

SN

中华人民共和国出入境检验检疫行业标准

SN/T 0533—2016
代替 SN 0287—1993、SN 0533—1996

出口水果中乙氧喹啉残留量检测方法

Method for the determination of ethoxyquin residue in fruits for export

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中 华 人 民 共 和 国
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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 0287—1993《出口水果中乙氧喹残留量检验方法 液相色谱法》和 SN 0533—1996《出口水果中乙氧三甲喹啉残留量检验方法》，与 SN 0287—1993、SN 0533—1996 相比，除编辑性修改外主要技术变化如下：

- 变更标准名称；
- 采用液相色谱-荧光检测法和液相色谱-质谱/质谱法，代替原有荧光分光光度法和液相色谱-紫外检测法；
- 将检测范围的适用基质扩大到苹果、梨、桃、李、柑橘；
- 修改了样品前处理方法；
- 降低了方法测定低限。

请注意本文件的某些内容可能涉及专利。本文件的发布机构不承担识别这些专利的责任。

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：浙江省检验检疫科学技术研究院。

本标准主要起草人：黄超群、谢文、楼成杰、陈玲玲、张文华、童赞恺、吴娟。

本标准所代替标准的历次版本发布情况为：

- SN 0287—1993；
- SN 0533—1996。

出口水果中乙氧喹啉残留量检测方法

1 范围

本标准规定了出口水果中乙氧喹啉测定的高效液相色谱和液相色谱-质谱/质谱检测方法。
本标准适用于出口苹果、梨、桃、李、柑橘中乙氧喹啉的测定。

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

第一法 高效液相色谱法

3 方法提要

不同水果采用相应方法制备试样,以正己烷为溶剂,振荡提取、浓缩并定容后,高效液相色谱-荧光法测定,外标法定量。

4 试剂和材料

除特殊注明外,所用试剂均为色谱纯,所用水为符合 GB/T 6682 规定的一级水。

4.1 乙腈。

4.2 甲醇。

4.3 正己烷。

4.4 维生素 C:分析纯。

4.5 标准物质:乙氧喹啉, $C_{14}H_{19}NO$, CAS 登录号 91-53-2,纯度大于 97.5%。

4.6 标准储备溶液:准确称取适量标准物质(4.5),用甲醇溶解并定容,配制成溶液浓度为 200 $\mu\text{g/mL}$ 的标准储备液,0 $^{\circ}\text{C}$ ~4 $^{\circ}\text{C}$ 保存,有效期 6 个月。

4.7 标准工作溶液:根据需要,将标准储备液(4.6)用甲醇稀释成适当浓度的标准工作溶液,0 $^{\circ}\text{C}$ ~4 $^{\circ}\text{C}$ 保存。

4.8 微孔滤膜: $\leq 0.45 \mu\text{m}$,有机相。

5 仪器和设备

5.1 高效液相色谱仪:配有荧光检测器。

5.2 组织捣碎机。

5.3 分析天平:感量 0.000 1 g 和 0.01 g。

5.4 涡旋混合器。

5.5 振荡器。

5.6 台式离心机:转速不低于 4 000 r/min。

5.7 氮吹仪。

6 试样制备与保存

6.1 试样制备

6.1.1 苹果、梨、桃、李

取代表性样品约 500 g,将其可食用部分切碎(不可用水洗涤)后,按 1 g 维生素 C/100 g 样品比例,加入一定量维生素 C,用捣碎机将样品加工成浆状。混匀,装入洁净的盛样容器中,密封并标明标记。

6.1.2 柑橘

取代表性样品约 500 g,将其可食用部分切碎(不可用水洗涤)后,用捣碎机将样品加工成浆状。混匀,装入洁净的盛样容器中,密封并标明标记。

6.2 试样保存

试样于-18℃以下冷冻保存。在制样的操作过程中,应防止样品受到污染或发生残留物含量的变化。

7 测定步骤

7.1 样品处理

准确称取 10 g 试样(精确至 0.01 g),置于 50 mL 塑料离心管中,加入 20 mL 正己烷,涡旋混匀,振荡提取 15 min 后,4 000 r/min 离心 3 min。准确移取 4.0 mL 上清液至 10 mL 离心管中,缓慢氮吹至干,加入 1.0 mL 甲醇,涡旋,经滤膜过滤,供液相色谱仪测定。

7.2 测定

7.2.1 液相色谱条件

液相色谱条件如下:

- 色谱柱: C_{18} , 250 mm \times 4.6 mm(内径), 5 μ m, 或相当者;
- 流动相: 乙腈-水, 梯度洗脱程序见表 1;

表 1 液相色谱法梯度洗脱程序

时间 min	乙腈 %	水 %
0.00	20	80
2.00	20	80
6.00	95	5
8.00	95	5
12.00	20	80
16.00	20	80

- c) 检测波长:激发波长 360 nm,发射波长 435 nm;
- d) 柱温:25 ℃;
- e) 流速:1.0 mL/min;
- f) 进样量:20 μL。

7.2.2 液相色谱测定

按 7.2.1 液相色谱条件测定标准工作溶液和样液,以外标曲线法计算样液中的乙氧喹啉含量。如果样液中乙氧喹啉的含量超出标准曲线范围,应用甲醇稀释后再进行分析。在上述色谱条件下,乙氧喹啉的参考保留时间约为 10.8 min。标准溶液色谱图参见附录 A 中图 A.1。

7.3 空白试验

除不加试样外,均按上述操作步骤进行。

8 结果计算和表述

用色谱数据处理机或按式(1)计算试样中乙氧喹啉的含量:

$$X_i = \frac{c_i \times V \times 1\,000}{m \times 1\,000} \dots\dots\dots (1)$$

式中:

- X_i ——试样中乙氧喹啉的含量,单位为毫克每千克(mg/kg);
 - c_i ——从标准曲线上得到的待测物质的溶液浓度,单位为微克每毫升(μg/mL);
 - V ——样液最终定容体积,单位为毫升(mL);
 - m ——最终样液所代表的试样质量,单位为克(g)。
- 计算结果应扣除空白值。

9 测定低限和回收率

9.1 测定低限

本方法对于苹果、梨、桃、李和柑橘中乙氧喹啉的测定低限均为 0.05 mg/kg。

9.2 回收率

本方法回收率试验数据见表 2。

表 2 回收率试验数据(n=6)

添加浓度 mg/kg	回收率范围 %				
	苹果	梨	桃	李	柑橘
0.05	90.0~108.0	84.0~98.0	84.0~102.0	76.0~92.0	84.0~94.0
0.10	—	—	84.0~100.0	80.0~93.0	84.0~92.0
1.0	79.4~97.5	89.0~107.0	82.1~101.0	79.1~85.3	89.5~95.9
3.0	90.3~104.0	96.7~107.0	—	—	—
6.0	92.7~101.5	93.7~105.8	—	—	—

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第二法 液相色谱-质谱/质谱法

10 方法提要

不同水果采用相应方法制备试样,以正己烷为溶剂,振荡提取、浓缩并定容后,液相色谱-质谱/质谱法测定,外标法定量。

11 试剂和材料

所用试剂和材料均同 4.1~4.8。

12 仪器和设备

除特殊注明外,所用仪器和设备均同 5.2~5.7。

12.1 液相色谱-质谱/质谱仪,配电喷雾离子源。

13 试样制备与保存

试样制备与保存同 6.1 和 6.2。

14 测定步骤

14.1 样品处理

准确称取 10 g 试样(精确至 0.01 g),置于 50 mL 塑料离心管中,加入 20 mL 正己烷,涡旋混匀,振荡提取 15 min 后,4 000 r/min 离心 3 min。准确移取 1.0 mL 上清液至 10 mL 离心管中,缓慢氮吹至干,加入 5.0 mL 甲醇,涡旋,经滤膜过滤,供液相色谱-质谱/质谱仪测定。

14.2 测定

14.2.1 液相色谱-质谱/质谱条件

液相色谱-质谱/质谱条件如下:

- a) 色谱柱: C₁₈, 150 mm×4.6 mm(内径), 5 μm, 或相当者;
- b) 流动相: 乙腈-水, 梯度洗脱程序见表 3;

表 3 液相色谱-质谱/质谱法梯度洗脱程序

时间 min	乙腈 %	水 %
0.00	20	80
2.00	20	80
4.00	95	5

表 3 (续)

时间 min	乙腈 %	水 %
9.00	95	5
10.00	20	80
15.00	20	80

- c) 柱温:20 ℃;
- d) 流速:0.4 mL/min;
- e) 进样量:10 μL;
- f) 离子源:电喷雾离子源;
- g) 扫描方式:正离子扫描;
- h) 检测方式:多反应监测;
- i) 监测离子对见附录 B 中表 B.1。

14.2.2 定量测定

按 14.2.1 仪器条件测定标准工作溶液和样液,以外标曲线法计算样液中的乙氧喹啉含量。如果样液中乙氧喹啉的含量超出标准曲线范围,应用甲醇稀释后再进行分析。在上述条件下,乙氧喹啉的参考保留时间约为 10.6 min。标准溶液多反应监测色谱图参见附录 A 中图 A.2。

14.2.3 定性测定

按照上述仪器条件测定样液和标准工作溶液,如果样液与标准工作液中待测物质色谱峰相对保留时间偏差在±2.5%范围内;定性离子对的相对丰度与浓度相当的标准工作溶液的相对丰度一致,相对丰度允许误差不超过表 4 规定的范围,则可判断样品中存在相应的被测物。

表 4 相对离子丰度最大容许误差

相对离子丰度(基峰) %	50~100	20~50	10~20	<10
最大容许误差 %	±30	±30	±30	±30

14.3 空白试验

除不加试样外,均按上述操作步骤进行。

15 结果计算和表述

同第 8 章。

16 测定低限和回收率

16.1 测定低限

本方法对于苹果、梨、桃、李和柑橘中乙氧喹啉的测定低限均为 0.05 mg/kg。

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16.2 回收率

本方法回收率试验数据见表 5。

表 5 回收率试验数据($n=6$)

添加浓度 mg/kg	回收率范围 %				
	苹果	梨	桃	李	柑橘
0.05	70.0~86.0	84.0~90.0	86.0~110.0	84.0~98.0	72.0~96.0
0.10	—	—	87.0~102.0	75.0~99.0	78.0~88.0
1.0	74.6~83.7	79.3~87.4	74.8~88.7	72.3~82.5	76.0~83.0
3.0	77.0~103.0	86.0~95.7	—	—	—
6.0	73.0~94.0	84.7~100.0	—	—	—

附 录 A
(资料性附录)

高效液相色谱法和液相色谱-质谱/质谱法标准溶液色谱图

乙氧喹啉标准溶液的液相色谱图见图 A.1。

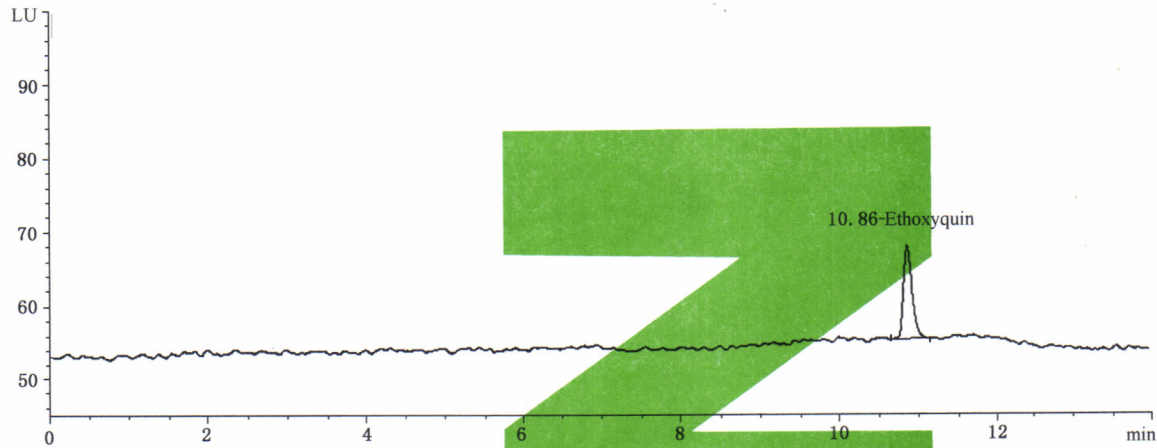


图 A.1 乙氧喹啉标准溶液的液相色谱图(0.10 $\mu\text{g/mL}$)

乙氧喹啉标准溶液的多反应监测(MRM)色谱图见图 A.2。

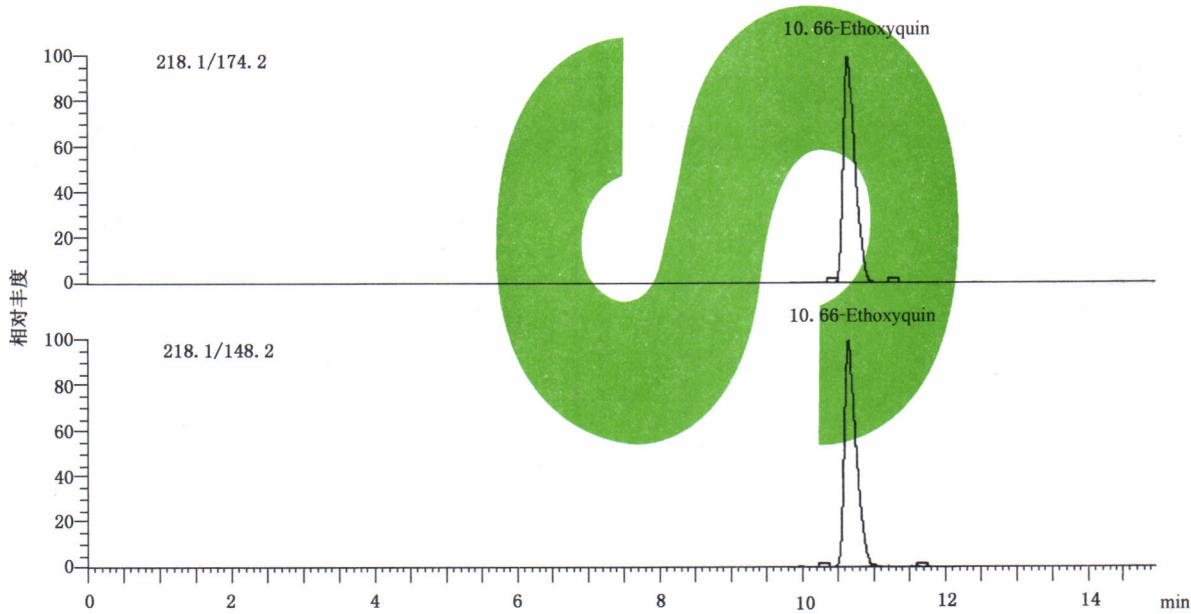


图 A.2 乙氧喹啉标准溶液的多反应监测(MRM)色谱图(0.005 $\mu\text{g/mL}$)

附 录 B
(资料性附录)

液相色谱-质谱/质谱法仪器参数与监测离子¹⁾

液相色谱-质谱/质谱法仪器参数与监测离子条件如下:

- a) 电喷雾电压:3 500 V。
- b) 离子源温度:300 ℃。
- c) 鞘气压力:50 Arb。
- d) 辅助气压力:20 Arb。
- e) 毛细管温度:270 ℃。
- f) 碰撞气压力:1.5 mTorr。
- g) 鞘气、辅助气均为高纯氮气;碰撞气为高纯氩气;使用前应调节各参数使质谱灵敏度达到检测要求。
- h) 监测离子对信息、碰撞能量等参数见表 B.1。

表 B.1 多反应监测离子对和碰撞能量

化合物	母离子 <i>m/z</i>	子离子 <i>m/z</i>	碰撞能量 V
乙氧喹啉	218.1	174.2*	30
		148.2	21
注：* 为定量离子。			

1) 非商业性声明:附录 B 所列参考质谱条件是在 TSQ Quantum ULTRA AM 液质联用仪上完成,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或型号的仪器。

Foreword

This standard was drafted in accordance with the GB/T 1.1—2009.

This standard is the revision of SN 0287—1993《Method for the determination of ethoxyquin residues in fruits for export—Liquid chromatography》and SN 0533—1996《Method for the determination of ethoxyquin residues in fruits for export》.

The differences between this standard and SN 0287—1993, SN 0533—1996 are as follows:

- Changed the name of standard;
- Adopted HPLC with fluorescence detection and LC-MS/MS method, replaced fluorescence spectrophotometry method and HPLC with ultraviolet detection;
- Extended the range of matrix to apple, pear, peach, plum and orange;
- Amended sample preparation method;
- Lowered the limit of quantitation.

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

This standard was drafted by Zhejiang Academy of Science & Technology for Inspection & Quarantine.

The main drafters of this standard are Huang Chaoqun, Xie Wen, Lou Chenjie, Chen Lingling, Zhang Wenhua, Tong Yunkai and Wu Juan.

This standard replaces the standard previously published as:

- SN 0287—1993;
- SN 0533—1996.

Method for the determination of ethoxyquin residue in fruits for export

1 Scope

This standard specifies the determination of ethoxyquin in fruits by high performance liquid chromatography and liquid chromatography-tandem mass spectrum.

This standard is applicable to the determination of ethoxyquin in apple, pear, peach, plum and orange for export.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including amendments) applies.

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

Method I HPLC Method

3 Principle

Samples were adopted different preparation method by species. The test samples are extracted with hexane. The extract is evaporated and made up to a definite volume. Determination is made by liquid chromatograph equipped with fluorescence detector, using external standard method.

4 Reagents and materials

Unless otherwise specified, all the reagents used should be chromatographic pure, “water” is distilled water.

4.1 Acetonitrile.

4.2 Methanol.

4.3 Hexane.

4.4 Vitamin C: analytically pure.

4.5 Standard: Ethoxyquin, $C_{14}H_{19}NO$, CAS NO.91-53-2, Purity $\geq 97.5\%$.

4.6 Standard stock solution: Accurately weigh appropriate standard(4.5), dissolved with methanol, the concentration of solution is 200 $\mu\text{g/mL}$, stored under $0\text{ }^{\circ}\text{C} \sim 4\text{ }^{\circ}\text{C}$. Standard stock solution is stable for 6 months.

4.7 Standard work solution: According to the requirement, diluted the standard stock solution(4.6) with methanol.

4.8 Millipore filter: $\leq 0.45\text{ }\mu\text{m}$, organic phase.

5 Apparatus and equipment

5.1 High performance liquid chromatograph, equipped with fluorescence detector.

5.2 Tissue blender.

5.3 Electronic balance: accurate to 0.000 1 g and 0.01 g.

5.4 Vortex mixer.

5.5 Oscillator.

5.6 Centrifuge (4 000 r/min) .

5.7 Nitrogen blowing instrument.

6 Preparation and storage of test sample

6.1 Preparation

6.1.1 Apple, pear, peach, plum

Take approximately weight of representative sample, added with vitamin C with the proportion of 1 g vitamin C/100 g sample. Homogenized thoroughly in a high speed blender, and then are placed in a clean container, which is sealed and labeled.

6.1.2 Orange

Take approximately weight of representative sample. Homogenized thoroughly in a high speed blender, and then are placed in a clean container, which is sealed and labeled.

6.2 Storage

The test samples should be stored below $-18\text{ }^{\circ}\text{C}$. In the course of sampling and sample preparation, precaution must be taken to avoid contamination or any factors which may cause the change of residue content.

7 Procedure

7.1 Sample treatment

10 grams of sample was weighted into a polypropylene tube, then 20 mL of hexane was added. After violent shaking for 15 minutes, the sample solution was centrifuged at 4 000 r/min for 3 minutes. 4.0 mL of the upper solution was transferred into a 10 mL centrifuge tube, evaporated under nitrogen blow. Dissolve the residue and dilute exactly to 1.0 mL with methanol for LC.

7.2 Determination

7.2.1 HPLC operative condition

HPLC operating condition is as follows:

- a) Column: C_{18} , 250 mm \times 4.6 mm (i.d), 5 μm , or the equivalent;
- b) Mobile phase: acetonitrile + water, for gradient elute condition, see table 1;
- c) Wavelength: ex 360 nm, em 435 nm;
- d) Column temperature: 25 $^{\circ}\text{C}$;
- e) Flow: 1.0 mL/min;
- f) Injection volume: 20 μL .

Table 1 Gradient elute condition

Time min	Acetonitrile %	Water %
0.00	20	80
2.00	20	80
6.00	95	5
8.00	95	5
12.00	20	80
16.00	20	80

7.2.2 Analysis by LC

Analyze working standard solutions and samples with external standard method by HPLC, using conditions established in sec.7.2.1. If the concentration of ethoxyquin exceeds the linear range of the calibration curve, the final solution should be diluted with methanol and reanalyzed. Under the above chromatographic conditions, the retention time of chromatographic peak of ethoxyquin is about 10.8 min. For the standard chromatogram refer to fig A.1 in Appendix A.

7.3 Blank test

The operation of the blank test is the same as that described in the method of determination, but with omission of sample addition.

8 Calculation and expression of the result

Calculate the content of ethoxyquin in the test sample according to the following formula:

$$X_i = \frac{c_i \times V \times 1\,000}{m \times 1\,000} \dots\dots\dots (1)$$

Where:

X_i —the content of ethoxyquin in the test sample, mg/kg;

c_i —the concentration of ethoxyquin in sample solution calculated by standard curve, $\mu\text{g/mL}$;

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

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

9 Limit of quantitation and recovery

9.1 Limit of quantitation

The limits of quantitation for ethoxyquin in apple, pear, peach, plum and orange are 0.05 mg/kg.

9.2 Recovery

The results of recovery test are listed in Table 2.

Table 2 Results of recovery test ($n=6$)

Spike level mg/kg	Recovery range %				
	Apple	Pear	Peach	Plum	Orange
0.05	90.0~108.0	84.0~98.0	84.0~102.0	76.0~92.0	84.0~94.0
0.10	—	—	84.0~100.0	80.0~93.0	84.0~92.0
1.0	79.4~97.5	89.0~107.0	82.1~101.0	79.1~85.3	89.5~95.9
3.0	90.3~104.0	96.7~107.0	—	—	—
6.0	92.7~101.5	93.7~105.8	—	—	—

Method II LC-MS/MS Method

10 Principle

Samples were adopted different preparation method by species. The test samples are extracted with hexane. The extract is evaporated and made up to a definite volume. Determination is made by liquid chromatography tandem mass spectrometry, using external standard method.

11 Reagents and materials

All of reagents and materials are the same as section 4.1 to 4.8.

12 Apparatus and equipment

Unless otherwise specified, apparatus are the same as section 5.2 to 5.7.

12.1 Liquid chromatography tandem mass spectrometry, equipped with ESI.

13 Preparation and storage of test sample

Preparation and storage of test samples are the same as section 6.1 and 6.2.

14 Procedure

14.1 Sample treatment

10 grams of sample was weighted into a polypropylene tube, then 20 mL of hexane was added. After violent shaking for 15 minutes, the sample solution was centrifuged at 4 000 r/min for 3 minutes. 1.0 mL of the upper solution was transferred into a 10 mL centrifuge tube, evaporated under nitrogen blow. Dissolve the residue and dilute exactly to 5.0 mL with methanol for LC-MS/MS.

14.2 Determination

14.2.1 LC-MS/MS operating condition

LC-MS/MS operating condition is as follows:

- a) Column: C_{18} , 150 mm \times 4.6 mm (i.d.), 5 μ m, or the equivalent;
- b) Mobile phase: acetonitrile + water, for gradient elute condition, see table 3;

Table 3 Gradient elute condition

Time min	Acetonitrile %	Water %
0.00	20	80
2.00	20	80
4.00	95	5
9.00	95	5
10.00	20	80
15.00	20	80

- c) Column temperature: 20 $^{\circ}$ C ;

- d) Flow: 0.4 mL/min;
- e) Injection volume: 10 μ L;
- f) ion source: ESI;
- g) Scan mode: positive;
- h) Detect mode: Multiple reaction monitoring(MRM);
- i) Ion-pairs are listed in table B.1 in Appendix B.

14.2.2 Quantitation criteria

Analyze working standard solutions and samples with external standard method by LC-MS/MS, using conditions established in section 14.2.1. If the concentration of ethoxyquin exceeds the linear range of the calibration curve, the final solution should be diluted with methanol and reanalyzed. Under the above conditions, the retention time of chromatographic peak of ethoxyquin is about 10.6 min. For the MRM chromatogram refer to fig A.2 in Appendix A.

14.2.3 Qualification criteria

The relative intensities of the detected ions, expressed as a percentage of the intensity of the most intense ion or transition, shall correspond to those of the calibration standard, either from calibration standard solutions or from spiked samples, at comparable concentrations, measured under the same conditions, within the following tolerances.

Table 4 Maximum permitted tolerances for relative ion intensities using a range of mass spectrometric techniques

Relative intensity (base peak) %	50~100	20~50	10~20	<10
Maximum permitted tolerances %	± 30	± 30	± 30	± 30

14.3 Blank test

The operation of the blank test is the same as that described in the method of determination, but with omission of sample addition.

15 Calculation and expression of the result

Same as section 8.

16 Limit of quantitation and recovery

16.1 Limit of quantitation

The limits of quantitation for ethoxyquin in apple, pear, peach, plum and orange are 0.05 mg/kg.

16.2 Recovery

The results of recovery test are listed in Table 5.

Table 5 Results of recovery test($n=6$)

Spike level mg/kg	Recovery range %				
	Apple	Pear	Peach	Plum	Orange
0.05	70.0~86.0	84.0~90.0	86.0~110.0	84.0~98.0	72.0~96.0
0.10	—	—	87.0~102.0	75.0~99.0	78.0~88.0
1.0	74.6~83.7	79.3~87.4	74.8~88.7	72.3~82.5	76.0~83.0
3.0	77.0~103.0	86.0~95.7	—	—	—
6.0	73.0~94.0	84.7~100.0	—	—	—

Annex A
(Informative annex)
Chromatograms of ethoxyquin

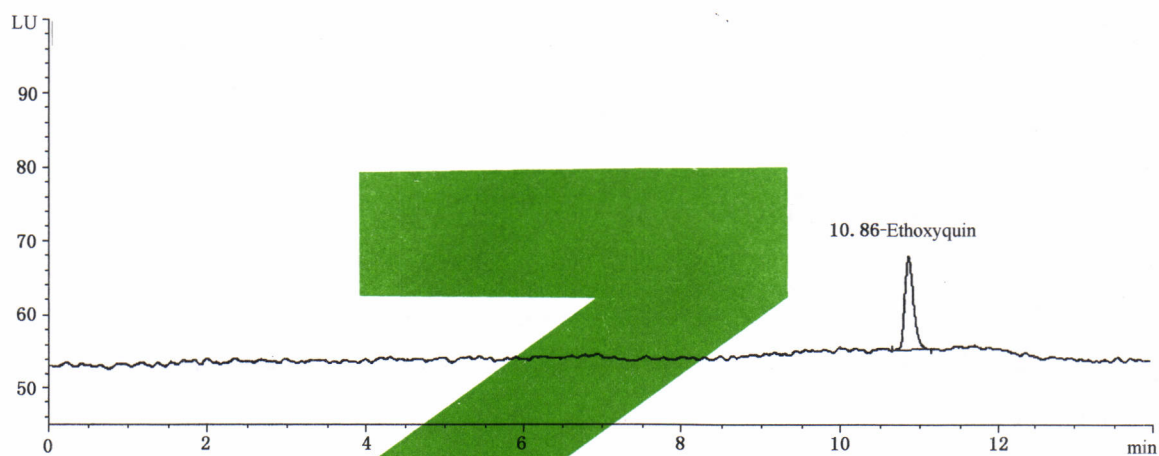


Figure A.1 Chromatogram of ethoxyquin(0.10 µg/mL)

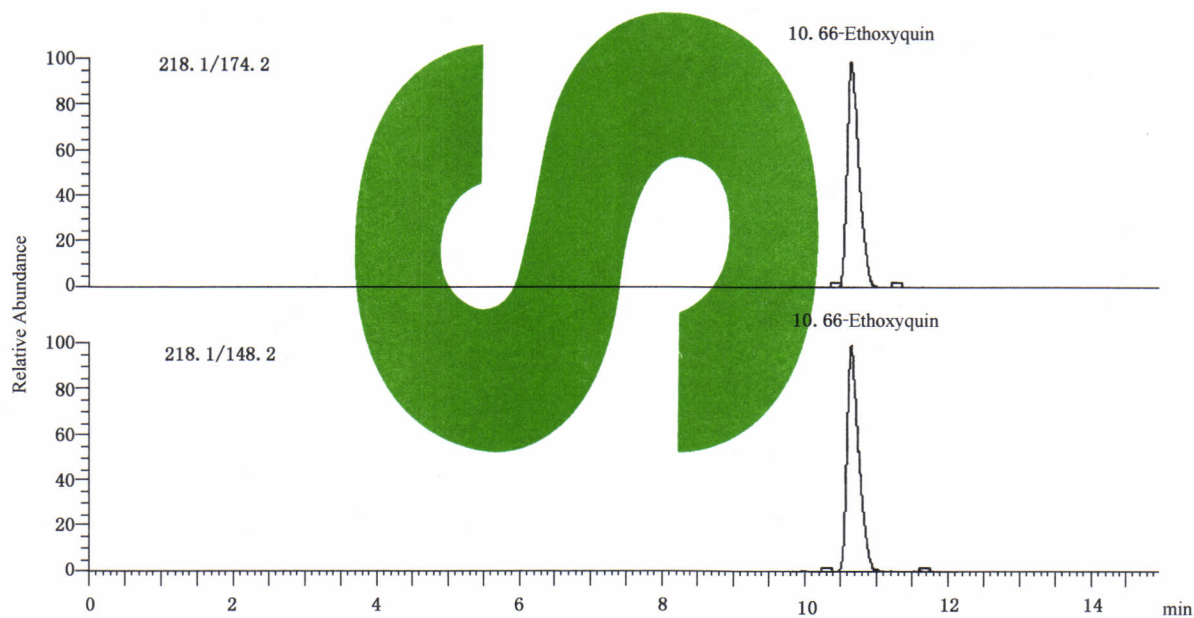


Figure A.2 MRM chromatogram of ethoxyquin(0.005 µg/mL)

Annex B
(Informative annex)
Reference mass parameters¹⁾

Reference mass parameters is as follows:

- a) Spray voltage;3 500 V.
- b) Vaporizer temperature;300 ℃.
- c) Sheath gas pressure;50 Arb.
- d) Aux gas pressure;20 Arb.
- e) Capillary temperature;270 ℃.
- f) Collision cell pressure;1.5 mTorr.
- g) Sheath gas,auxiliary gas;high-purity nitrogen;collision gas,high-purity argon.
- h) Ion-pairs and collision energy are listed in Table B.1.

Table B.1 Main mass parameters

Compound	Precursor ion <i>m/z</i>	Daughter ions <i>m/z</i>	Collision energy V
Ethoxyquin	218.1	174.2 *	30
		148.2	21
* : Quantitative ion.			

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

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