:101831-37-2), toltrazuril(CAS No:69004-03-1), toltrazuril sulfoxide (CAS No: 69004-15-5), toltrazuril sulfone (CAS No: 69004-04-2) and toltrazuril-D_3: purity≥98.0%

4.10 Stock standard solution: Accurately weigh 10 mg (accurate to 0.1 mg) standard, dissolve in 50 mL methanol and mix to homogeneity. The concentration of the solutions is 200 μg/mL. The solutions should be stored below −18 ℃ in dark for more than 12 months.

4.11 Mixed standard solution: Accurately measure 0.5 mL～10 mL stock standard solution(4.10) respectively into a 10 mL umber volumetric flask, dilute with methanol to 10 mL and mix to homogeneity. The concentration of the solution is 10 μg/mL. The solutions should be stored at −18 ℃ in dark for more than 6 months.

4.12 Standard working solution: Accurately measure an appropriate volume of mixed standard solution(4.11), and dilute to suitable concentration with methanol-water(80+20) just before use.

4.13 Stock internal standard solution: Accurately weigh 10 mg (accurate to 0.1 mg) internal standard, dissolve in 50 mL methanol and mix to homogeneity. The concentration of the solutions is 200 μg/mL. The solutions should be stored below −18 ℃ in dark for more than 12 months.

4.14 Internal standard working solution: Accurately measure 0.5 mL～10 mL internal stock standard solution (4.13) respectively into a 10 mL umber volumetric flask, dilute with methanol to 10 mL and mix to homogeneity. The concentration of the solution is 10 μg/mL. The solutions should be stored at −18 ℃ in dark for more than 6 months.

4.15 0.45 μm filter.

5 Apparatus and equipment

5.1 Liquid chromatography-mass spectrograph, equipped with electrospray ion source.

5.2 Gel permeation chromatography with UV detector(fixed wavelength: 254 nm).

5.3 Vortex shaker.

5.4 Rotary vacuum evaporator.

5.5 Homogenizer.

5.6 Centrifuge: the max rotate speed not less than 3 000 r/min.

6 Sample preparation

6.1 Animal muscle, liver and kidney

About 0.5 kg representative edible samples then grined and blended by a tissue blender to produce homogenous samples, divided into equal portions. Place in clean sample containers, seal and label. The test samples should be stored below −18 ℃ and kept away from light.

6.2 Egg

About 0.5 kg representative samples should be taken from all samples. Discard the eggshells and homogenize to produce homogenous samples. Then divide into equal portions and place in clean sample containers. After being sealed and labeled, the test samples should be stored below −18 ℃ and kept away from light.

7 Procedure

7.1 Extraction

For egg and animal muscle sample, weigh 5 g (accurate to 0.01 g) of test sample, for animal liver and kidney sample, weigh 2.5 g (accurate to 0.01 g) of test sample into a 50 mL centrifuge tube, add 50 μL internal standard working solution and votexed for 30 s. Add 5 g anhydrous sodium sulfate and 25 mL ethyl acetate, homogenize for 1 min at 10 000 r/min, and centrifuge for 10 min at 3 000 r/min. Accurately transfer 5 mL extraction for liver, kidney and muscle sample, 10 mL extraction for egg sample into 10 mL glass tube. Evaporate the exactions to dryness at 45 ℃ under nitrogen flow. Dissovle the residues with 10 mL cyclohexane-ethyl acetate (4.7) for further GPC clean up.

7.2 Clean up

7.2.1 Conditions of GPC

a) Column: Packed with 22 g S-X3 Bio-Breads, 200 mm × 20 mm(i.d.), or equivalent;

b) Mobile phase: Cyclohexane-ethyl acetate (50 + 50, *V*/*V*), flow rate: 5.0 mL/min;

c) Injection volume: 5 mL;

d) Clean-up fraction: The first fraction of 0 min～7.5 min was discarded; the second fraction of 7.5 min～12.5 min was collected.

7.2.2 Cleanup procedure

Clean up the ready solution with GPC in above conditions. Evaporate the collection to dryness and the residues are dissolved with 1 mL methanol-water (4.8) and vortex to homogeneity. After being filtrated with 0.45 μm filter, the final solution is ready for analysis by HPLC-MS/MS.

7.3 Determination by HPLC-MS/MS

7.3.1 HPLC conditions

a) Column: C_{18} 150 mm × 2.1 mm (i.d.), 5 μm particle size or equivalent;

b) Mobile phase: 0.1% acetic acid solution-acetontrile (45 + 55, *V*/*V*);

c) Flow rate: 200 μL/min;

d) Column temperature: 30 ℃;

e) Injection volume: 10 μL.

7.3.2 MS conditions

a) Ionization mode: ESI-;

b) Scan mode: MRM;

c) Other MS conditions refers to Annex A.

7.3.3 Determination

7.3.3.1 Quantitative analysis

According to the approximate concentration of analyte in the test sample solution, select the standard working solution with similar responses to that of sample solution. The responses of the analyte in the standard working solution and the sample solution should be within the linear range of the instrument detection. The mixed standard working solution and the sample solution should be injected alternatively with equal injection volume. Under the above operating condition, the reference retention times of toltrazuril sulfoxide, toltrazuril sulfone, diclazuril, toltrazuril and toltrazuril -D_3 are about

4.73 min, 7.81 min, 11.02 min, 13.61 min and 13.53 min respectively. The chromatogram of the standard refers to Annex B.

7.3.3.2 Qualitative analysis

For toltrazuril sulfoxide and diclazuril, because of their qualification ions include one precurdor ion and two daughter ions, qualitative analysis can carry out. For the same analysis batch and the same compoud, if the variation of the relative intensity of the two daughter ions for the unkown sample and the standard working solution at the similar concentration within the range of table 1, then the corresponding analyte must be present in the sample.

Table 1—Maximum permitted tolerances for relative ion intensities while confirmation

Relative intensity/%	>50	20～50	10～20	≤10
Permitted tolerance/%	±20	±25	±30	±50

7.4 Blank test

The operation of the blank test is the same as the described in the method of determination, only without samples.

8 Calculation an expression of result

Calculation the content of residues in test sample according to formula (1)

$$X = \frac{c_s \times A \times c_i \times A_{si} \times V}{A_s \times c_{si} \times A_i \times m} \quad \cdots\cdots (1)$$

where:

X —the residue content of analyte in test sample, μg/kg;

c_s —the concentration of analyte in the standard working solution, ng/mL;

A —the peak area of analyte in the sample solution;

A_s —the peak area of analyte in the standard working solution;

c_i —the concentration of internal standard in test sample, ng/mL;

c_{si} —the concentration of internal standard in the standard working solution, ng/mL;

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

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

V —the final volume of the sample solution, mL;

m —the sample weight corresponding to the final solution, g.

9 Limit of determination and recovery

9.1 Limit of determination

For the liver and kidney, the limit of quantification for the four analytes is 20 μg/kg; for the muscle, the limit of quantification for the four analytes is 10 μg/kg; for egg, the limit of quantification for the four analytes is 1 μg/kg.

9.2 Recovery

The ranges of recovery in different atrix at three spiked levels were showed in annex C.

Annex A
(Informative)
Mass conditions[1)]

Reference MS conditions

a) Sheath gas:30 unit;

b) Auxilliar gas:8 unit;

c) Ion spay voltage in ESI-mode: −3 500 V;

d) Capillary temperature:320 ℃;

e) Source CID:10 V;

f) Width of Q1 and Q3:0.7;

g) Collision gas:Argon with high purity;

h) Collision gas pressure:1.5 mTorr;

i) Other MS operating conditions are list in table A.1.

Table A.1—Scan segment,ion pairs and collision energy of the analytes

Compound	Retention time	Ion pairs	Collision energy/eV
Toltrazuril sulfoxide	4.73	440.1/371.1[a] 440.1/383.1	18 14
Toltrazuril sulfone	7.81	456.0/456.0[a]	0
Diclazuril	11.02	404.1/334.0[a] 404.1/335.0	19 19
Toltrazuril	13.53	424.0/424.0[a]	0
Toltrazuril-D_3	13.61	427.0/427.0[a]	0
Note: The different MS equipment,the parameters may be different,and the MS parameters should be optimiazed before analysis.			
[a] The quantification ion pair.			

1) Non-commercial statement:the reference mass parameters in Annex A are accomplished by Thermo TSQ Quantum Ultra AM LC/MS/MS,the equipment and its type involved in the standard method is only for reference and not related to any commercial aim,and the analysts are encouraged to use equipment of different corporation or different type.

Annex B
（Informative）
MRM chromatogram of standard

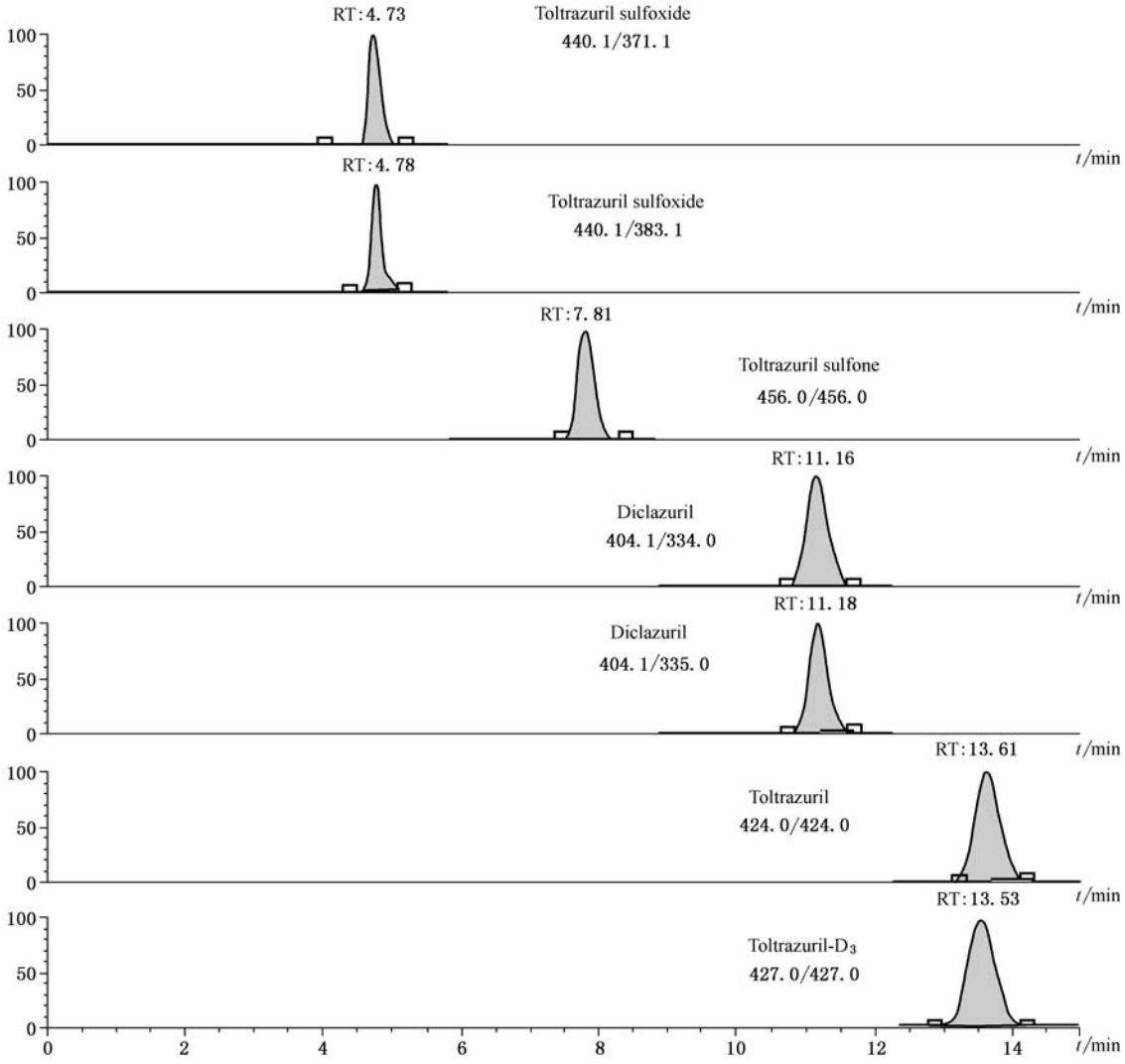

Figure B. 1—MRM chromatogram of standard working solution

Annex C
(Informative)
Ranges of recovery

Table C.1—**Recoveries of toltrazuril sulfoxide, toltrazuril sulfone, diclazuril and toltrazuril residues**

Matrices	Spiked level/(μg/kg)	Recoveries/%			
		Toltrazuril sulfoxide	Toltrazuril sulfone	Diclazuril	Toltrazuril
Egg	1	87.0～101	82.0～102	84.0～105	87.0～106
	10	88.8～114	85.2～105	90.1～108	86.5～109
	50	86.6～110	87.2～107	89.0～108	86.0～105
Chicken	10	78.9～91.3	90.0～104	96.0～109	91.0～106
	100	81.4～103	88.2～101	90.8～110	96.1～108
	500	80.5～104	87.0～109	85.0～98.5	88.5～102
Chicken liver	20	80.5～93.5	92.0～107	92.5～106	93.5～108
	100	81.8～104	88.6～102	91.2～104	96.5～108
	600(3 000)[a]	80.2～101	85.0～107	83.2～96.4	86.6～99.6
Chicken kidney	20	82.5～95.5	90.5～104	93.0～104	91.0～107
	100	83.4～108	86.2～103	92.6～105	92.5～105
	400(1 000)[a]	86.4～99.6	82.8～106	80.8～94.0	84.4～96.8
Pork	10	81.0～94.1	93.1～105	95.7～105	98.7～109
	100	84.0～106	90.2～103	92.9～103	98.3～108
	500	85.0～104	91.5～107	89.0～102	92.5～105
Swine liver	20	88.5～107	83.5～102	83.5～101	84.5～102
	100	86.1～99.7	85.7～99.3	85.0～103	82.0～103
	500(3 000)[a]	85.0～107	90.8～107	83.6～107	86.0～108
Swine kidney	20	93.0～102	88.0～105	87.5～105	92.5～105
	100	92.4～106	94.2～104	94.3～109	91.7～106
	250(1 000)[a]	91.2～104	88.4～106	86.8～107	91.2～107
Rabbit muscle	10	84.3～98.0	91.4～104	94.2～105	86.3～102
	100	84.1～102	91.1～103	93.0～104	98.5～108
	500	82.5～108	86.0～108	88.5～102	91.5～105
Rabbit liver	20	91.0～105	86.5～104	86.0～104	86.5～104
	100	89.7～103	89.8～109	87.4～105	90.1～109
	500(3 000)[a]	96.4～105	92.8～104	90.6～103	90.8～106
Rabbit kidney	20	94.0～105	91.0～108	88.5～108	91.5～105
	100	89.8～106	96.7～107	89.4～105	88.2～107
	250(1 000)[a]	91.2～101	88.8～107	87.2～107	92.0～107

[a] The content in bracket was the level of diclazuril.

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