



中华人民共和国出入境检验检疫行业标准

SN/T 0123—2010
代替 SN 0123—1992, SN 0214—1993

进出口动物源食品中有机磷农药 残留量检测方法 气相色谱-质谱法

Determination of organophosphorus multiresidues in foodstuffs
of animal origin for import and export—GC-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

本部分按照 GB/T 1.1—2009 给出的规则起草。

本部分代替 SN 0123—1992《出口肉及肉制品中敌敌畏、二嗪磷、倍硫磷、马拉硫磷残留量检验方法》、SN 0214—1993《出口肉及肉制品中敌敌畏、二嗪磷、皮蝇磷、毒死蜱、杀螟硫磷、对硫磷、乙硫磷、蝇毒磷残留量检验方法》。

本部分与 SN 0123—1992、SN 0214—1993 相比，主要技术变化如下：

- 使用范围；
- 指标包括原 2 项相关标准的内容；
- 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。

本部分起草单位：中华人民共和国吉林出入境检验检疫局。

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本部分所代替标准的历次版本发布情况为：

- ZBB 22016—1988、SN 0123—1992；
- SN 0214—1993。

进出口动物源食品中有机磷农药 残留量检测方法 气相色谱-质谱法

1 范围

本标准规定了进出口动物源食品中 9 种有机磷农药残留量(敌敌畏、二嗪磷、皮蝇磷、杀螟硫磷、马拉硫磷、毒死蜱、倍硫磷、对硫磷、乙硫磷)的气相色谱-质谱检测方法。

本标准适用于清蒸猪肉罐头、猪肉、鸡肉、牛肉、鱼肉中有机磷农药残留量的测定和确证。

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

试样用水-丙酮溶液均质提取,二氯甲烷液-液分配,凝胶色谱柱净化,再经石墨化炭黑固相萃取柱净化,气相色谱-质谱检测,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 丙酮:残留级。
 - 4.2 二氯甲烷:残留级。
 - 4.3 环己烷:残留级。
 - 4.4 乙酸乙酯:残留级。
 - 4.5 正己烷:残留级。
 - 4.6 氯化钠。
 - 4.7 无水硫酸钠:650 ℃灼烧 4 h,储于密封容器中备用。
 - 4.8 氯化钠水溶液(5%):称取 5.0 g 氯化钠,用水溶解,并定容至 100 mL。
 - 4.9 乙酸乙酯-正己烷(1+1,体积比):量取 100 mL 乙酸乙酯和 100 mL 正己烷,混匀。
 - 4.10 环己烷-乙酸乙酯:(1+1,体积比):量取 100 mL 环己烷和 100 mL 正己烷,混匀。
 - 4.11 10 种有机磷农药标准品:纯度均≥95%。
 - 4.12 标准储备溶液:分别准确称取适量的每种农药标准品(见附录 A),用丙酮分别配制成浓度为 100 μg/mL~1 000 μg/mL 的标准储备溶液。
 - 4.13 混合标准工作溶液:根据需要再用丙酮逐级稀释成适用浓度的系列混合标准工作溶液。
 - 4.14 氟罗里硅土固相萃取柱:Florisil,500 mg,6 mL,或相当者。
 - 4.15 石墨化炭黑固相萃取柱:ENVI-Carb,250 mg,6 mL,或相当者,使用前用 6 mL 乙酸乙酯-正己烷预淋洗。

- 4.16 有机相微孔滤膜:0.45 μm。
- 4.17 石墨化炭黑:60目~80目。

5 仪器与设备

- 5.1 气相色谱-质谱仪:配有电子轰击源(EI)。
- 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 具塞锥形瓶:250 mL。
- 5.7 分液漏斗:250 mL。
- 5.8 浓缩瓶:250 mL。
- 5.9 离心机:4 000 r/min 以上。

6 试样制备与保存

6.1 试样制备

取代表性样品约 1 kg,取可食部分,经捣碎机充分捣碎均匀,装入洁净容器,密封,标明标记。

6.2 试样保存

试样于-18℃保存。在抽样及制样的操作过程中,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

称取解冻后的试样 20 g(精确到 0.01 g)于 250 mL 具塞锥形瓶中,加入 20 mL 水和 100 mL 丙酮(4.1),均质提取 3 min。将提取液过滤,残渣再用 50 mL 丙酮重复提取一次,合并滤液于 250 mL 浓缩瓶中,于 40℃水浴中浓缩至约 20 mL。

将浓缩提取液转移至 250 mL 分液漏斗中,加入 150 mL 氯化钠水溶液(4.8)和 50 mL 二氯甲烷(4.2),振摇 3 min,静置分层,收集二氯甲烷相。水相再用 50 mL 二氯甲烷重复提取两次,合并二氯甲烷相。经无水硫酸钠脱水,收集于 250 mL 浓缩瓶中,于 40℃水浴中浓缩至近干。加入 10 mL 环己烷-乙酸乙酯(4.10)溶解残渣,用 0.45 μm 滤膜过滤,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:23 min~31 min。

7.2.1.2 凝胶色谱净化步骤

将 10 mL 待净化液按 7.2.1.1 规定的条件进行净化,收集 23 min~31 min 区间的组分,于 40 ℃下浓缩至近干,并用 2 mL 乙酸乙酯-正己烷溶解残渣,待固相萃取净化。

7.2.2 固相萃取(SPE)净化

将石墨化炭黑固相萃取柱(对于色素较深试样,在石墨化炭黑固相萃取柱上加 1.5 cm 高的石墨化炭黑(4.17))用 6 mL 乙酸乙酯-正己烷预淋洗,弃去淋洗液;将 2 mL 待净化液倾入上述连接柱中,并用 3 mL 乙酸乙酯-正己烷分 3 次洗涤浓缩瓶,将洗涤液倾入石墨化炭黑固相萃取柱中,再用 12 mL 乙酸乙酯-正己烷洗脱,收集上述洗脱液至浓缩瓶中,于 40 ℃水浴中旋转蒸发至近干,用乙酸乙酯溶解并定容至 1.0 mL,供气相色谱-质谱测定和确证。

7.3 测定

7.3.1 气相色谱-质谱条件

气相色谱-质谱条件如下:

- a) 色谱柱:30 m×0.25 mm(内径),膜厚 0.25 μm,DB-5MS 石英毛细管柱,或相当者;
- b) 色谱柱温度:50 ℃(2 min)^{30 ℃/min} 180 ℃(10 min)^{30 ℃/min} 270 ℃(10 min);
- c) 进样口温度:280 ℃;
- d) 色谱-质谱接口温度:270 ℃;
- e) 载气:氦气,纯度≥99.999%,流速 1.2 mL/min;
- f) 进样量:1 μL;
- g) 进样方式:无分流进样,1.5 min 后开阀;
- h) 电离方式:EI;
- i) 电离能量:70 eV;
- j) 测定方式:选择离子监测方式;
- k) 选择监测离子(m/z):见表 1 和参见附录 B;
- l) 溶剂延迟:5 min;
- m) 离子源温度:150 ℃;
- n) 四级杆温度:200 ℃。

表 1 选择离子监测方式的质谱参数表

通道	时间/min	选择离子/amu
1	5.00	109,125,137,145,179,185,199,220,270,285,304
2	17.00	109,127,158,169,214,235,245,247,258,260,261,263,285,286,314
3	19.00	153,125,384,226,210,334

7.3.2 气相色谱-质谱检测及确证

根据样液中被测物含量情况,选定浓度相近的标准工作溶液,对标准工作溶液与样液等体积参插进样测定,标准工作溶液和待测样液中每种有机磷农药的响应值均应在仪器检测的线性范围内。

如果样液与标准工作溶液的选择离子色谱图中,在相同保留时间有色谱峰出现,则根据附录 B 中每种有机磷农药选择离子的种类及其丰度比进行确证。在上述气相色谱-质谱条件下,9 种有机磷农药标准物的参考保留时间和气相色谱-质谱选择离子色谱图参见附录 B 和附录 C 中图 C.1。

7.4 结果计算和表述

试样中每种有机磷农药残留量按式(1)计算:

$$X_i = \frac{A_i \times c_i \times V}{A_{is} \times m} \dots\dots\dots (1)$$

式中:

- X_i —— 试样中每种有机磷农药残留量,单位为毫克每千克(mg/kg);
- A_i —— 样液中每种有机磷农药的峰面积(或峰高);
- c_i —— 标准工作液中每种有机磷农药的浓度,单位为微克每毫升($\mu\text{g/mL}$);
- V —— 样液最终定容体积,单位为毫升(mL);
- A_{is} —— 标准工作液中每种有机磷农药的峰面积(或峰高);
- m —— 最终样液代表的试样质量,单位为克(g)。

8 测定低限、回收率

8.1 测定低限

本方法对进出口动物源食品中 9 种有机磷农药残留量的测定低限参见附录 B。

8.2 回收率

- 8.2.1 清蒸猪肉罐头中 10 种有机磷农药在 0.02 mg/kg~1.00 mg/kg 时,回收率为 70.0%~94.9%。
- 8.2.2 猪肉中 9 种有机磷农药在 0.02 mg/kg~1.00 mg/kg 时,回收率为 71.2%~97.1%。
- 8.2.3 鸡肉中 9 种有机磷农药在 0.02 mg/kg~1.00 mg/kg 时,回收率为 74.3%~94.8%。
- 8.2.4 牛肉中 9 种有机磷农药在 0.02 mg/kg~1.00 mg/kg 时,回收率为 70.6%~96.9%。
- 8.2.5 鱼肉中 9 种有机磷农药在 0.02 mg/kg~1.00 mg/kg 时,回收率为 76.3%~93.3%。

附 录 A
(规范性附录)
9 种有机磷农药种类表

表 A.1 9 种有机磷农药种类表

序号	农药名称	英文名称	CAS 编号	化学分子式
1	敌敌畏	dichlorvos	000062-73-7	$C_4H_7Cl_2O_4P$
2	二嗪磷	diazinon	000333-41-5	$C_{12}H_{21}N_2O_3PS$
3	皮蝇磷	fenchlorphos	000299-84-3	$C_8H_8Cl_3O_3PS$
4	杀螟硫磷	fenitrothion	000122-14-5	$C_9H_{12}HO_5PS$
5	马拉硫磷	malathion	000121-75-5	$C_{10}H_{19}O_6PS_2$
6	毒死蜱	chlorpyrifos	002921-88-2	$C_9H_{11}Cl_3NO_3PS$
7	倍硫磷	fenthion	000055-38-9	$C_{10}H_{15}O_3PS_2$
8	对硫磷	parathion	000056-38-2	$C_{10}H_{14}NO_5PS$
9	乙硫磷	ethion	000563-12-2	$C_9H_{22}O_4P_2S_4$

附 录 B
(资料性附录)

9 种有机磷农药的保留时间、定量和定性选择离子及测定低限表

表 B.1 9 种有机磷农药的保留时间、定量和定性选择离子及测定低限表

序号	农药名称	保留时间/ min	特征碎片离子/amu			测定低限 μg/g
			定量	定性	丰度比	
1	敌敌畏	6.57	109	185,145,220	37 : 100 : 12 : 07	0.02
2	二嗪磷	12.64	179	137,199,304	62 : 100 : 29 : 11	0.02
3	皮蝇磷	16.43	285	125,109,270	100 : 38 : 56 : 68	0.02
4	杀螟硫磷	17.15	277	260,247,214	100 : 10 : 06 : 54	0.02
5	马拉硫磷	17.53	173	127,158,285	07 : 40 : 100 : 10	0.02
6	毒死蜱	17.68	197	314,258,286	63 : 68 : 34 : 100	0.01
7	倍硫磷	17.80	278	169,263,245	100 : 18 : 08 : 06	0.02
8	对硫磷	17.90	291	109,261,235	25 : 22 : 16 : 100	0.02
9	乙硫磷	20.16	231	153,125,384	16 : 10 : 100 : 06	0.02

附 录 C
(资料性附录)

9 种有机磷农药标准物质的气相色谱-质谱图

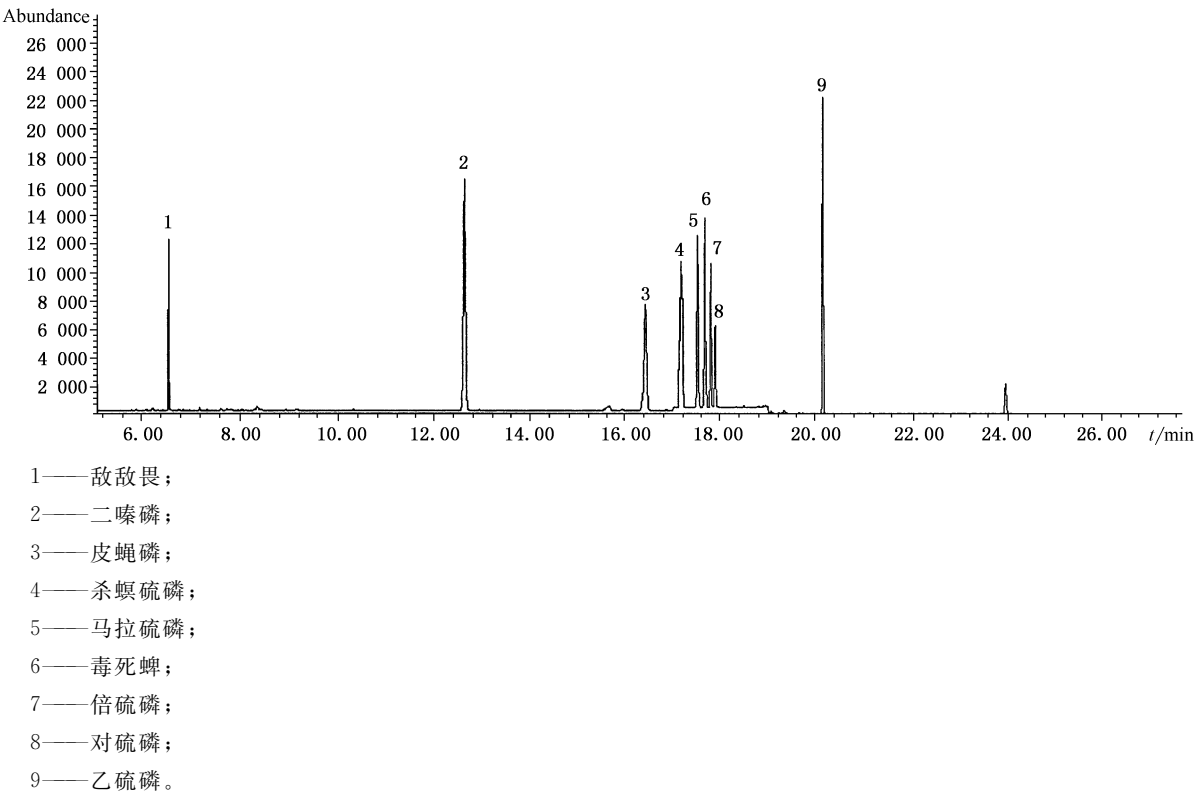


图 C.1 9 种有机磷农药标准物的气相色谱-质谱选择离子色谱图 (GC-MSD)

Foreword

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

This section of the original 2 standards (SN 0123—1992, SN 0214—1993) 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.

This main drafter of this standard is Li Aijun, Wang Mingtai, Mu Jun, Lu Iijun, Zhou Xiao, Wang Ying.

Determination of organophosphorus multiresidues in foodstuffs of animal origin for import and export—GC-MS method

1 Scope

This standard specifies the determination and confirmation of 10 organophosphorous pesticides residues by gas chromatography-mass spectrometry in foodstuffs of animal origin for import and export.

This standard is applicable to the determination and confirmation of residue content of 9 organophosphorous pesticides in canned steamed pork, pork, chicken, beef, fish for import and 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 are extracted with water-acetone. The extract is partitioned with dichloromethane. Cleaned up by passing through on GPC and active carbon column. The elutes solution is evaporated and made up to a definite volume. Determination and confirmation is made by GC-MS, using external standard method.

4 Reagents and materials

Unless otherwise specified, all the reagents used should be analytically pure, “water” is the first level water described by GB/T 6682.

- 4.1 Acetone;Pesticide grade.
- 4.2 Dichloromethane;Pesticide grade.
- 4.3 Cyclohexane;Pesticide grade.
- 4.4 Ethyl acetate;Pesticide grade.
- 4.5 *n*-Hexane;Pesticide grade.
- 4.6 Sodium chloride.
- 4.7 Anhydrous sodium sulfate;Ignite at 650 °C for 4 h,and keep in a tightly closed container.
- 4.8 Sodium chloride aqueous solution;20 g/L.
- 4.9 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.
- 4.10 Ethyl acetate-Cyclohexane(1 + 1, V/V);Volume 100 mL ethyl acetate into 250 mL erlenmeyer flask,then add 100 mL cyclohexane,mix them.
- 4.11 Organophosphrous pesticides standard:Purity $\geq 95\%$.
- 4.12 Standard stock solution: Accurately weigh an adequate amount of organophosphrous pesticides standard (see annex A) and dissolve in a small volume of acetone. Dilute with acetone to form a standard stock solution of 100 $\mu\text{g/mL}$ ~1 000 $\mu\text{g/mL}$ in concentration.
- 4.13 Standard working solution: Then dilute the standard stock solution with acetone to the required concentration as the standard working solution.
- 4.14 Florisil SPE tube;Florisil,500 mg,6 mL,or equivalent.
- 4.15 Active carbon SPE tube;ENVI-Carb,250 mg,6 mL,or equivalent.
- 4.16 Membrane filter;0.45 μm .
- 4.17 Graphitic carbon;60 mesh~80 mesh.

5 Apparatus and equipment

- 5.1 GC-MS:Equipped with electro-Impact source (EI).

- 5.2 Electronic balance; Accurate to 0.000 1 g.
- 5.3 Gel permeation chromatograph (GPC) equipped with isocratic pump and fraction collector.
- 5.4 Homogenizer.
- 5.5 Rotary vacuum evaporator.
- 5.6 Separator funnel; 250 mL.
- 5.7 Conical flask; 250 mL, with stopper.
- 5.8 Concentrate bottle; 250 mL.
- 5.9 Centrifuge; 4 000 r/min.

6 Preparation and storage of test sample

6.1 Preparation of test sample

Take approximately 1 kg of representative sample. Collect the edible pieces. Crush with a crusher. Mix thoroughly. Put in clean containers. Seal and label them.

6.2 Storage of test sample

The test samples shall be frozen and stored below $-18\text{ }^{\circ}\text{C}$. While sampling and preparing sample, please avoid contamination or any factors that may change residue content.

7 Procedure

7.1 Extraction

Weigh ca 20 g (accurate to 0.01 g) of the test sample into a 250 mL conical flask with stopper, add 20 mL of water and 100 mL of acetone (3.1), extract for 3 min on a high speed homogenizer. Filter the extract into a 250 mL concentrate bottle. Extract the residue with 50 mL of acetone once more, filter and combine the washings in the same concentrate bottle, evaporate to 20 mL in a rotary evaporator with a bath temperature below $40\text{ }^{\circ}\text{C}$.

Transfer the concentrated solution into a 250 mL separator funnel, add 150 mL of sodium chloride aqueous solution and 50 mL of dichloromethane, shake for 3 min and set aside for separating. Collect the dichloromethane phase. The water phase is again extracted with $2 \times 50\text{ mL}$ of dichloromethane. Combined the dichloromethane phases, and let pass through a column of anhydrous sodium sulfate to

remove the water. Collect the effluent in a 250 mL concentrate bottle and evaporate to near dryness in a rotary evaporator with a bath temperature below 40 °C. Dissolve the residue with 5 mL of cyclohexane-ethyl acetate (1 + 1), and filter through 0.45 μm membrane filter.

7.2 Clean up

7.2.1 GPC Clean 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: 23 min ~ 31 min.

7.2.1.2 GPC clean up operating

Transfer the above 10 mL solution into an GPC column, proceed as section 7.2.2.1. Combined the eluates in the 50 mL concentrate bottle, evaporate to dryness in a rotary evaporator with a bath temperature below 40 °C. Dissolve the residue with 2 mL of *n*-hexane.

7.2.2 SPE Clean up

Rinse the an ENVI-Carb column with 6 mL of *n*-hexane before use (for samples containing much colorants, add 1.5 cm high graphic carbon (4.17) in the ENVI-Carb column). Discard the washings. Transfer 2 mL of the sample solution into the upper column. Elute with 3 mL of *n*-hexane-ethyl acetate (1 + 1). Collect eluates into a concentrate bottle. Evaporate to nearly dry in 40 °C water bath. Dissolve the residue and dilute exactly to 1.0 mL with ethyl acetate for GC-MS determination and confirmation.

7.3 Determination

7.3.1 GC-MS operating condition

- a) Chromatographic column: 30 m × 0.25 mm (i. d.), 0.25 μm film thickness, DB-5 MS, silica

- capillary column or equivalent;
- b) Column temperature:50 °C (2 min) $\xrightarrow{30\text{ }^{\circ}\text{C/min}}$ 180 °C (10 min) $\xrightarrow{30\text{ }^{\circ}\text{C/min}}$ 270 °C (10 min) ;
- c) Injection port temperature:280 °C ;
- d) Interface temperature:270 °C ;
- e) Carrier gas:Helium,purity $\geq 99.999\%$,flow rate 1.2 mL/min;
- f) Injection volume:1 μL ;
- g) Injection mode:Splitless,purge on after 1.5 min;
- h) Electron ionization mode:EI;
- i) Ionization energy:70 eV;
- j) Determination mode:SIM mode;
- k) Selected monitoring ion (m/z) :See table 1 and annex B;
- l) Solvent protection delay:5 min;
- m) Ion source temperature:150 °C ;
- n) Temperature:200 °C .

Table 1—Parameter of selected monitoring ion in MS table

Channels	Time/min	Selected monitoring ion/amu
1	5.00	109,125,137,145,179,185,199,220,270,285,304
2	17.00	109,127,158,169,214,235,245,247,258,260,261,263,285,286,314
3	19.00	153,125,384,226,210,334

7.3.2 GC-MS determination and confirmation

According to the approximate concentration of the pesticide in the sample solution, select the standard working solution with similar peak height to that of the sample solution. The standard working solution should be randomly injected in-between the injections of the sample solution of equal volume. The responses of per organophosphrous pesticides in the standard working solution and sample solution should be within the linear range of the instrumental detection.

If there is any peak of sample solution appeared at the same retention time as such peak of the standard solution, it must be confirmed by selected monitoring ions (m/z) of species and abundance ratio, see annex B. Under the above GC-MS condition, the retention time of 9 organophosphorous pesticides for GC-MS chromatogram (TIC) of the standard, see annex B and in annex C.

7.4 Calculation and expression of the result

Calculate the content of per organophosphorous pesticides residues in the test sample by GC-MS data processor or according to the formula.

$$X_i = \frac{A_i \times c_i \times V}{A_{is} \times m} \dots\dots\dots (1)$$

where

- X_i —the residue content of per organophosphorous pesticides in the test sample, mg/kg;
- A_i —the peak area (height) of per organophosphorous pesticides in the sample solution;
- c_i —the concentration of per organophosphorous pesticides in the standard working solution, $\mu\text{g/mL}$;
- V —the final volume of the sample solution, mL;
- A_{is} —the peak area (height) of per organophosphorous pesticides in the standard working solution;
- m —the corresponding mass of the test sample in the final sample solution, g.

8 Limit of determination and recovery

8.1 Limit of determination

The limit of determination of this method for organophosphorous pesticides residues in foodstuffs of animal origin for import and export, see annex B.

8.2 Recovery

- 8.2.1 The fortifying concentrations 0.02 mg/kg ~ 1.00 mg/kg of 9 organophosphorous pesticides in canned steamed pork and its recovery is 70.0% ~ 94.9%.
- 8.2.2 The fortifying concentrations 0.02 mg/kg ~ 1.00 mg/kg of 9 organophosphorous pesticides in pork and its recovery is 71.2% ~ 97.1%.
- 8.2.3 The fortifying concentrations 0.02 mg/kg ~ 1.00 mg/kg of 9 organophosphorous pesticides in chicken, and its recovery is 74.3 ~ 94.8%.

8.2.4 The fortifying concentrations 0.02 mg/kg~1.00 mg/kg of 9 organophosphorous pesticides in beef and its recovery is 70.6%~96.9%.

8.2.5 The fortifying concentrations 0.02 mg/kg~1.00 mg/kg of 9 organophosphorous pesticides in fish and its recovery is 76.3~93.3%.

Annex A
(normative)

The species of 9 organophosphorous pesticides

Table A. 1—Species of 9 organophosphorous pesticides

Order number	Pesticide name in chinese	Pesticide name in english	CAS. No	Chemical formula
1	敌敌畏	dichlorvos	000062-73-7	C ₄ H ₇ Cl ₂ O ₄ P
2	二嗪磷	diazinon	000333-41-5	C ₁₂ H ₂₁ N ₂ O ₃ PS
3	皮蝇磷	fenchlorphos	000299-84-3	C ₈ H ₈ Cl ₃ O ₃ PS
4	杀螟硫磷	fenitrothion	000122-14-5	C ₉ H ₁₂ HO ₅ PS
5	马拉硫磷	malathion	000121-75-5	C ₁₀ H ₁₉ O ₆ PS ₂
6	毒死蜱	chlorpyrifos	002921-88-2	C ₉ H ₁₁ Cl ₃ NO ₃ PS
7	倍硫磷	fenthion	000055-38-9	C ₁₀ H ₁₅ O ₃ PS ₂
8	对硫磷	parathion	000056-38-2	C ₁₀ H ₁₄ NO ₅ PS
9	乙硫磷	ethion	000563-12-2	C ₉ H ₂₂ O ₄ P ₂ S ₄

Annex B
(informative)

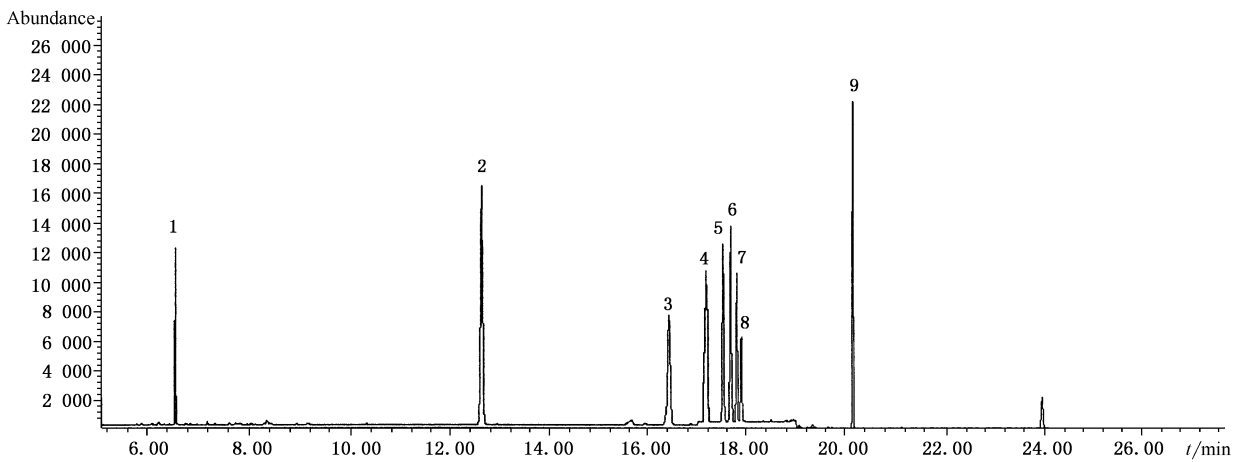
The retention time,determination and confirmation selected monitoring ion
and limit of determination of 9 organophosphrous pesticides

Table B. 1—Retention time,determination and confirmation selected monitoring ion
and limit of determination of 9 organophosphrous pesticides

No.	Pesticides	t / min	Characteristic fragment ion/amu			Limit μg/g
			determination	confirmation	abundance ratio	
1	Dichlorvos	6. 57	109	185,145,220	37 : 100 : 12 : 07	0. 02
2	Diazinon	12. 64	179	137,199,304	62 : 100 : 29 : 11	0. 02
3	Fenchlorphos	16. 43	285	125,109,270	100 : 38 : 56 : 68	0. 02
4	Fenitrothion	17. 15	277	260,247,214	100 : 10 : 06 : 54	0. 02
5	Malathion	17. 53	173	127,158,285	07 : 40 : 100 : 10	0. 02
6	Fenthion	17. 68	197	314,258,286	63 : 68 : 34 : 100	0. 01
7	Chlorpyrifos	17. 80	278	169,263,245	100 : 18 : 08 : 06	0. 02
8	Ethion	17. 90	291	109,261,235	25 : 22 : 16 : 100	0. 02
9	Parathion	20. 16	231	153,125,384	16 : 10 : 100 : 06	0. 02

Annex C
(informative)

GC-MS chromatogram (TIC) of the 9 organophosphorous pesticides standard



- 1—Dichlorvos;
- 2—Diazinon;
- 3—Diazinon;
- 4—Fenchlorphos;
- 5—Malathion;
- 6—Fenthion;
- 7—Chlorpyrifos;
- 8—Ethion;
- 9—Parathion。

Figure C. 1—GC-MS chromatogram (TIC) of the 9 organophosphorous pesticides standard