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中华人民共和国出入境检验检疫行业标准

SN/T 4046—2014

出口食品中噻虫啉残留量的测定

Determination of thiacloprid residues in food for export

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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、南京师范大学、上海交通大学。

本标准主要起草人：倪昕路、王传现、赵波、陈昌云、王正武、黄帆、宋业萍。

出口食品中噻虫啉残留量的测定

1 范围

本标准规定了出口农产品中噻虫啉残留量的高效液相色谱测定方法。

本标准适用于西红柿、大白菜、萝卜、马铃薯、苹果、香蕉、大豆、韭菜、洋葱、猪肉、鸡肉、猪肝、油脂、蜂蜜、大米、鸡蛋、牛奶、茶叶中噻虫啉残留量的测定。

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实验方法

3 方法提要

样品中的噻虫啉经乙酸乙酯或乙腈提取，SCX 强阳离子交换固相萃取柱净化，用高效液相色谱仪测定，外标法定量。

4 试剂和材料

除另有规定外，所用试剂均为分析纯，水为符合 GB/T 6682 规定的一级水。

4.1 乙腈：色谱纯。

4.2 甲醇：色谱纯。

4.3 石油醚：色谱纯。

4.4 乙酸乙酯：色谱纯。

4.5 正己烷：色谱纯。

4.6 氯化钠。

4.7 无水硫酸钠：用前在 650 ℃灼烧 4 h，贮于干燥器中，冷却后备用。

4.8 饱和氯化钠溶液。

4.9 石油醚-乙酸乙酯溶液(85+15，体积比)：取 850 mL 石油醚，加入 150 mL 乙酸乙酯，摇匀备用。

4.10 石油醚-乙酸乙酯溶液(30+70，体积比)：取 300 mL 石油醚，加入 700 mL 乙酸乙酯，摇匀备用。

4.11 乙腈-水溶液(30+70，体积比)：取 300 mL 乙腈，加入 700 mL 水，摇匀备用。

4.12 噻虫啉标准品(thiacloprid，$C_{10}H_9ClN_4S$，CAS 编号 111988-49-9)，纯度大于或等于 95%。

4.13 标准贮备液：准确称取适量标准品(精确至 0.000 1 g)，用甲醇溶解配制成浓度为 1 mg/mL 标准贮备液。置于−18 ℃下保存，有效期为 12 个月。

4.14 标准工作液：根据实际使用需要，用甲醇将标准贮备液逐级稀释成适当浓度的标准工作液。置于 4 ℃保存，有效期为 3 个月。

4.15 强阳离子交换固相萃取小柱：SCX，0.5 g，6 mL，或相当者。

4.16 微孔滤膜：0.22 μm，有机相型。

5 仪器和设备

5.1 高效液相色谱仪：配有紫外检测器。
5.2 分析天平：感量 0.000 1 g 和 0.01 g。
5.3 离心机：4 000 r/min。
5.4 组织捣碎机。
5.5 均质器。
5.6 固相萃取装置。
5.7 氮吹仪。
5.8 涡旋混匀器：转速可调。

6 试样制备与保存

6.1 一般要求

在制样的操作过程中，应防止样品污染或发生待测物含量的变化。

6.2 水果、蔬菜类

称取苹果、大白菜等水果或蔬菜样品 500 g，不可用水洗涤，取其可食用部分切碎后，用匀浆机将样品加工成浆状，混匀，分装入洁净的容器内，密闭并标明标记。于－18 ℃下冷冻保存。

6.3 鸡肉、猪肉、猪肝、鸡蛋等

称取样品 500 g，将其切碎后（鸡蛋去壳），用匀浆机将样品加工成浆状，混匀，分装入洁净容器内，密闭并标明标记。于－18 ℃下冷冻保存。

6.4 蜂蜜

取有代表性样品约 500 g。对于无结晶的样品将其搅拌均匀；对于有结晶析出的样品，在密闭情况下，将样品置于不超过 60 ℃的水浴中温热，待样品全部融化后搅拌均匀，迅速冷却至室温。在融化时必须注意防止水分挥发。装入洁净容器内，密封并标明标记。室温下保存。

6.5 牛奶、油脂等液体或可融为液态的样品

取有代表性样品约 500 mL。混匀后装入洁净容器作为试样，密封并标明标记。于－18 ℃下冷冻保存。

6.6 大米、茶叶、大豆等干样品

取代表性样品约 500 g，用粉碎机粉碎，混匀，装入洁净的容器内，密封，标明标记，室温下保存。

7 测定步骤

7.1 提取

7.1.1 水果、蔬菜类

准确称取 2 g（精确到 0.01 g）试样于 10 mL 塑料离心管中，加入 2 mL 饱和氯化钠，混匀。再加入

4 mL 乙酸乙酯，用涡旋混合器混匀后，超声提取 30 min，于 4 000 r/min 转速下离心 10 min 后取上清液于氮吹管。残渣按上述过程重复提取一次，合并两次提取液，将提取液于氮吹仪 45 ℃浓缩至近干。用 4 mL 石油醚-乙酸乙酯混合溶剂(85+15，体积比)溶解残渣。

7.1.2 鸡肉、猪肉、猪肝、蜂蜜、大米、鸡蛋、牛奶、大豆、茶叶等

准确称取 2 g(精确到 0.01 g)试样于 10 mL 塑料离心管中，加入 2 mL 水，混匀。再加入 4 mL 乙酸乙酯，用涡旋混合器混匀后，超声提取 30 min，于 4 000 r/min 转速下离心 10 min 后取上清液于氮吹管。残渣按上述过程重复提取一次，合并两次提取液，将提取液于氮吹仪 45℃ 浓缩至近干。用 4 mL 甲醇溶解残渣，加入 3 mL 正己烷，用涡旋混合器混匀 5 min，于 4 000 r/min 转速下离心 5 min 后将上层液体弃去，下层液体按上述过程重复萃取一次，合并萃取液体，于氮吹仪 45 ℃浓缩至近干。用 4 mL 石油醚-乙酸乙酯混合溶剂(85+15，体积比)溶解残渣。

7.1.3 油脂等脂肪类样品

准确称取 2 g(精确到 0.01 g)试样于 10 mL 塑料离心管中，加入 4 mL 乙腈。置于涡旋混合器混合 5 min 后，超声提取 30 min，于 4 000 r/min 转速下离心 10 min 后取上清液于氮吹管。残渣再用 2 mL 乙腈按上述过程重复提取一次，合并两次提取液。加入 3 mL 正己烷，用涡旋混合器混匀 5 min，与 4 000 r/min 转速下离心 5 min 后将上层液体弃去，下层液体上述过程重复萃取一次，合并萃取液体，于氮吹仪 45 ℃浓缩至近干。用 4 mL 石油醚-乙酸乙酯混合溶剂(85+15，体积比)溶解残渣。

7.2 净化

取 SCX 小柱依次用 5 mL 甲醇和 3 mL 水活化，抽干后，将试样溶液移入小柱，抽干弃去流出液。再用 10 mL 石油醚＋乙酸乙酯(30＋70，体积比)洗脱，收集洗脱液于氮吹管，于氮吹仪 50 ℃下浓缩至近干，用流动相乙腈-水溶液(30＋70，体积比)定容至 1 mL，经 0.22 μm 滤膜过滤后，用于 HPLC 分析。

7.3 测定

7.3.1 参考液相色谱质谱条件

液相色谱质谱条件如下：

a) 色谱柱：C_{18}柱，柱长 150 mm，内径 4.6 mm，粒径 5.0 μm，或相当者。
b) 流动相：乙腈-水(30＋70，体积比)。
c) 流速：1.0 mL/min。
d) 进样量：20 μL。
e) 柱温：室温。
f) 检测波长：240 nm。
g) 质谱参数条件：参见附录 A。

7.3.2 定量测定

根据样液中噻虫啉含量情况，选定峰面积相近的标准工作液，标准工作液和待测样液中噻虫啉的响应值均应在仪器检测线性范围内。对标准工作液和样液等体积参插进样测定，外标法定量。在上述色谱条件下，噻虫啉的保留时间约为 7.2 min，其高效液相色谱谱图参见图 B.1。

7.3.3 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 1 规定的范围，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

7.4　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

用色谱数据处理器或按式(1)计算试样中待测物的含量，计算结果需扣除空白值：

$$X=\frac{A\times c\times V}{A_s\times m} \qquad \cdots\cdots(1)$$

式中：

X ——试样中噻虫啉的含量，单位为毫克每千克(mg/kg)；

A ——样液中噻虫啉的色谱峰面积；

c ——标准溶液中噻虫啉的浓度，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)；

A_s——标准溶液中噻虫啉的色谱峰面积；

m ——最终样液所代表的样品质量，单位为克(g)。

9　测定低限和回收率

9.1　测定低限

本方法的测定低限为 0.01 mg/kg。

9.2　回收率

本方法对西红柿、大白菜、萝卜、马铃薯、苹果、香蕉、大豆、韭菜、洋葱、猪肉、鸡肉、猪肝、油脂、蜂蜜、大米、鸡蛋、牛奶、茶叶等食品基质进行添加回收试验，噻虫啉的添加回收率参见表 C.1。

附 录 A
（资料性附录）
质谱/质谱仪参数[1)]

质谱/质谱仪参数如下：

a） 离子化模式：电喷雾电离正离子模式（ESI+）；

b） 质谱扫描方式：多反应监测（MRM）；

c） 分辨率：单位质量分辨率；

d） 其他参考质谱条件参见表 A.1。

表 A.1 多反应监测条件

化合物名称	母离子 m/z	子离子 m/z	碰撞气能量/V	去簇电压/V
噻虫啉	253.0	126.0（定量离子）	29	67
		186.0	19	66

1） 非商业性声明：附录 A 所列参数是在 API 5000 质谱/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）
噻虫啉标准品的液相色谱图

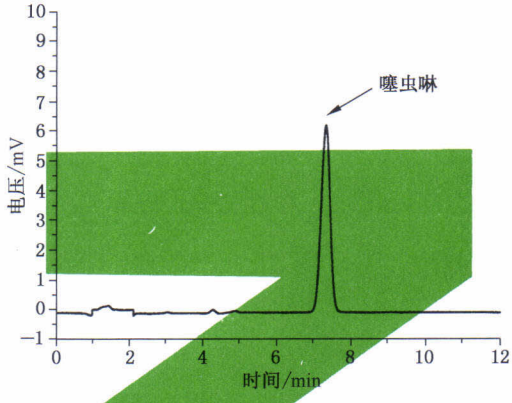

图 B.1 噻虫啉标准品(0.2 mg/L)的色谱图

附 录 C
（资料性附录）
添加回收率

表 C.1 农产品中噻虫啉的添加回收率（$n=10$）

基质名称	添加水平/(mg/kg)	回收率范围/%	基质名称	添加水平/(mg/kg)	回收率范围/%
西红柿	0.01	77.5～99.2	大米	0.01	75.9～103.6
	0.1	82.2～106.4		0.1	85.3～107.7
	5.0	93.5～101.8		2.0	86.8～103.7
大白菜	0.01	74.7～100.5	蜂蜜	0.01	77.0～97.7
	0.1	81.2～106.9		0.1	82.1～109.3
	5.0	93.6～100.5		2.0	85.5～106.1
萝卜	0.01	73.7～109.4	猪肉	0.01	78.3～105.2
	0.1	81.2～105.3		0.1	86.5～104.3
	5.0	93.6～101.8		2.0	84.5～105.6
马铃薯	0.01	74.7～105.5	鸡肉	0.01	77.3～107.1
	0.1	80.8～103.2		0.1	85.1～107.2
	2.0	86.3～104.8		2.0	82.9～107.8
苹果	0.01	76.7～103.2	猪肝	0.01	74.6～103.8
	0.1	81.7～104.8		0.1	75.2～101.6
	5.0	88.7～100.4		2.0	76.5～101.8
香蕉	0.01	76.4～105.3	油脂	0.01	75.9～102.8
	0.1	83.7～106.2		0.1	86.9～105.7
	5.0	91.9～102.2		2.0	87.5～104.4
大豆	0.01	78.4～102.4	鸡蛋	0.01	77.8～106.2
	0.1	83.7～104.1		0.1	82.1～107.9
	2.0	87.8～106.2		2.0	90.1～104.4
韭菜	0.01	74.3～104.3	牛奶	0.01	74.8～106.6
	0.1	84.0～101.0		0.1	82.2～104.3
	5.0	88.4～101.5		2.0	88.0～103.4
洋葱	0.01	78.3～104.7	茶叶	0.01	76.8～104.2
	0.1	81.6～108.9		0.1	82.7～105.6
	5.0	88.9～101.6		2.0	88.4～103.9

Foreword

This standard was drafed in according with the GB/T 1.1—2009.

This standard was proposed by and is under the jurisdiction of the Certification and Accreditation Adiministration 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, Shanghai Jiao Tong University.

This standard was mainly drafted by Ni Xinlu, Wang Chuanxian, Zhao Bo, Chen Changyun, Wang Zhengwu, Huang Fan, Song Yeping.

Note: This English version, a translation from the Chinese text, is only for reference.

Determination of thiacloprid residues in food for export

1 Scope

This standard specifies the methods of determination of thiacloprid residues in agriculure products by liquid chromatography.

This standard is applicable to determination of thiacloprid residues in tomatoes, cabbage, turnips, potatoes, apples, bananas, soybeans, leeks, onions, pork, chicken, liver, fat, honey, rice, eggs, milk and tea.

2 Normative reference

The following documents for the application of this text are necessory. For dated reference, only the live vision of the document applied to this text. For undated documents, the lastest edition of the document(include all amendments) applies to this text.

GB/T 6682 Water for analytical laboratory use—Specifications and test methods (ISO 3696: 1987, MOD)

3 Principle

The contents of thiacloprid in the test sample are extracted with ethyl acetate or acetonitrile, purified with SCX SPE Column, the contents are determined by HPLC, quantified by external standard method.

4 Reagents and materials

Unless specified, all reagents used should be of analytical grade; "water" is the first grade water prescribed by GB/T 6682.

4.1 Acetonitrile: Chromatography pure.

4.2 Methanol: Chromatography pure.

4.3 Petroleum ether: Chromatography pure.

4.4 Ethyl acetate: Chromatography pure.

4.5 *n*-Hexane: Chromatography pure.

4.6 Sodium chloride.

4.7 Anhydrous sodium sulfate: bake at 650 ℃ for 4 h, and store in air-tight container, use after cool.

4.8 Sodium chloride saturated solution.

4.9 Petroleum ether-Ethyl acetate (85 + 15, *V*/*V*): Mix 850 mL petroleum ether and 150 mL ethyl acetate.

4.10 Petroleum ether-Ethyl acetate (30 + 70, *V*/*V*): Mix 300 mL petroleum ether and 700 mL ethyl acetate.

4.11 Acetonitrile-water (30 + 70, *V*/*V*): Mix 300 mL acetonitrile and 700 mL water.

4.12 Thiacloprid standard chemicals (thiacloprid, $C_{10}H_9ClN_4S$, GAS No.: 111988-49-9): purity ≥95%.

4.13 Standard stock solution: Accurately weigh appropriate standard (accurate to 0.000 1 g), dissolve and quantitatively with methanol. The concentration of the solution is 1.0 mg/mL. The solution is storded in refrigerator at −18 ℃, period of validity is 1 year.

4.14 Standard working solution: According to the requirement, dilute standard stock solution with methanol to appropriate concentration series. The solution is storded in refrigerator at 4 ℃, period of validity is 3 months.

4.15 SCX column (500 mg, 6 mL) or equivalent.

4.16 Membrane: 0.22 μm, for organic phase.

5 Apparatus and equipments

5.1 High performance liquid chromatograph: Equipped with UV-VIS detector.

5.2 Analytical balance: 0.000 1 g and 0.01 g.

5.3 Centrifuge: 4 000 r/min.

5.4 Tissue blender.

5.5 Homogenizer.

5.6 Solid-phase extraction apparatus.

5.7 Nitrogen evaporator.

5.8 Vortex mixer: speed adjustable.

6 Sample preparation and storage

6.1 Requirement

In the course of sampling and sample preparation, precautions shall be take to avoid the contamination or any factors which may cause the change of residue content.

6.2 Fruits, vegetables

Take approximately 500 g representative samples. Samples can not be washed with water, the edible parts are blended and homogenized by a tissue blender. Put in suitable clean containers, seal and label, store below −18 ℃.

6.3 Chicken, pork, pork liver, egg

Take approximately 500 g representative samples. The edible parts are blended and homogenized by a tissue blender. Put in suitable clean containers, seal and label, store below −18 ℃.

6.4 Honey

Take approximately 500 g representative samples, for non-crystalline samples ,Stir absolutly; for crystallized sample, melt the sample in a sealed case in a water bath less than 60 ℃, stir the samples after melting, and cool it to room temperature. Attention must be taken to prevent water evaporation while melting. Put in suitable clean containers, seal and label, store at room temperature.

6.5 Milk, fat liquid samples or can be melt to liquid samples

Take approximately 500 g representative samples. Put in suitable clean containers, seal and label, store at room temperature.

6.6 Rice, Tea, soybean

Take approximately 500 g representative samples, the samples are pulverized with a pulverizer and mix thoroughly, place in suitable clean containers, seal and label, store at room temperature.

7 Analytical procedure

7.1 Extraction

7.1.1 Fruits, vegetables

Accurately weigh 2.0 g of the test sample (accurate to 0.01 g) into a 10 mL centrifuge tube, add 2 mL saturated aqueous sodium chloride and shake. Then add 4 mL ethyl acetate in the glass, and mixed with a vortex mixer, extract for 30 min under ultrasonic wave. After centrifugation at 4 000 rpm for 10 min, the supernatant is collected in a graduated tube. The residues are re-extracted once as the above procedure, combine the supernatants. Evaporate the extract solution to dryness at 45 ℃ under nitrogen flow. Resolve the residues with 4 mL Petroleum ether-Ethyl acetate (85 + 15, *V*/*V*).

7.1.2 Chicken, pork, pork liver, honey, rice, eggs, milk, soybean, tea

Accurately weigh 2.0 g of the test sample (accurate to 0.01 g) into a 10 mL glass centrifuge tube, add 2 mL water and mix. Then add 4 mL ethyl acetate in the glass, and mixed with a vortex mixer, extract for 30 min under ultrasonic wave. After centrifugation at 4 000 rpm for 10 min, the supernatant is collected in a graduated tube. The residues are re-extracted once as the above procedure, combine the supernatants. Evaporate the extract solution to dryness at 45 ℃ under nitrogen flow. Resolve the residues with 4 mL methnol, then add 3 mL hexane and mix with a vortex mixer for 5 min. After centrifugation at 4 000 rpm for 5 min, the extraction liquid is collected in a graduated tube. The residues are re-extracted once as the above procedure, combine the extraction liquid. Evaporate the extract solution to dryness at 45 ℃ under nitrogen flow. Resolve the residues with 4 mL Petroleum ether-Ethyl acetate (85 + 15, *V*/*V*).

7.1.3 Fat

Accurately weigh 2.0 g of the test sample (accurate to 0.01 g) into a 10 mL glass centrifuge tube, add 4 mL acetonitrile ,extract for 5 min in a high speed homogenizer and for 30 min under ultrasonic wave. After centrifugation at 4 000 rpm for 10 min, the supernatant is collected in a graduated tube. The residues are re-extracted once with 2 mL acetonitrile as the above procedure, combine the supernatants, then add 3 mL hexane and mix with a vortex mixer for 5 min. After centrifugation at 4 000 rpm for 5 min, the extraction liquid is collected in a graduated tube. The residues are re-extracted once as the above procedure, combine the extraction liquid. Evaporate the extract solution to dryness at 45 ℃ under nitrogen flow. Resolve the residues with 4 mL Petroleum ether-Ethyl ace-

tate (85+15, *V*/*V*).

7.2 Cleanup

The SCX column was activated using 5 mL of methanol and 3 mL of water, respectively, transfer the above solution into the activation of SCX column. Rinse the column with 10 mL petroleum ether-Ethyl acetate (30+70, *V*/*V*). All elutes is collected in a graduated tube, concentrated to dryness by nitrogen instrument at 50 ℃, residue was dissolved in 1 mL acetonitrile-water (30+70, *V*/*V*). After being filtrated with a 0.22 μm filter, the final solution is used for HPLC determination.

7.3 Determination

7.3.1 LC operating conditions

LC conditions as follow:

a) Column: C_{18} column, length 150 mm, inner diameter 4.6 mm , particle size 5 μm. Or equivalent;

b) Mobile phase: acetonitrile-water (30+70, *V*/*V*);

c) Flow rate: 1.0 mL/min;

d) Injection volume: 20 μL;

e) Column temperature: Room temperature;

f) Detection wavelength: 240 nm;

g) Mass parameters: listed in the Annex A.

7.3.2 Determination

According to the approximate concentration of thiacloprid in the sample solution, select the standard working solution with similar responses to that of the sample solution. The responses of thiacloprid in the standard working solution and sample solution should be within the linear range of the instrumental detection. The standard working solution should be randomly injected in－between the injections of sample solution of equal volume. Under the above operating condition, the retention time of thiacloprid is about 7.2 min. The chromatogram of the standard working solutionis shown at figure B.1.

7.3.3 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 peak are consistent with that of working solution, and the relative abundance ratio tolerance meets the requirements listed in the Table 1, it can be concluded that this compound does exist in the sample.

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

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

7.4 Blank test

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

8 Calculation and expression of result

Calculate the content of thiacloprid residue in the test sample by HPLC data processor or according to the formula (1). The blank value should be subtracted from the result of calculation.

$$X = \frac{A \times c \times V}{A_s \times m} \quad \cdots\cdots (1)$$

Where:

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

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

c —the concentration of thiacloprid in standard solution, μg/mL;

V —the final volume of sample solution, mL;

A_s—the peak area of thiacloprid in the standard solution;

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

9 Limit of quantification and recovery

9.1 Limit of quantification

The Limit of quantification of this method is 0.01 mg/kg.

9.2 Recovery

The recovery range of thiacloprid is listed in table C.1.

Annex A
(Informative)
Main Mass Parameters[1)]

MS/MS parameters see the followings as reference:

a) Ion Source: ESI, Positive;

b) Monitor mode: Multiple reaction monitoring, MRM;

c) Resolution: Unit mass resolution;

d) Other parameters: see the follow table A.1.

Table A.1—Related parameters and qualifier and quantifier in MRM

Compound name	Parent ions, m/z	Ions, m/z	Collision energy/V	Cone Voltage/V
thiacloprid	253.0	126.0(Transition for quantification)	29	67
		186.0	19	66

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 or different type.

Annex B
(Informative)
Chromatogram of the thiacloprid standard

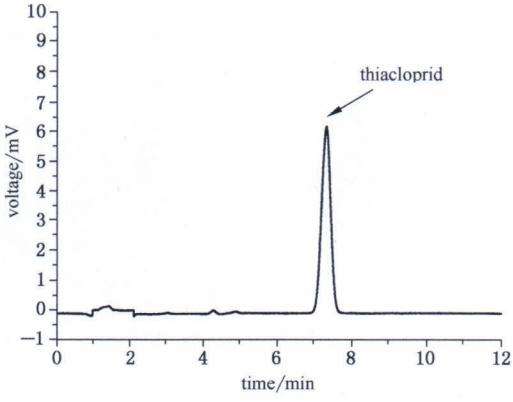

Figure B.1—Liquid chromatogram of thiacloprid (0.2 mg/L)

Annex C
(Informative)
Results of recoveries of thiacloprid residues

Table C.1—Fortifying concentration of thiacloprid in samples and its corresponding recoveries ($n=10$)

Sample	Spiked level/ (mg/kg)	Recovery range/ %	Sample	Spiked level/ (mg/kg)	Recovery range/ %
Tomatoe	0.01	77.5～99.2	Rice	0.01	75.9～103.6
	0.1	82.2～106.4		0.1	85.3～107.7
	5.0	93.5～101.8		2.0	86.8～103.7
Cabbage	0.01	74.7～100.5	Honey	0.01	77.0～97.7
	0.1	81.2～106.9		0.1	82.1～109.3
	5.0	93.6～100.5		2.0	85.5～106.1
Turnip	0.01	73.7～109.4	Pork	0.01	78.3～105.2
	0.1	81.2～105.3		0.1	86.5～104.3
	5.0	93.6～101.8		2.0	84.5～105.6
Potatoe	0.01	74.7～105.5	Chicken	0.01	77.3～107.1
	0.1	80.8～103.2		0.1	85.1～107.2
	2.0	86.3～104.8		2.0	82.9～107.8
Apple	0.01	76.7～103.2	Liver	0.01	74.6～103.8
	0.1	81.7～104.8		0.1	75.2～101.6
	5.0	88.7～100.4		2.0	76.5～101.8
Banana	0.01	76.4～105.3	Fat	0.01	75.9～102.8
	0.1	83.7～106.2		0.1	86.9～105.7
	5.0	91.9～102.2		2.0	87.5～104.4
Soybean	0.01	78.4～102.4	Egg	0.01	77.8～106.2
	0.1	83.7～104.1		0.1	82.1～107.9
	2.0	87.8～106.2		2.0	90.1～104.4
Leek	0.01	74.3～104.3	Milk	0.01	74.8～106.6
	0.1	84.0～101.0		0.1	82.2～104.3
	5.0	88.4～101.5		2.0	88.0～103.4
Onion	0.01	78.3～104.7	Tea	0.01	76.8～104.2
	0.1	81.6～108.9		0.1	82.7～105.6
	5.0	88.9～101.6		2.0	88.4～103.9