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中华人民共和国出入境检验检疫行业标准

SN/T 1738—2006

进出口粮谷和油籽中虫酰肼残留量的 检验方法 气相色谱串联质谱法

Inspection of tebufenozide residues in cereals and
oil seeds for import and export—
Gas chromatography mass spectrometry method

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前 言

本标准的附录 A 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。

本标准起草单位：中华人民共和国吉林出入境检验检疫局。

本标准主要起草人：王明泰、牟 峻、邹明强、卢利军、吕清双、刘洪涛。

本标准系首次发布的出入境检验检疫行业标准。

进出口粮谷和油籽中虫酰肼残留量的 检验方法 气相色谱串联质谱法

1 范围

本标准规定了进出口粮谷和油籽中虫酰肼残留量检验的抽样、制样和气相色谱-质谱检测方法。

本标准适用于进出口糙米、玉米、大豆、花生仁中虫酰肼残留量的检验。

2 抽样和制样

2.1 检验批

以不超过 200 t 为一检验批。200 t 袋装糙米约 4 000 袋；袋装玉米或大豆约 2 200 袋；袋装花生仁约 2 400 袋。玉米或大豆有时为散装品。

同一检验批的商品应具有相同特征，如包装、标记、产地、规格和等级等。

2.2 抽样数量

2.2.1 袋装货品

按式(1)计算抽样袋数：

$$a = \sqrt{N} \dots\dots\dots (1)$$

式中：

N ——全批袋数；

a ——抽样袋数。

注： a 值取整数，小数部分向前进位为整数。

2.2.2 散积货品(玉米或大豆)

货堆高度不超过 2 m。按货堆面积划区设点。以 50 m² 为一个取样区，每区设中心及四角(距边线 1 m 处)5 个点。每增加一个取样区，增设 3 个点。

2.3 抽样工具

2.3.1 金属单管取样器：全长 55 cm(包括手柄)，直径 1.5 cm~2.0 cm，沟槽长度应超过袋对角线长度的一半。

2.3.2 金属双套管取样器：全长分 1 m、2 m(均包括手柄)两种。内、外管同部位分段开几个槽口，每个槽口长 15 cm~20 cm，口宽 2.0 cm~2.5 cm。内管的内径为 2.5 cm~3.0 cm；取样器的探头长约 7 cm。

2.3.3 取样铲或取样勺。

2.3.4 分样板。

2.3.5 盛样器：筒或袋，可密封。

2.3.6 分样布或适用铺垫物。

2.4 抽样方法

2.4.1 袋装抽样

2.4.1.1 倒包抽样：从堆垛的各部位随机抽取 2.2.1 规定的应抽样袋数的 10%(每批一般不少于 3 袋)，将袋口缝线全部拆开，平置于分样布或其他洁净的铺垫物上，双手紧握袋底两角，提起约成 45°倾角，倒拖约 1 m，使袋内货物全部倒出。查看袋内和袋间品质是否均匀。确认情况正常后，用取样铲随机在各部位抽取样品，并立即将样品倒入盛样器内。每袋抽取样品的数量应基本一致。

2.4.1.2 袋内抽样:按 2.2.1 规定的应抽样袋数(扣除倒包抽样袋数),在堆垛四周的上、中、下各层以曲线形走向随机抽取。然后按糙米、玉米、大豆、花生仁,用下述方法进行取样:

对糙米,用金属单管取样器(2.3.1)槽口朝下,从每袋一角依斜对角方向插入袋内,然后将管槽旋转朝上,抽出取样器,立即将样品倒入盛样器内。

对玉米或大豆,用 1 m 长的金属双套管取样器(2.3.2),关闭槽口,从每袋一角依斜对角方向插入袋内,然后旋转内管以开启槽口,待样品流满内管后,再旋转内管以关闭槽口。抽出取样器,立即将样品倒入盛样器内。

对花生仁,将应抽各袋拆开袋口缝线 3 针~5 针,用取样勺从开口处抽取样品。立即缝好袋口,并将所取样品倒入盛样器内。

每袋抽取样品的数量应与 2.4.1.1 基本一致。每批所抽取的样品总量应不少于 4 kg。

2.4.2 散积抽样

按 2.2.2 规定的取样点,逐点抽取样品。将取样器(2.3.2)槽口关闭,以倾斜 45°插入货堆至相应深度,旋转取样器内管以开启槽口,待样品流满内管后,再旋转内管以关闭槽口。抽出取样器,立即将样品倒入盛样器内,从各点所抽取样品的数量应基本保持一致。

每批所抽取的样品总量应不少于 4 kg。

2.4.3 大样缩分

袋装样品:合并从倒包和袋内抽样所取全部样品,倒于分样布上,用分样板按四分法缩分样品至不少于 2 kg,倒入盛样器内,加封后标明标记,并及时送交实验室。

散积样品:将抽取的全部样品,倒于分样板上,以下按上述袋装样品方法进行。

2.5 试样制备

2.5.1 制样工具

2.5.1.1 磨碎机。

2.5.1.2 粉碎机。

2.5.1.3 筛子:2.0 mm 圆孔筛。

2.5.1.4 分样板。

2.5.1.5 盛样瓶:具塞广口瓶。

2.5.2 制样方法

2.5.2.1 糙米、玉米或大豆试样制备:将样品按四分法缩分至 1 kg,用磨碎机(2.5.1.1)全部磨碎并通过 20 目筛。混匀,均分成两份作为试样,分装入洁净的盛样瓶内,密闭,标明标记。

2.5.2.2 花生仁试样制备:将样品按四分法缩分至 500 g,用粉碎机(2.5.1.2)粉碎,使全部通过 2.0 mm 圆孔筛。混匀,均分成两份作为试样,分装入洁净的盛样瓶内,密闭,标明标记。

2.6 试样保存

将试样于 0℃~4℃ 以下避光保存。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。

3 测定方法

3.1 方法提要

试样用水-丙酮提取,提取液经二氯甲烷液-液分配后,以弗罗里硅土柱净化,用气相色谱-质谱测定,外标法定量。

3.2 试剂和材料

除另有规定外,所用试剂均为分析纯,水为二次蒸馏水。

3.2.1 丙酮:重蒸馏。

3.2.2 二氯甲烷:重蒸馏。

- 3.2.3 正己烷:重蒸馏。
- 3.2.4 乙酸乙酯:重蒸馏。
- 3.2.5 无水硫酸钠:650℃灼烧4 h,贮于密封容器中备用。
- 3.2.6 硫酸钠水溶液:20 g/L。
- 3.2.7 弗罗里硅土:层析用,100目~200目,650℃灼烧5 h,用前于130℃活化4 h,冷却后加入2%(质量分数)水脱活,贮于密封容器中备用。
- 3.2.8 虫酰肼标准品:纯度≥99%。
- 3.2.9 虫酰肼标准溶液:准确称取适量的虫酰肼标准品,用少量的丙酮溶解,并以丙酮配制成浓度为1.00 mg/mL的标准储备液。根据需要再用丙酮稀释成适用浓度的标准工作溶液。

3.3 仪器和设备

- 3.3.1 气相色谱-质谱仪:配有质量选择检测器(MSD)。
- 3.3.2 振荡器。
- 3.3.3 旋转蒸发器。
- 3.3.4 无水硫酸钠柱:7.5 cm×1.5 cm(内径),内装5 cm高无水硫酸钠。
- 3.3.5 弗罗里硅土柱:25 cm×1.5 cm(内径),自下而上依次填装2 cm高无水硫酸钠、10 g弗罗里硅土、2 cm高无水硫酸钠。使用前用50 mL正己烷预淋洗。

3.4 测定步骤

3.4.1 提取

称取试样约20 g(精确至0.1 g)于250 mL具塞锥形瓶中,加入40 mL水,放置2 h。然后加入100 mL丙酮,振荡提取30 min。将提取液过滤于500 mL分液漏斗中,残渣再用50 mL丙酮重复提取一次,合并滤液于上述分液漏斗中。

加入200 mL硫酸钠水溶液和100 mL二氯甲烷,振摇3 min,静置分层,收集二氯甲烷相。水相再用2×50 mL二氯甲烷重复提取两次,合并二氯甲烷相。经无水硫酸钠柱脱水,收集于250 mL浓缩瓶中,于40℃水浴中旋转浓缩至近干,加入5 mL正己烷溶解残渣。

3.4.2 净化

将样液倾入弗罗里硅土柱中,依次用50 mL正己烷和50 mL乙酸乙酯+正己烷(1+9)进行淋洗,弃去流出液,再用50 mL乙酸乙酯+正己烷(1+1)进行洗脱,收集全部洗脱液于250 mL浓缩瓶中,于40℃水浴中旋转浓缩至近干,用丙酮溶解并定容至5 mL,供气相色谱-质谱测定。

3.4.3 测定

3.4.3.1 气相色谱-质谱条件

- 色谱柱:30 m×0.25 mm(内径),膜厚0.25 μm,DB-17石英毛细管柱,或相当者;
- 色谱柱温度:50℃(2 min) $\xrightarrow{30^{\circ}\text{C}/\text{min}}$ 200℃(1 min) $\xrightarrow{10^{\circ}\text{C}/\text{min}}$ 270℃(7 min);
- 进样口温度:270℃;
- 色谱-质谱接口温度:260℃;
- 载气:氦气,纯度≥99.999%,1.2 mL/min;
- 进样量:1 μL;
- 进样方式:无分流进样,1.5 min后开阀;
- 电离方式:EI;
- 电离能量:70 eV;
- 电子倍增器电压:1.6 kV;
- 测定方式:选择离子监测方式;
- 选择监测离子(m/z):193、207、278;

m) 溶剂延迟:5 min。

3.4.3.2 气相色谱-质谱测定

根据样液中被测农药含量,选定浓度相近的标准工作溶液。标准工作溶液和待测样液中农药的响应值均应在仪器检测的线性范围内。对标准工作溶液与样液等体积参插进样测定。在上述气相色谱-质谱条件下,虫酰肼的保留时间约为 17 min。标准品的气相色谱-质谱选择离子色谱图和质谱图参见附录 A 中图 A.1 和附录 B 中图 B.1。

3.4.3.3 气相色谱-质谱确证

对标准溶液及样液均按 3.4.3.1 规定的条件进行测定时,如果样液与标准溶液的选择离子图中,在相同保留时间有峰出现,则根据选择离子 m/z 193、207、278(其丰度比 86:35:100)对其确证。

3.4.4 空白试验

除不加试样外,均按上述测定步骤进行。

3.5 结果计算和表述

用色谱数据处理机或按式(2)计算试样中虫酰肼残留量:

$$X = \frac{A \cdot c \cdot V}{A_s \cdot m} \dots\dots\dots (2)$$

式中:

X ——试样中虫酰肼残留量,单位为毫克每千克(mg/kg);

A ——样液中虫酰肼的色谱峰高,单位为毫米(mm);

A_s ——标准工作液中虫酰肼的色谱峰高,单位为毫米(mm);

c ——标准工作液中虫酰肼的浓度,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);

m ——最终样液所代表的试样质量,单位为克(g)。

4 测定低限、回收率

4.1 测定低限

本方法的测定低限为 0.10 mg/kg。

4.2 回收率

4.2.1 糙米中虫酰肼的添加浓度及其回收率实验数据:

在 0.10 mg/kg~1.00 mg/kg 时,回收率为 88.2%~95.4%。

4.2.2 玉米中虫酰肼的添加浓度及其回收率实验数据:

在 0.10 mg/kg~1.00 mg/kg 时,回收率为 88.6%~94.4%。

4.2.3 大豆中虫酰肼的添加浓度及其回收率实验数据:

在 0.10 mg/kg~1.00 mg/kg 时,回收率为 92.7%~97.7%。

4.2.4 花生仁中虫酰肼的添加浓度及其回收率实验数据:

在 0.10 mg/kg~1.00 mg/kg 时,回收率为 91.8%~94.1%。

附 录 A
(资料性附录)

虫酰肼标准品的气相色谱-质谱选择离子色谱图

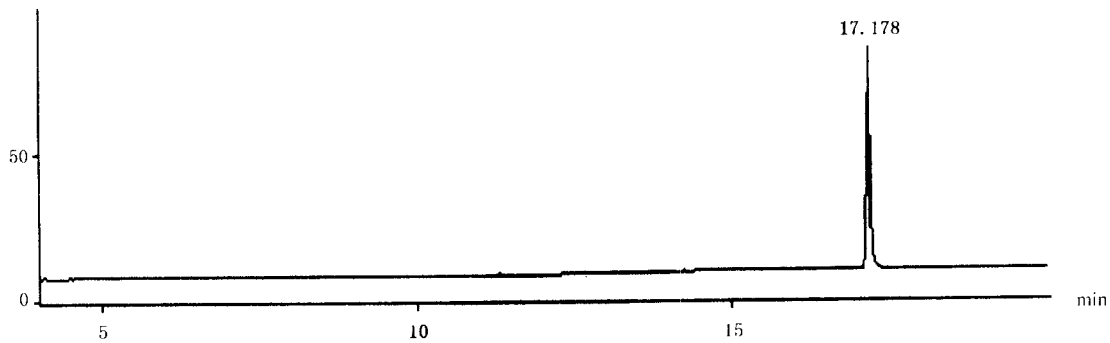


图 A.1 虫酰肼标准品的气相色谱-质谱选择离子色谱图

附 录 B
(资料性附录)

虫酰肼标准物的气相色谱-质谱图

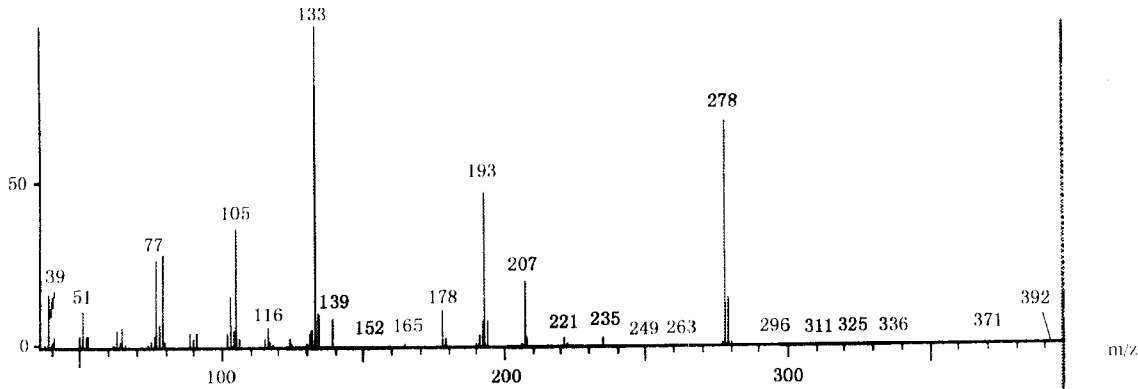


图 B.1 虫酰肼标准物的气相色谱-质谱图

Foreword

Annex A of this standard are informative annex.

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 Wang Mingtai, Mu Jun, Zou Mingqiang, Lu Lijun, Lu Qingshuang, Liu Hongtao.

This standard is a professional standard promulgated for the first time.

Note: This English version, a translation from the Chinese text, is solely for guidance.

Inspection of tebufenozide residues in cereals and oil seeds for import and export— Gas chromatography mass spectrometry method

1 Scope

This standard specifies the method of sampling, sample preparation and determination of tebufenozide residues by gas chromatography-mass spectrometry in cereals and oil seeds for import and export.

This standard is applicable to the determination of residue content of tebufenozide in unpolished rice, maize, soybean and peanut for import and export.

2 Sampling and sample preparation

2.1 Inspection lot

Each inspection lot should not exceed 200 t. An inspection lot of 200 t, for unpolished rice in bags shall be ca 4 000 bags; for maize or soybean in bags shall be ca 2 200 bags; and for peanut in bags shall be ca 2 400 bags. The cargo of maize or soybean is sometimes in bulk.

The characteristics of the cargo within the same inspection lot, such as packing, mark, origin, specification, grade etc., should be the same.

2.2 Quantity of sample taken

2.2.1 Cargo in bags

Calculate the number of bags to be taken by formula (1).

$$a = \sqrt{N} \dots\dots\dots (1)$$

where;

N ——total number of bags in a lot;

a ——number of bags to be taken.

Note: If value a is with decimal, round off the decimal part, which is added as unity to the integral part of a .

2.2.2 Cargo in bulk (maize or soybean)

The height of the cargo pile should not exceed 2 m. Set up areas and spots for sampling on the pile

surface, 50 m² is considered as an area, in which 5 spots shall be fixed, one in the center and four at four corners (1 m from the margins) of the area. For an additional area, three more sampling spots shall be fixed.

2.3 Sampling tools

2.3.1 Metallic sampler; Length (including handle): 55 cm; diameter: 1.5 cm-2.0 cm; groove length: longer than half the diagonal length of the bag.

2.3.2 Metallic double-casing sampler; length 1 m, 2 m (both including handle) with some slots on different sections and respectively at the same heights for both inner and outer casings; length of slots: 15 cm~20 cm, width of the slots: 2.0 cm~2.5 cm; inside diameter of the inner casing: 2.5 cm~3.0 cm; the probe length of the sampler: ca 7 cm.

2.3.3 Sampling shovel or ladle.

2.3.4 Plate for quartering.

2.3.5 Sample container; Can or bag, which can be sealed.

2.3.6 Cloth (or other suitable material) sheet; For sample dividing (quartering).

2.4 Sampling procedure

2.4.1 For cargo in bags

2.4.1.1 Sampling by emptying out: Draw 10 percent of the number of bags specified in 2.2.1 (not less than 3 bags) at any part of the pile at random. Unseam and open the bag, and lay it on a clean cloth sheet (or other clean sheet). Grasp tight two corners of the bag bottom and raise up to an angle of 45°, tug backward for ca 1 m until all content of the bag is emptied out. Check whether the quality of goods is uniform within and between the bags. After confirming the goods are in normal condition, scoop up the sample from different parts of the out-poured content at random, and promptly place in a sample container. The quantity of sample drawn from each bag should be basically the same.

2.4.1.2 Sampling from inside the bags: Draw the samples from the number of bags specified in 2.2.1 (by deducting the number of bags drawn in 2.4.1.1) as follows: Along the sine wave of the pile, draw the samples from the bags of upper, middle and lower parts around the pile at random. For unpolished rice or maize proceed as follows:

For unpolished rice, using the metallic sampler (2.3.1), insert it with its groove facing downward, diagonally into each bag, then turn the sampler 180°, draw out the sampler and promptly pour the sample into a container.

For maize or soybean, using the metallic double-casing sampler (2.3.2) (the slots should be closed while inserting in) diagonally into each bag. Turn the inner casing to open the slots so that the sample may fill up the inner tube. Again turn the inner casing to close the slots and draw out the sampler. Promptly pour the sample into a sample container.

For peanut, partially unseam (3 stitches~5 stitches) the bag mouth, scoop out the sample from each bag (with a ladle) at the bag opening. Promptly close the bag by seaming, and pour the sample into a sample container.

The amount of sample drawn from each bag should be basically the same as in 2.4.1.1. The total weight of the sample of each lot should be not less than 4 kg.

2.4.2 Sampling from the cargo in bulk:

Insert the double-casing sampler (2.3.2) successively into the pile at the spots specified in 2.2.2, to appropriate depth at 45° (the slots should be closed while inserting in). Turn the inner casing to open the slots so that the sample may fill up the inner tube. Again turn the inner casing to close the slots and draw out the sampler. Promptly pour the sample into a sample container. The amount of sample drawn from all the spots shall be basically the same.

The total weight of the sample of each lot should be not less than 4 kg.

2.4.3 Reduction of gross sample

For cargo in bags: Pour all the samples [from both a) and b)] on a clean sheet. Reduce to not less than 2 kg by quartering with a plate. Place in a sample container, seal, label and send to the laboratory in time.

For cargo in bulk: Pour all the drawn sample onto a clean sheet and proceed as for cargo in bags described above.

2.5 Preparation of test sample

2.5.1 Sampling tools

2.5.1.1 Grinder.

2.5.1.2 Pulverizer.

2.5.1.3 Sieve: 2.0 mm round-hole sieve.

2.5.1.4 Plate for quartering.

2.5.1.5 Sample container: Wide-mouth bottle, with ground stopper.

2.5.2 Procedure

2.5.2.1 For unpolished rice, maize or soybean: Reduce by quartering to ca 1 kg. Grind with a grinder(2.5.1.1) to let all pass through a 20-mesh sieve. Mix thoroughly and divide into two equal portions, place in clean sample containers as the test samples, seal and label.

2.5.2.2 For peanut: Reduce by quartering to ca 500 g, pulverize with a pulverizer(2.5.1.2) to let all pass through a 2.0 mm round-hole sieve. Mix thoroughly and divide into two equal portions as the test samples, place in clean sample containers, seal and label.

2.6 Storage of test sample

The test samples should be stored 0℃ ~4℃ and kept away from light. In the course of sampling and sample preparation, precautions must be taken to avoid contamination or any factors that may cause the change of residue content.

3 Method of determination

3.1 Principle

The test sample are extracted with water-acetone, the extract is partitioned with dichloromethane. Cleaned up by passing through a on Florisil column. Determination is made by means of a GC-MS, using external standard method.

3.2 Reagents and materials

Unless otherwise specified, all the reagents used should be analytically pure, "water" is distilled water.

3.2.1 Acetone: Redistilled.

3.2.2 Dichloromethane: Redistilled.

3.2.3 *n*-hexane: Redistilled.

3.2.4 Ethyl acetate: Redistilled.

3.2.5 Anhydrous sodium sulfate: Ignite at 650℃ for 4 h, and keep in a tightly closed container.

3.2.6 Sodium sulfate aqueous solution: 20 g/L.

3.2.7 Florisil: For chromatography, 100 mesh~200 mesh, ignite at 650°C for 5 h. Before use, heat at 130°C for 4 h and deactivate by adding 2% (m/m) water after cooling. Keep in a tightly closed container.

3.2.8 Tebufenozide standard: Purity $\geq$ 99%.

3.2.9 Tebufenozide standard solution: Accurately weigh an adequate amount of tebufenozide standard and dissolve in a small volume of acetone. Dilute with acetone to form a standard stock solution of 1.00 mg/mL in concentration. Then dilute the standard stock solution with acetone to the required concentration as the standard working solution.

3.3 Apparatus and equipment

3.3.1 Gas chromatograph equipped with mass selective detector(MSD).

3.3.2 Shaker.

3.3.3 Rotary vacuum evaporator.

3.3.4 Column of anhydrous sodium sulfate: 7.5 cm $\times$ 1.5 cm(i. d.), packed with 5 cm height of anhydrous sodium sulfate.

3.3.5 Florisil column: 25 cm $\times$ 1.5 cm(i. d.), add in sequence 2 cm (height) of anhydrous sodium sulfate, 10 g of Florisil, 2 cm (height) of anhydrous sodium sulfate. Rinse the column with 50 mL of *n*-hexane before use.

3.4 Procedure

3.4.1 Extraction

Weigh ca 20 g (accurate to 0.1 g) of the test sample into a 250 mL conical flask with stopper, add 40 mL of water and let stand for 2 h. Add 100 mL of acetone, extract for 30 min on a shaker. Filter the extract into a 500 mL separator. Extract the residue with 50 mL of acetone once more, filter and combine the washings in the same separator.

Add 200 mL of sodium sulfate aqueous solution and 100 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 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 10 mL of *n*-hexane.

3.4.2 Cleanup

Transfer the above solution into an Florisil column. Wash the column with 50 mL of *n*-hexane and 50 mL of ethyl acetate-*n*-hexane(1+9) and discard the effluent. Elute with 100 mL of ethyl acetate-*n*-hexane(1+1), collect all the eluate in a 250 mL concentrate bottle and evaporate to dryness in a rotary evaporator with a bath temperature below 40°C. Dissolve the residue and dilute exactly to 5 mL with acetone for GC-MS determination.

3.4.3 Determination

3.4.3.1 GC-MS operating condition

- a) Chromatographic column: 30 m × 0.25 mm(i. d.), 0.25 μm film thickness, DB-17, silica capillary column or equivalent;
- b) Column temperature: 50°C (2 min) $\xrightarrow{30^{\circ}\text{C}/\text{min}}$ 200°C (1 min) $\xrightarrow{10^{\circ}\text{C}/\text{min}}$ 270°C (7 min);
- c) Injection port temperature: 270°C;
- d) Interface temperature: 260°C;
- e) Carrier gas: Helium, purity ≥ 99.999%, 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) Electronic multiplier voltage: 1.6 kV;
- k) Determination mode: SIM mode;
- l) Selected monitoring ion(*m/z*): 193, 207, 278;
- m) Solvent protection delay: 5 min.

3.4.3.2 GC-MS determination

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 responses of pesticide 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 the sample solution of equal volume. Under the above chromatographic condition, the retention time of tebufenozide is ca 17 min. For GC-MS chromatogram (TIC) and mass spectrum of the standard, see Figure A. 1 in annex A and Figure B. 1 in annex B.

3.4.3.3 GC-MS Confirmation

According to the operation condition assigned in 3.4.3.1, analyze the standard solution and sample solution. 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) 193, 207, 278 (abundance ratio is ca 86 : 35 : 100) with GC-MS.

3.4.4 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.

3.5 Calculation and expression of the result

Calculate the content of tebufenozide residues in the test sample by GC data processor or according to the formula (2).

$$X = \frac{A \cdot c \cdot V}{A_s \cdot m} \dots\dots\dots (2)$$

where:

- X — the residue content of tebufenozide in the test sample, mg/ kg;
- A — the peak height of tebufenozide in the sample solution, mm;
- A_s — the peak height of tebufenozide in the standard working solution, mm;
- c — the concentration of tebufenozide in the standard working solution, $\mu\text{g}/\text{mL}$;
- V — the final volume of the sample solution, mL;
- m — the corresponding mass of the test sample in the final sample solution, g.

4 Limit of determination and recovery

4.1 Limit of determination

The limit of determination of this method is 0.10 mg/ kg.

4.2 Recovery

4.2.1 According to the experimental data, the fortifying concentrations of tebufenozide in unpolished rice and its corresponding recoveries are:

0.10 mg/kg~1.00 mg/kg, the recovery 88.2%~95.4%.

4.2.2 According to the experimental data, the fortifying concentrations of tebufenozide in maize and its corresponding recoveries are:

0.10 mg/kg~1.00 mg/kg, the recovery 88.6%~93.4%.

4.2.3 According to the experimental data, the fortifying concentration of tebufenozide in soybean and its corresponding recoveries are:

0.10 mg/kg~1.00 mg/kg, the recovery 92.7%~97.7%.

4.2.4 According to the experimental data, the fortifying concentrations of tebufenozide in peanut and its corresponding recoveries are:

0.10 mg/kg~1.00 mg/kg, the recovery 91.8%~94.1%.

Annex A

(Informative)

GC-MS chromatogram(TIC)of the tebufenozide standard

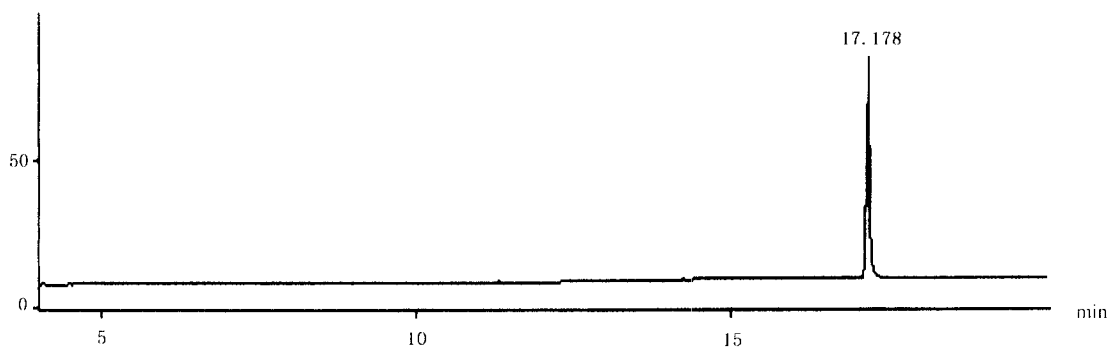


Figure A. 1 GC-MS chromatogram(TIC) of the tebufenozide standard

Annex B

(Informative)

GC-MS spectrum of the tebufenozide standard

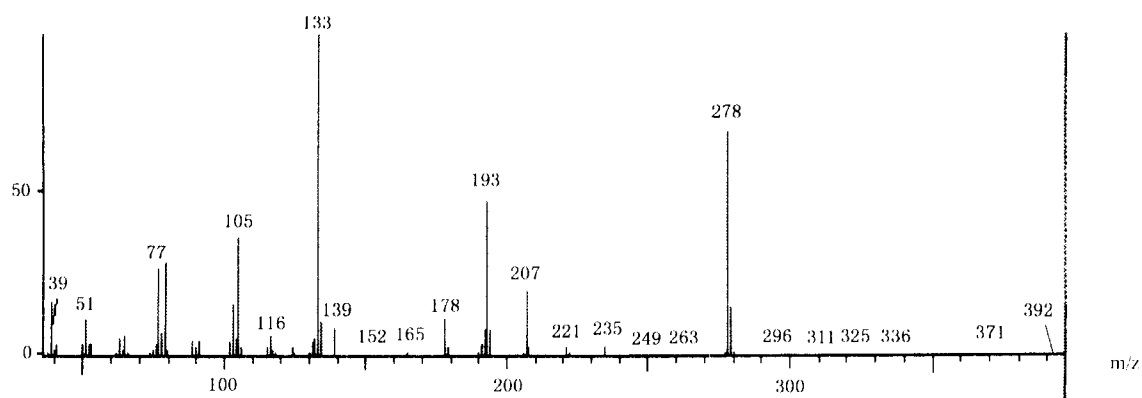


Figure B. 1 GC-MS spectrum of the tebufenozide standard

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行 业 标 准
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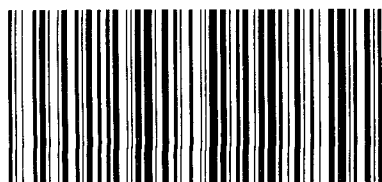
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