

SN

中华人民共和国出入境检验检疫行业标准

SN/T 0125—2010
代替 SN 0125—1992

进出口食品中敌百虫残留量检测方法 液相色谱-质谱/质谱法

Determination of trichlorfon residues in meat and meat products for import
and export—LC-MS/MS method

2010-11-01 发布

2011-05-01 实施

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 0125—1992《出口肉及肉制品中敌百虫残留量的检验方法》。

本标准与 SN 0125—1992 相比,主要技术变化如下:

- 使用范围、指标包括原标准的内容;
- 采用气相色谱质谱法;
- 增加阳性确证内容;
- 整合前处理条件。

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。

本部分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:中华人民共和国吉林出入境检验检疫局、中华人民共和国天津出入境检验检疫局。

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本标准所代替标准历次版本发布情况为:

- ZBX 71002—1987、ZBX 22009—1988、SN 0125—1992。

进出口食品中敌百虫残留量检测方法

液相色谱-质谱/质谱法

1 范围

本标准规定了出口农产品中敌百虫残留量的液相色谱-质谱/质谱检测和确证方法。

本标准适用于出口清蒸猪肉罐头、猪肉、鸡肉、牛肉、鱼肉、香肠、糙米、玉米、洋葱、核桃中敌百虫残留量的测定。

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试验方法

3 方法提要

试样用环己烷+乙酸乙酯(1+1,体积比)提取,经凝胶色谱净化,用甲醇+水(1+1,体积比)定容,供液相色谱-质谱/质谱仪测定,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 GB/T 6682 规定的一级水。

- 4.1 环己烷:高效液相色谱级。
- 4.2 甲醇:高效液相色谱级。
- 4.3 乙酸乙酯:残留级。
- 4.4 无水硫酸钠:650℃灼烧4h,储于密封容器中备用。
- 4.5 环己烷+乙酸乙酯:(1+1,体积比):量取100mL环己烷和100mL正己烷,混匀。
- 4.6 乙酸铵:优级纯。
- 4.7 50mmol/L乙酸铵溶液:称取0.385g乙酸铵溶于1000mL水中。
- 4.8 敌百虫标准品(trichlorfon,CAS编号:52-68-6,分子式: $C_4H_8Cl_3O_4P$):纯度大于等于98.0%。
- 4.9 敌百虫标准储备溶液:准确称取适量的敌百虫标准品,用甲醇配制成浓度为100 $\mu\text{g}/\text{mL}$ 的标准储备溶液。该溶液在0℃~4℃冰箱中保存。
- 4.10 敌百虫标准工作溶液:根据需要将敌百虫标准储备溶液(3.10)用甲醇稀释成适用浓度的标准工作溶液。该溶液在0℃~4℃冰箱中保存。
- 4.11 有机微孔滤膜:0.20 μm ,0.45 μm 。

5 仪器与设备

- 5.1 液相色谱-质谱/质谱仪:配有电喷雾离子源(ESI)。

- 5.2 电子天平:感量:0.000 1 g。
- 5.3 凝胶色谱仪:配有单元泵、馏分收集器。
- 5.4 均质器。
- 5.5 旋转蒸发器。
- 5.6 浓缩瓶:250 mL。
- 5.7 离心机:3 000 r/min 以上。
- 5.8 离心管:四氟乙烯,50 mL。
- 5.9 涡旋混匀器。

6 试样制备与保存

6.1 试样制备

- 6.1.1 肉:将所取全部样品,缩分出有代表性样品不少于 500 g,经捣碎机充分捣碎均匀,装入洁净容器,密封,标明标记。
- 6.1.2 罐头:将所取全部样品正罐倒出,缩分出有代表性样品不少于 500 g,经捣碎机充分捣碎均匀,装入洁净容器,密封,标明标记。
- 6.1.3 糙米、玉米、洋葱、核桃:取代表性样品约 500 g,用粉碎机粉碎,混匀,装入洁净容器,密封,标明标记。

6.2 试样保存

肉类试样于-18℃以下冷冻保存;粮谷类、坚果类及其他类试样于 0℃~4℃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 提取

称取解冻试样 10 g(精确到 0.01 g)于 50 mL 离心管(5.8)中,加 6 g 无水硫酸钠(4.5)和 20 mL 环己烷+乙酸乙酯(4.6),均质 2 min,在 3 000 r/min 离心 3 min,吸出环己烷+乙酸乙酯层通过装有无水硫酸钠的桶形漏斗,收集于 250 mL 浓缩瓶中,残渣分别用 15 mL 环己烷+乙酸乙酯提取两次,提取液合并于 250 mL 浓缩瓶中,于 40℃下浓缩至约 2 mL,将 2 mL 提取液转移至试管中,并用 3 mL 环己烷-乙酸乙酯(4.6)分三次洗涤浓缩瓶,合并洗涤液于试管中,在室温下,氮吹浓缩近干,用环己烷-乙酸乙酯(4.6)定容 10 mL,过 0.45 μm 滤膜,待凝胶色谱(GPC)净化。

7.2 净化

7.2.1 凝胶色谱(GPC)净化

7.2.1.1 凝胶色谱条件

凝胶色谱条件如下:

- a) 凝胶净化柱:Bio Beads S-X3,700 mm×25 mm(内径),或相当者;
- b) 流动相:乙酸乙酯-环己烷(1+1,体积比);
- c) 流速:4.7 mL/min;
- d) 样品定量环:10 mL;

- e) 预淋洗时间:10 min;
- f) 凝胶色谱平衡时间:5 min;
- g) 收集时间:21 min~28 min。

7.2.1.2 凝胶色谱净化步骤

将 10 mL 待净化液(7.1)按 7.2.1.1 规定的条件进行净化,收集组分于室温下氮吹浓缩近干,用甲醇+水(1+1,体积比)溶解并定容至 1.0 mL,过 0.20 μm 滤膜,供液相色谱-质谱/质谱仪测定。

7.3 测定

7.3.1 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:kromasil 100-5 C_{18} 色谱柱,150 mm \times 2.1 mm(内径),粒径 5 μm 或相当者;
- b) 流动相:甲醇(4.4)-50 mmol/L 乙酸胺溶液(4.9),梯度洗脱程序见表 1:

表 1 流动相梯度洗脱程序

时间 min	甲醇 %	50 mmol/L 乙酸胺溶液 %
0	10.0	90.0
5.00	10.0	90.0
10.00	95.0	5.0
18.00	95.0	5.0
18.01	10.0	90.0
25.00	10.0	90.0

- c) 流速:0.20 mL/min;
- d) 柱温:40 $^{\circ}\text{C}$;
- e) 进样量:10 μL ;
- f) 离子源:电喷雾离子源;
- g) 扫描方式:正离子;
- h) 检测方式:多反应检测(MRM);
- i) 质谱条件参见附录 A。

7.3.2 色谱测定

根据试样中被测样液的含量情况,选取响应值相近的标准工作液进行色谱分析。标准工作液和样液中待测物的响应值均应在仪器线性响应范围内。在上述色谱条件下敌百虫的参考保留时间约为:15.75 min,敌百虫标准品多反应检测(MRM)色谱图参见附录 B 中 B.1;外标法定量。

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,定量测定时采用标准曲线法。定性时应当与浓度相当标准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,则可判定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

7.3.4 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

用色谱数据处理机或用标准曲线按式(1)计算试样中敌百虫的残留量,计算结果需扣除空白值:

$$X = \frac{c \times V \times n}{m \times 1000} \dots\dots\dots (1)$$

式中:

X —— 试样中敌百虫残留量,单位为毫克每千克(mg/kg);

c —— 从标准曲线上得到的待测液中敌百虫的浓度,单位为纳克每毫升(ng/mL);

V —— 样液最终定容体积,单位为毫升(mL);

n —— 稀释倍数;

m —— 最终样液所代表的试样质量,单位为克(g)。

注:计算结果应扣除空白值。

9 测定低限(LOQ)和回收率

9.1 测定低限(LOQ)

本方法对出口清蒸猪肉罐头、猪肉、鸡肉、牛肉、鱼肉、香肠中敌百虫残留量的测定低限均为 0.002 mg/kg(LOQ);糙米、玉米、洋葱、核桃中敌百虫残留量的测定低限均为 0.004 mg/kg(LOQ)。

9.2 回收率

回收率的实验数据(在不同添加浓度范围内)见表 3。

表 3 本方法添加浓度及回收率范围

样品名称	添加浓度 mg/kg	回收率范围 %	测定低限 mg/kg
清蒸猪肉罐头	0.002	70.0~86.5	0.002
	0.010	71.0~107.0	
	0.200	81.8~99.2	
猪肉	0.002	69.5~95.5	0.002
	0.010	70.0~101.0	
	0.200	76.4~102.7	

表 3 (续)

样品名称	添加浓度 mg/kg	回收率范围 %	测定低限 mg/kg
鸡肉	0.002	71.0~91.5	0.002
	0.010	68.0~109.0	
	0.200	85.1~95.8	
牛肉	0.002	70.5~96.5	0.002
	0.010	67.0~86.0	
	0.200	85.7~100.0	
鱼肉	0.002	69.0~101.5	0.002
	0.010	71.0~105.0	
	0.200	89.6~110.1	
香肠	0.002	65.0~86.5	0.002
	0.010	71.0~107.0	
	0.200	74.9~99.2	
糙米	0.004	70.0~87.5	0.004
	0.010	70.0~82.0	
	0.200	71.1~98.9	
玉米	0.004	70.0~96.0	0.004
	0.010	71.0~95.0	
	0.200	72.3~87.9	
洋葱	0.004	71.0~92.5	0.004
	0.010	70.0~96.0	
	0.500	93.4~100.9	
核桃	0.004	70.0~95.0	0.004
	0.010	70.0~94.0	
	0.500	71.6~92.6	

附录 A

(资料性附录)

API 4000 LC/MS/MS 检测敌百虫色谱条件
电喷雾离子源参考条件

检测离子对及电压参数:

- a) 气帘气(CUR):15.00 psi;
- b) 雾化气(GS1):40.00 psi;
- c) 辅助加热气(GS2):45.00 L/min;
- d) 碰撞气(CAD):7.00 psi;
- e) 离子源喷雾电压(IS):5 000.00 V;
- f) 离子源温度(TEM):550 °C;
- g) 定性离子对、定量离子对、去簇电压、碰撞能量、碰撞室出口电压见表 A. 1。

表 A. 1 Q₁、Q₃、去簇电压、碰撞能量、碰撞室出口电压表

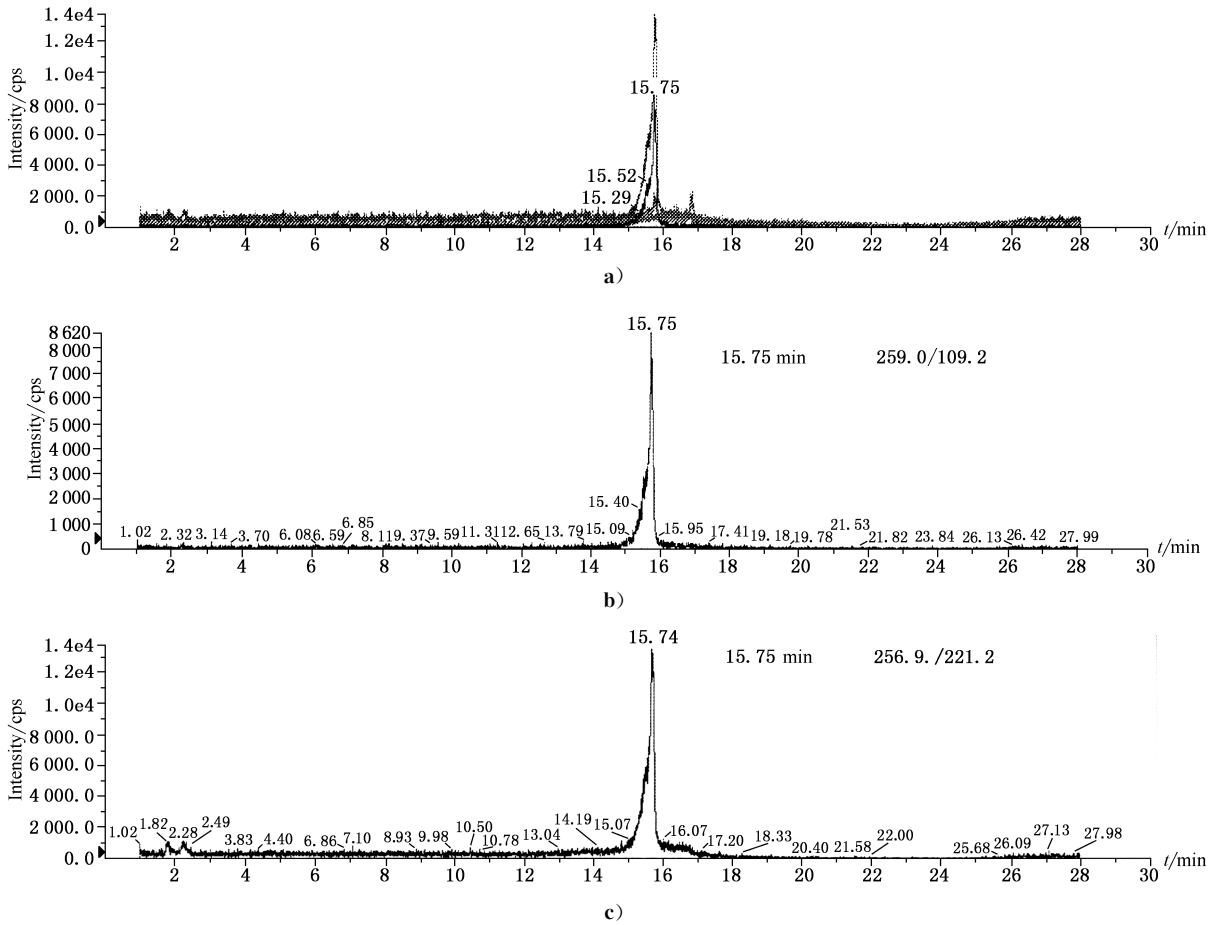
名称	Q ₁ m/z	Q ₃ m/z	去簇电压 V	碰撞能量 eV	碰撞室出口电压 V
敌百虫 trichlorfon	259.0	109.2	63.0	26.5	19.0
	256.9	221.2	63.0	16.0	13.0

1) 非商业声明:附录 A 所列参考质谱条件是在 API 4000 液相色谱-质谱/质谱仪上完成的,此处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)

敌百虫标准品多反应检测(MRM)色谱图

图 B.1 敌百虫标准品多反应检测(MRM)色谱图(0.01 $\mu\text{g}/\text{mL}$)

Foreword

This standard was drafted according to GB/T 1.1—2009.

This section of the original standards(SN/T 0125—1992) was revised and consolidated index,use the range, including the original 2-related standards, using gas chromatography-mass spectrometry, increase positive confirmation of content,integration of pretreatment conditions.

Please note that some of the content of this document may involve patent. This file is not responsible for the publishing sector recognizes the responsibility of these patents.

This standard was 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 the Jilin Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Tianjin Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

This main drafter of this standard is Li Aijun., Wang Mingtai, Ma Xu, Mu Jun, Lu Lijun, Ma Shumin, Zhou Xiao.

Determination of trichlorfon residues in meat and meat products for import and export— LC-MS/MS method

1 Scope

This standard specifies the method of testing trichlorfon residue in meat and meat products for export by LC-MS/MS method.

This standard is applicable to the determination of residue content of trichlorfon in canned steamed pork, pork, chicken, beef, fish, sausage, rice (brown rice), corn, onion, walnut for export.

2 Normative references

This items of the following listed standard become the items of this standard due to the quotation by this standard. The cited references with date would not apply to this standard if their amendment (not including corrected printing errors) or revision appear. However, it is encouraged of study of the newest edition of these references can be used. The newest edition is applicable to this standard if the references are not quoted with date.

GB/T 6682 Water for laboratory use—Specifications

3 Principle

The test sample is extracted with ethyl acetate-*n*-hexane (1 + 1, V/V), after being concentrated, cleaned up by sequentially passing through gel permeation chromatography (GPC), The residues is determined by liquid chromatography-mass spectrometry, quantified by external standard method.

4 Reagents and materials

All the reagents used should be analytically pure unless otherwise specified. “Water” is the first level water described by GB/T 6682.

4.1 Cyclohexane: HPLC grade.

- 4.2 Methanol:HPLC grade.
- 4.3 Ethyl acetate:Residue grade.
- 4.4 Anhydrous sodium sulfate:Ignited at 650 °C for 4 h,and then stored in a tightly sealed container.
- 4.5 Ethyl acetate-*n*-Hexane (1 + 1, V/V): Volume 100 mL ethyl acetate into 250 mL erlenmeyer flask, then add 100 mL *n*-Hexane,mix them.
- 4.6 Ammonium acetate:GR.
- 4.7 50 mmol/L Ammonium acetate:Weigh ammonium acetate 0.385 g dissolve with water and dilute to 1 000 mL.
- 4.8 Trichlorfon standard:CAS No. 52-68-6, C₄H₈Cl₃O₄P, Purity ≥ 98%.
- 4.9 Standard stock solution:Accurately weigh certain amount of trichlorfon standard and dissolve it with Methanol to make the standard stock solution of 100 µg/mL. The solution is stored in a refrigerator at 0 °C ~ 4 °C.
- 4.10 Standard working solution:Dilute the standard stock solution with methanol to the required concentration to make the standard working solution. The solution is stored in a refrigerator at 0 °C ~ 4 °C.
- 4.11 Membrane filter:0.20 µm,0.45 µm.

5 Apparatus and equipment

- 5.1 Liquid chromatography-mass spectrometry, equipped with electrospray ion source and triquadruple mass spectrometer.
- 5.2 Electronic balance; accurate to 0.000 1 g.
- 5.3 GPC; equipped with isocratic pump and fraction collector.
- 5.4 Homogenizer.
- 5.5 Rotary vacuum evaporator.
- 5.6 Concentrate bottle; 250 mL.
- 5.7 Centrifuge; 3 000 r/min.

5.8 Plastic centrifuge tube:50 mL.

5.9 Mechanical shaker.

6 Preparation and storage of test sample

6.1 Preparation of test sample

6.1.1 Meat: Take approximately 500 g of representative sample. Collect the edible pieces. Crush with a crusher. Mix thoroughly. Put in clean containers. Seal and label them.

6.1.2 Canned steamed pork: Take all the content of canned steamed pork, crush with a crusher. Mix thoroughly. Put in clean containers. Seal and label them.

6.1.3 Corn, brown-rice, onion, walnut: Take approximately 500 g of representative sample. Smash thoroughly by a breaker. Mix thoroughly. Put in clean containers. Seal and label them.

6.2 Storage of test sample

The test samples of meat should be frozen and stored below $-18\text{ }^{\circ}\text{C}$. Other samples should be stored between $0\text{ }^{\circ}\text{C}\sim 4\text{ }^{\circ}\text{C}$.

While sampling and preparing sample, please avoid contamination or any factors that may change residue content.

7 Procedure

7.1 Extraction

Weigh 10 g (accurate to 0.01 g) of the test sample into a 50 mL centrifuge tube. Add 6 g of anhydrous sodium sulfate and 20 mL ethyl acetate-*n*-hexane. Homogenize for 2 min. Centrifuge for 5 min at 3 000 *r/min*. Take the layer of ethyl acetate-*n*-hexane into 250 mL concentrate bottle, the residue was homogenized and extracted with $2\times 15\text{ mL}$ ethyl acetate-*n*-hexane for 2 times. Combine all the layer of ethyl acetate-*n*-hexane into 250 mL concentrate bottle, Evaporate the ethyl acetate-*n*-hexane to about 2 mL below $40\text{ }^{\circ}\text{C}$, remove the residues to a centrifuge tube, wash concentrate bottle with $3\times 1\text{ mL}$ ethyl acetate-*n*-hexane for 3 times, combine the ethyl acetate-*n*-hexane into the centrifuge tube, evaporate the extract to dryness on N-Evap at room temperature. Add 10 mL of ethyl acetate-*n*-hexane(1+1) to dissolve the residue, filter the solution with 0.45 μm membrane and then wait for cleaning-up.

7.2 Cleaning-up

7.2.1 GPC Cleaning-up

7.2.1.1 GPC operating condition

- a) GPC column: 700 mm × 25 mm (i. d.), Bio Beads S-X3 or equivalent;
- b) Mobile phase: Cyclohexane-ethyl acetate (1 + 1);
- c) Flow rate: 4.7 mL/min;
- d) Injection volume at sample loop: 10 mL;
- e) Time of pre-rinsing: 10 min;
- f) GPC balance time: 5 min;
- g) Time of collecting the eluates: 21 min ~ 28 min.

7.2.1.2 GPC cleaning-up procedure

Purify 10 mL of the solution which is waiting for cleaning-up (7.1) according to the condition described in the section 7.2.1.1. Condense the collected constituents to evaporate the extract to dryness on N-Evap at room temperature. Dissolve the residue and dilute exactly to 1.0 mL with Acetonitrile, filter the solution with 0.20 μm membrane. The solution is ready for LC-MS/MS determination.

7.3 Determination

7.3.1 LC-MS/MS operating conditions

- a) LC column: Kromasil 100-5 C₁₈, 150 mm × 2.1 mm (i. d.), 5 μm (or other equivalent one);
- b) Mobile phase: Methanol-50 mmol/L ammonium acetate (4.9); elution gradient is listed in table 1;

Table 1—Elution gradient

Time min	Methanol %	50 mmol/L ammonium acetate %
0	10.0	90.0
5.00	10.0	90.0
10.00	95.0	5.0
18.00	95.0	5.0

Table 1 (continued)

Time min	Methanol %	50 mmol/Lammonium acetate %
18.01	10.0	90.0
25.00	10.0	90.0

- c) Flow rate:0.2 mL/min;
- d) Column temperature:40 °C ;
- e) Injector volume:10 μL;
- f) Ion source:ESI;
- g) Scanning model:Postive ion;
- h) Monitoring model:Multiple reaction monitoring(MRM);
- i) Referenced conditions seen Annex A.

7.3.2 LC-MS/MS Determination

According to the approximate concentration of analyte in sample solution, select the standard working solution with similar responses to that of sample solution. The responses of analyte in the standard working solution and the sample solution should be within the linear range of the instrument detertion. Under the above LC-MS/MS operating conditions, the retention time of trichlorfon is about 15.75 min. Reconstituted ion chromatogram of standard working solution is listed in Annex B. 1,quantified by external standard method.

7.3.3 Confirmation of LC-MS/MS

Under the LC-MS/MS operating conditions, the standard working solution and sample solution is injected. If the retention times of sample chromatogram pecks are consistent with that of standard solution, calibration curve method is used for quantitative measurement. The relative intensities of sample transiyions shall correspond to those of standard solution transitions for confirmation. The concentration of standard solution should be same with those of sample solution. The permitted tolerances listed in table 2, then the corresponding analyte must be present in sample.

Table 2—Maximum permitted tolerances relative ion intensities while confirmation

relative intensity/%	>50	>20~50	>10~20	≤10
permitted to lerances/%	± 20	± 25	± 30	± 50

7.3.4 Blank test

Undergo according to the above procedures excluding the sample.

8 Calculation and expression of the result

Calculate the content of Trichlorfon residue in the test sample by GC-MS data processor or according to the followed formula(1).

$$X = \frac{c \times V \times n}{m \times 1000} \dots\dots\dots (1)$$

where

X —the residue content of Trichlorfon in the test sample. Unit is milligram per kilogram,mg/kg;

c —the concentration of Trichlorfon in the standard working solution. Unit is microgram per milliliter,ng/mL;

V —the final volume of the sample solution. Unit is milliliter,mL;

n —dilute times;

m —the corresponding mass of the test sample representing the final sample solution. Unit is gram,g.

Note: The result of calculation should be deducted with blank value.

9 Detection limit and recovery

9.1 Limit of determination

The method detection limit for canned steamed pork, pork, chicken, beef, fish, sausage is 0.002 mg/kg (LOQ);Rice (brown rice),corn,onion,walnut is 0.004 mg/kg.

9.2 Range of fortification and recovery

The range of fortification and recovery of this method is shown in table 3.

Table 3—Range of fortification and recovery of this method

Sample	Fortified content mg/kg	Recovery range %	Limit of determination and confirmation mg/kg
canned steamed pork	0.002	70.0~86.5	0.002
	0.010	71.0~107.0	
	0.200	81.8~99.2	
pork	0.002	69.5~95.5	0.002
	0.010	70.0~101.0	
	0.200	76.4~102.7	
chicken	0.002	71.0~91.5	0.002
	0.010	68.0~109.0	
	0.200	85.1~95.8	
beef	0.002	70.5~96.5	0.002
	0.010	67.0~86.0	
	0.200	85.7~100.0	
fish	0.002	69.0~101.5	0.002
	0.010	71.0~105.0	
	0.200	89.6~110.1	
sausage	0.002	65.0~86.5	0.002
	0.010	71.0~107.0	
	0.200	74.9~99.2	
rice(brown rice)	0.004	70.0~87.5	0.004
	0.010	70.0~82.0	
	0.200	71.1~98.9	
corn	0.004	70.0~96.0	0.004
	0.010	71.0~95.0	
	0.200	72.3~87.9	
onion	0.004	71.0~92.5	0.004
	0.010	70.0~96.0	
	0.200	93.4~100.9	
walnut	0.004	70.0~95.0	0.004
	0.010	70.0~94.0	
	0.200	71.6~92.6	

Annex A
(Informative)

Referenced conditions for analysing trichlorfon by API 4000 LC-MS/MS

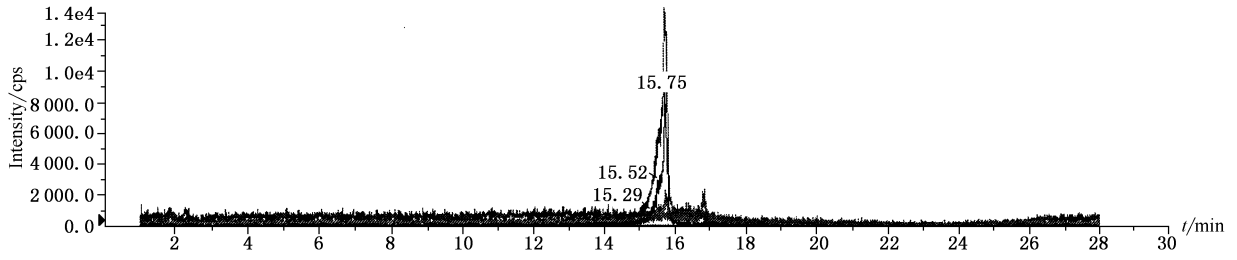
Referenced conditions for electrospray ionization source of API 4000 LC-MS/MS:

- a) Curtain gas(CUR) :15.00 psi;
- b) Nebulizer(GS1) :40.00 psi;
- c) Auxiliary gas(GS2) :45.00 L/min;
- d) Collision gas(CAD) :7.00 psi;
- e) Ion spray voltage(IS) :5 000.00 V;
- f) Probe temperature(TEM) :550.00 °C ;
- g) Quantitative ion, qualitative ion, Declustering potential voltage(V) , collision gas energy(eV) , see Table A. 1.

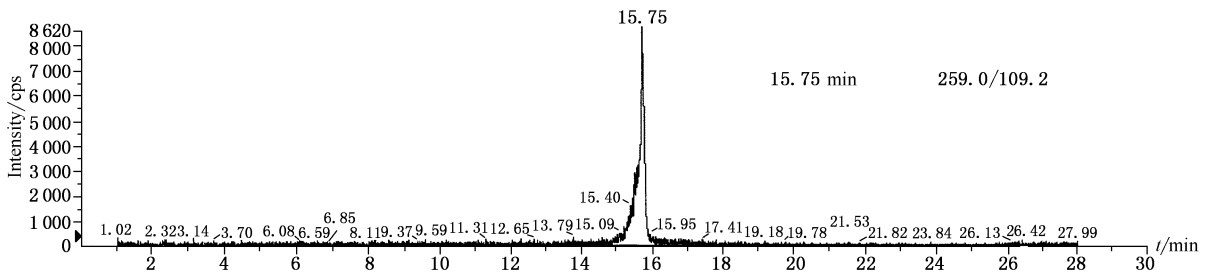
Table A. 1— Q_1 、 Q_3 、DP、CE and CXP

Name	Q_1 m/z	Q_3 m/z	DP V	CE eV	CXP V
trichlorfon	259.0	109.2	63.0	26.5	19.0
	256.9	221.2	63.0	16.0	13.0

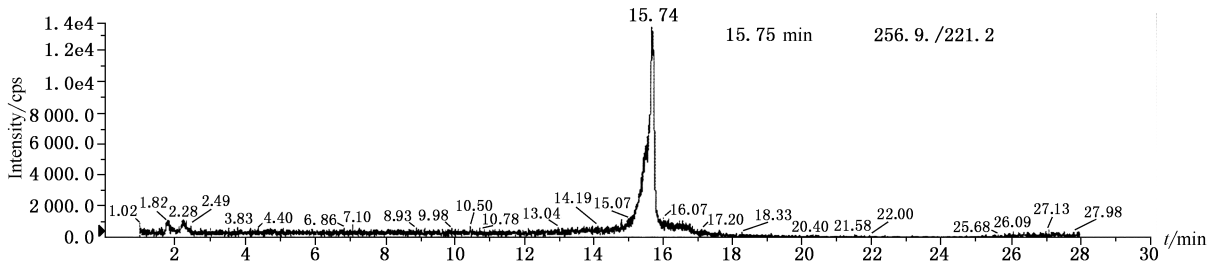
Annex B
(Informative)
MRM Chromatogram of the standards



a)



b)



c)

Figure B. 1—MRM chromatogram of Trichlorfon standards(0.01 $\mu\text{g}/\text{mL}$)