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中华人民共和国出入境检验检疫行业标准

SN/T 1968—2007

进出口食品中扑草净残留量检测方法 气相色谱-质谱法

Determination of prometryne residues in food for import and export—GC-MS Method

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前　言

本标准附录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：中国检验检疫科学研究院、中华人民共和国江苏出入境检验检疫局、中华人民共和国福建出入境检验检疫局。

本标准主要起草人：陈冬东、李淑娟、李晓娟、蔡慧霞、安娟、冯帆、李建中、唐英章。

本标准系首次发布的出入境检验检疫行业标准。

进出口食品中扑草净残留量检测方法 气相色谱-质谱法

1 范围

本标准规定了食品中扑草净残留量检测的制样和气相色谱-质谱检测方法。

本标准适用于大米、花生、胡萝卜、西兰花、西红柿、洋葱、蘑菇、苹果、柑橘、板栗、鸡肉、牛肉、鸡肾、紫菜中扑草净残留量的测定和确证。

2 方法提要

试样中残留的扑草净用乙腈提取，经阳离子交换固相萃取柱、石墨化碳黑固相萃取柱、*N*-丙基乙二氨键合硅胶固相萃取柱净化，用气相色谱-质谱仪测定，外标法定量。

3 试剂和材料

除另有规定外，所用试剂均为分析纯，水为蒸馏水。

3.1 乙腈：分析纯，色谱纯。

3.2 甲醇。

3.3 乙酸乙酯。

3.4 丙酮。

3.5 二氯甲烷。

3.6 正己烷：分析纯，色谱纯。

3.7 氨水：浓度 25%。

3.8 磷酸氢二钾。

3.9 磷酸二氢钾。

3.10 无水硫酸钠：在 650℃灼烧 4 h，贮于干燥器中，冷却后备用。

3.11 氯化钠。

3.12 磷酸盐缓冲溶液(0.5 mol/L，pH7.0)：称取 52.7 g 磷酸氢二钾和 30.2 g 磷酸二氢钾，加水 1 L 溶解，用 2 mol/L 氢氧化钠调 pH 值至 7.0。

3.13 乙酸乙酯＋正己烷混合溶液(1＋4)：量取 20 mL 乙酸乙酯和 80 mL 正己烷，混匀。

3.14 氨水-甲醇混合溶液(1 mol/L)：准确量取 1.86 mL 氨水用甲醇定容至 25 mL。

3.15 扑草净标准物质：纯度大于等于 99%，英文通用名(prometryne)，CAS No. 7287-19-6。

3.16 扑草净标准溶液：准确称取适量的扑草净标准物质，用乙腈配成浓度为 100.0 mg/L 的标准储备溶液，4℃下避光保存。根据需要用乙腈将储备液稀释成适当浓度的标准工作溶液，现用现配。

3.17 阳离子交换固相萃取柱(SCX)：500 mg，3 mL，或相当者。

3.18 石墨化碳黑固相萃取柱(Envi-Carb)：500 mg，6 mL，或相当者。

3.19 *N*-丙基乙二氨键合硅胶固相萃取柱(PSA)：200 mg，3 mL，或相当者。

4 仪器和设备

4.1 气相色谱-质谱仪：配有电子轰击电离源(EI)。

4.2 样品粉碎机。

4.3 振荡器。
4.4 旋转蒸发仪。
4.5 pH 计。
4.6 氮吹仪。
4.7 固相萃取装置。
4.8 涡旋振荡器。

5 试样制备和保存

5.1 试样制备

5.1.1 大米

取有代表性样品 500 g，粉碎并使其全部通过孔径为 2.0 mm 的样品筛。混合均匀，装入洁净的容器内，密封并标识。

5.1.2 胡萝卜、西兰花、西红柿、洋葱、蘑菇、苹果、柑橘

取有代表性样品 500 g，取可食部分后将其切成小块(不可水洗)，用组织捣碎机将样品匀浆，混合均匀，装入洁净的容器内，密封并标识。

5.1.3 花生、板栗

取有代表性样品 500 g，取可食部分，粉碎并使其全部通过孔径为 2.0 mm 的样品筛。混合均匀，装入洁净的容器内，密封并标识。

5.1.4 鸡肉、牛肉、鸡肾

取有代表性样品 500 g，切碎并用组织捣碎机将样品加工成浆状，混合均匀，装入洁净的容器内，密闭并标识。

5.1.5 紫菜

取有代表性样品 50 g，粉碎，混合均匀，装入洁净的容器内，密封并标识。

5.2 试样保存

粮谷、坚果类试样在－4℃避光保存；水果、蔬菜、肉、内脏等试样在－18℃避光保存。

取样、制样和样品保存过程中，应防止样品受到污染或发生残留物含量的变化。

6 测定步骤

6.1 提取

6.1.1 胡萝卜、西兰花、西红柿、洋葱、蘑菇、苹果、柑橘 、板栗、花生：称取 5 g 试样(精确至 0.01g)于 250 mL 锥形瓶中，加入 30 mL 乙腈，于振荡器上振荡 20 min，静置 10 min，过滤于 250 mL 分液漏斗。残渣再加入 30 mL 乙腈提取一次，合并两次滤液。滤液中加入 25 g 氯化钠和 60 mL 磷酸盐缓冲液(3.12)，振摇 15 min，静置分层，弃去水层，乙腈层过无水硫酸钠后，于 40℃浓缩至干，残渣用 2.0 mL 乙酸乙酯＋正己烷混合溶液(1＋4)溶解。

6.1.2 大米：称取 5 g 试样(精确至 0.01 g)于 250 mL 锥形瓶中，加 10 mL 水放置 15 min，加入 30 mL 乙腈，于振荡器上振荡 20 min，静置 10 min，过滤并收集滤液于 250 mL 分液漏斗中。残渣再加入 20 mL乙腈提取一次，过滤，合并两次滤液。滤液中加入 25 g 氯化钠和 60 mL 磷酸盐缓冲液(3.12)，振摇 15 min，静置分层，弃去水层，乙腈层过无水硫酸钠后，于 40℃浓缩至干，残渣用 2.0 mL 乙酸乙酯＋正己烷混合溶液(1＋4)溶解。

6.1.3 鸡肉、牛肉、鸡肾：称取 5 g 试样(精确至 0.01 g)于 50 mL 具塞离心管中，加入 30 mL 乙腈，于振荡器上振荡 20 min，静置 10 min，过滤于 250 mL 分液漏斗中。残渣再加入 30 mL 乙腈提取一次，合并两次滤液。滤液中加入 25 g 氯化钠和 60 mL 磷酸盐缓冲液(3.12)，振摇 15 min，静置分层，弃去水层。乙腈层加入 20 mL 用乙腈饱和的正己烷，振荡 10 min，弃去正己烷，乙腈层过无水硫酸钠后，于

40℃浓缩至干，残渣用 2.0 mL 乙酸乙酯＋正己烷混合溶液(1＋4)溶解。

6.1.4 紫菜：称取 3 g 试样(精确至 0.01 g)于 250mL 锥形瓶中，加 10 mL 水，放置 15 min，加入 30 mL 乙腈，于振荡器上振荡 20 min，静置 10 min，提取液过滤于 250 mL 分液漏斗中。残渣再加入 20 mL 乙腈提取一次，合并两次滤液。滤液中加入 25 g 氯化钠和 60 mL 磷酸盐缓冲液(3.12)，振摇 15 min，静置分层，弃去水层，乙腈层过无水硫酸钠后，于 40℃浓缩至干，残渣用 2.0 mL 乙酸乙酯＋正己烷混合溶液(1＋4)溶解。

6.2 净化

6.2.1 对于 6.1.1、6.1.2、6.1.3 提取的样品溶液，用 Envi-Carb 和 PSA 固相萃取柱净化。Envi-Carb 小柱和 PSA 小柱分别用 10 mL 和 4 mL 乙酸乙酯＋正己烷混合溶液(1＋4)活化，将活化好的 PSA 小柱连接在 Envi-Carb 小柱底部。转移提取液至串联柱上，控制流速小于等于 2 mL/min，用 3×2 mL 乙酸乙酯＋正己烷混合溶液(1＋4)洗涤鸡心瓶，并将洗涤液转移到串联柱中，收集全部流出液，于 40℃在氮吹仪上吹干，用 1.0 mL 乙腈定容，供气相色谱-质谱测定。

6.2.2 对于 6.1.4 提取的样品，用 Envi-Carb 和 SCX 固相萃取柱净化。Envi-Carb 小柱用 10 mL 乙酸乙酯＋正己烷混合溶液(1＋4)活化。转移提取液至 Envi-Carb 固相萃取柱，控制流速小于等于 2 mL/min，用 3×2 mL乙酸乙酯＋正己烷混合溶液(1＋4)洗涤鸡心瓶，并转移至柱上，抽干，收集全部流出液，于 40℃在氮吹仪上吹干，加入 1 mL 正己烷溶解。SCX 固相萃取柱分别用 2 mL 丙酮和 2 mL 二氯甲烷活化，转移上述正己烷溶解液至 SCX 小柱中，用 2×1 mL 正己烷洗涤鸡心瓶，并转移到柱中，用 2 mL 二氯甲烷，2×2 mL 丙酮依次淋洗，弃去淋洗液，用 2 mL 氨水-甲醇溶液(3.14)洗脱。洗脱液收集于分液漏斗中，收集之前在分液漏斗中预先加入 10 mL 磷酸盐缓冲液(3.12)。洗脱液摇匀后分别用 10 mL、10 mL、5 mL 二氯甲烷萃取，合并全部二氯甲烷提取液，并用无水硫酸钠脱水，浓缩至干，用 1.0 mL 乙腈定容，供气相色谱-质谱测定。

6.3 测定

6.3.1 气相色谱-质谱条件

a) 色谱柱：DB-5MS 石英毛细管柱，30 m×0.25 mm(内径)×0.25 μm，或相当者；
b) 色谱柱温度程序：70℃保持 1 min，然后以 25℃/min 升温至 180℃，再以 5℃/min 升温至 220℃，再以 15℃/min 升温至 280℃，保持 3 min；
c) 载气：氦气，纯度大于等于 99.999%；
d) 进样口温度：260℃；
e) 流速：1.0 mL/min；
f) 进样方式：不分流进样；
g) 进样量：1.0 μL；
h) 电子轰击电离源：70 eV；
i) 离子源温度：230℃；
j) 四极杆温度：150℃；
k) GC-MS 接口温度：280℃；
l) 监测离子(m/z)：241、184、226、199。

6.3.2 气相色谱质谱测定

根据样液中扑草净含量情况，选定峰面积相近的标准工作液。标准工作溶液和样液中扑草净响应值均应在仪器检测线性范围内。标准工作液和样液等体积参插进行测定。在上述色谱条件下，扑草净的保留时间约为 11.5 min。标准物质的总离子流色谱图参见附录 A。

扑草净的定性离子(m/z)为 241、184、226、199，定量离子(m/z)为 184，在扣除背景后的样品质谱图中，所选择的离子必须出现，离子丰度比变化范围见表 1。

表 1　扑草净定性离子与相对丰度比

化合物	定性离子(m/z)	标准丰度比/%	允许相对偏离范围/%
扑草净	241.00	100	—
	184.00	70～80	10
	226.00	50～60	10
	199.00	20～30	15

6.4　空白实验

除不加试样外，按上述测定步骤进行。

7　结果计算

用色谱数据处理机或按式(1)计算试样中扑草净的残留量：

$$X = \frac{A \cdot c_S \cdot V}{A_S \cdot m} \quad \cdots\cdots(1)$$

式中：

X——试样中扑草净的残留含量，单位为毫克每千克(mg/kg)；

A——样液中扑草净的峰面积；

A_S——标准工作液中扑草净的峰面积；

c_S——标准工作液中扑草净的浓度，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)；

m——最终样液所代表的试样量，单位为克(g)。

注：计算结果需扣除空白值。

8　测定低限、回收率

8.1　测定低限

本方法中扑草净的测定低限为 0.01 mg/kg。

8.2　添加浓度和回收率

回收率数据见表 2。

表 2　不同基质中扑草净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/%
大米	0.01	102.06～109.76
	0.02	98.18～109.47
	0.05	101.74～108.78
花生	0.01	82.23～95.34
	0.02	90.16～102.71
	0.05	86.73～95.86
胡萝卜	0.01	74.19～87.12
	0.02	70.21～90.12
	0.05	82.73～88.30

表 2(续)

样品名称	添加浓度/(mg/kg)	回收率范围/%
西兰花	0.01	90.74～115.38
	0.02	96.44～109.8
	0.05	84.08～108.86
西红柿	0.01	75.54～87.44
	0.02	86.67～95.00
	0.05	85.04～91.99
蘑菇	0.01	74.54～86.83
	0.02	82.67～96.88
	0.05	85.04～91.38
洋葱	0.01	77.55～96.05
	0.02	86.44～104.62
	0.05	88.53～102.24
苹果	0.01	81.40～102.17
	0.02	82.52～100.83
	0.05	86.85～102.20
柑橘	0.01	82.36～104.18
	0.02	85.83～108.60
	0.05	82.16～104.79
板栗	0.01	74.19～88.20
	0.02	85.04～96.26
	0.05	80.16～100.18
鸡肉	0.01	81.62～97.62
	0.02	83.54～97.61
	0.05	88.58～102.36
牛肉	0.01	82.16～102.48
	0.02	86.80～108.33
	0.05	92.58～107.13
鸡肾	0.01	88.40～102.84
	0.02	84.68～102.55
	0.05	79.73～87.80
紫菜	0.01	75.54～87.44
	0.02	86.67～93.94
	0.05	85.73～91.99

附 录 A
（资料性附录）
扑草净标准物质全扫描总离子流色谱图和质谱图

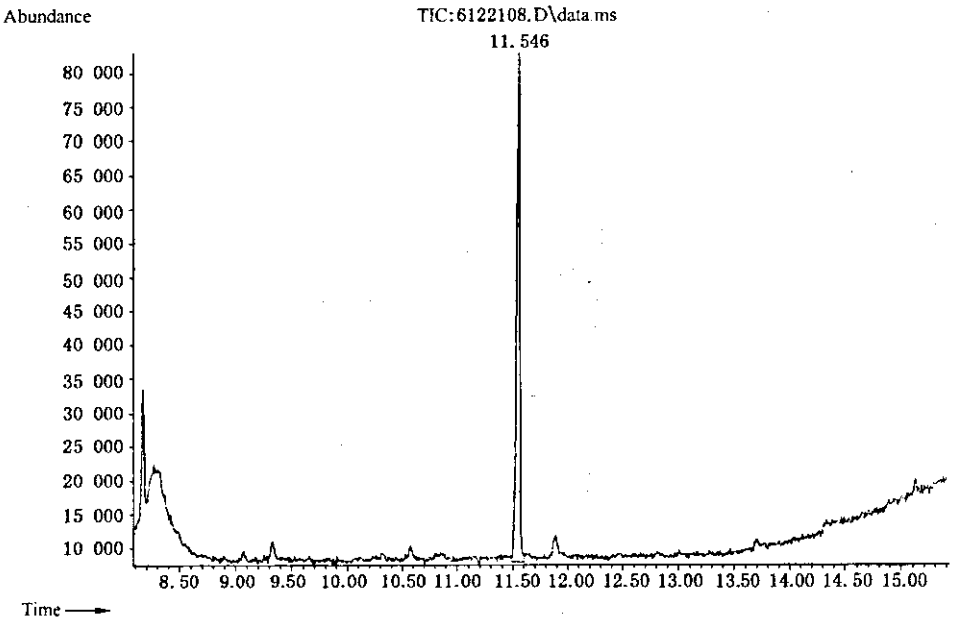

图 A.1 扑草净标准物质全扫描总离子流色谱图

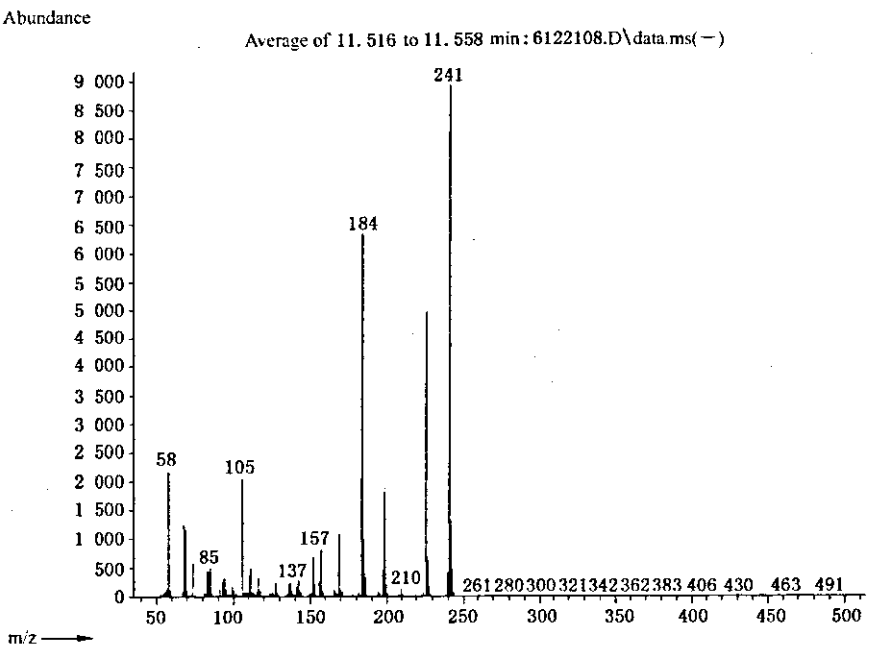

图 A.2 扑草净标准物质全扫描质谱图

Foreword

Annex A of this standard is an informative annex.

This standard is proposed by and is under the charge of National Regulatory Commission for Certification and Accreditation.

This standard was drafted by Chinese Academy of Inspection and Quarantine, Jiangsu Entry-Exit Inspection and Quarantine Bureau of People's Republic of China and Fujian Entry-Exit Inspection and Quarantine Bureau of People's Republic of China.

This standard was mainly drafted by Chen Dongdong, Li Shujuan, Li Xiaojuan, Cai Huixia, An Juan, Feng Fan, Li Jianzhong, Tang Yingzhang.

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

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

Determination of prometryne residues in foods for import and export—GC-MS Method

1 Scope

This standard specifies the method of sample preparation and determination of prometryne residue in food by GC-MS.

This standard is applicable for the determination and confirmation of prometryne residue in rice, peanut, carrot, broccoli, tomato, onion, mushroom, apple, citrus, castaneamollissima, chicken, beef, chicken kidney and laver.

2 Principle

Prometryne residue in sample is extracted with acetonitrile, wipes off water by salt out, the extraction is passed through SPE column to purify and determined by GC-MS, quantified by external standard method.

3 Reagent and materials

Unless specifically mentioned, all reagents used should be analytical grade. "water" is deionized water.

3.1 Acetonitrile, HPLC grade.

3.2 Methanol.

3.3 Ethyl acetate.

3.4 Acetone.

3.5 Dichloromethane.

3.6 *n*-Hexane, HPLC grade.

3.7 Ammonia water: 25%.

3.8 Dibasic potassium phosphate(K_2HPO_4).

3.9 Monopotassium phosphate(KH_2PO_4).

3.10 Anhydrous sodium sulfate: Ignited at 650℃ for 4 h, and stored in desiccator.

3.11 Sodium chloride(NaCl).

3.12 Phosphate buffer (0.5 mol/L, pH 7.0): Weigh 52.7 g of K_2HPO_4 and 30.2 g of KH_2PO_4 in 1 L cylinder, add water to dissolve and make up to the mark, adjust pH=7.0 by use 2 mol/L NaOH.

3.13 Ethyl acetate-*n*-Hexane(1+4): 20 mL of Ethyl acetate diluted to 100 mL with *n*-Hexane.

3.14 Ammoniated methanol(1 mol/L): 1.86 mL ammonia water diluted to 25 mL with methanol.

3.15 Prometryne standard: Purity≥99%, CAS No. 7287-19-6.

3.16 Prometryne standard solution: certain amount of prometryne weighed accurately and dissolved in acetonitrile to prepare 100.0 μg/mL standard stock solution, and diluted to suitable concentration with acetonitrile as standard working solution. The solution can be preserved at 4℃.

3.17 SCX, SPE: 500 mg, 3 mL, or equivalent.

3.18 Envi-Carb, SPE: 500 mg, 6 mL, or equivalent.

3.19 PSA, SPE: 200 mg, 3 mL, or equivalent.

4 Apparatus and equipment

4.1 Gas Chromatograph-Mass Spectrometry, equipped with EI.

4.2 Sample crusher.

4.3 Shaker.

4.4 Rotary evaporator.

4.5 pH meter.

4.6 Nitrogen evaporator.

4.7 SPE vacuum container.

4.8 Vortex oscillator.

5 Sample preparation and storage

5.1 Sample preparation

5.1.1 Rice

Represenative sample 500 g , grind thoroughly, pass through 2.0 mm sieve, mix thoroughly, then place in clean containers as the test samples by sealed and labeled.

5.1.2 carrot, broccoli tomato, onion, mushroom, apple, citrus

Represenative sample 500 g, cut into small pieces for edible part, homogenate thoroughly, then place in clean containers as the test samples by sealed and labeled.

5.1.3 peanut castanea mollissima

Represenative sample 500 g, grind thoroughly for edible part, pass through 2.0 mm sieve, mix thoroughly, then place in clean containers as the test samplesby sealed and labeled.

5.1.4 chicken, beef, chicken kidney

Represenative sample 500g, homogenate thoroughly, then place in clean containers as the test samples by sealed and labeled.

5.1.5 Laver

Represenative sample 50 g, grind and mix thoroughly, then place in clean containers as the test samples by sealed and labeled.

5.2 Sample storage

The test samples of cereals and nuts should be stored in −4℃, avoiding from the light; the other samples should be stored below −18℃, avoiding from the light.

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.

6 Procedure

6.1 Extraction

6.1.1 carrot, broccoli, tomato, onion, mushroom, apple, citrus, castanea mollissima, peanut, 5 g (accurating to 0.01 g) of test sample is weighed and put into a 250 mL conical flask. At the same time, 30 mL of acetonitrile is added into the conical flask. and shaked for 20 min, let standed for 10 min, and filtered into a 250 mL separatory funnel . The extraction is repeated with 30 mL of acetonitrile. Combine the supernatants to the funnel, add 25 g NaCl and 60 mL phosphate buffer(3.12), shake for 15 min, lay separation and acetonitrile through anhydrous sodium sulfate, and evaporate to nearly dry in a water bath under 40℃. The residue is redissolved in 2.0 mL of ethyl acetate-*n*-hexane(1+4).

6.1.2 Rice: 5 g (accurating to 0.01 g) of test sample is weighed and put into a 250 mL conical flask. 10 mL water is added into the conical flask let standed for 15 min. 30 mL of acetonitrile is added into the conical flask and shaked for 20 min, let standed for 10 min and filtered into a 250 mL separatory funnel . The extraction is repeated with 20 mL of acetonitrile. Combine the supernatants to the funnel, add 25 g NaCl and 60 mL phosphate buffer (3.12), shake for 15 min, lay separation and acetonitrile through anhydrous sodium sulfate, and evaporate to nearly dry in a water bath under 40℃. Then the residue is redissolved in 2.0 mL of ethyl acetate-*n*-hexane(1+4).

6.1.3 chicken, beef, chicken kidney: 5 g (accurating to 0.01 g) of test sample is weighed and put into a 50 mL centrifuge tube, 30 mL of acetonitrile is added into the centrifuge tube and shaked for 20 min, let standed for 10 min and filtered into a 250 mL separatory funnel . The extraction is repeated with 30 mL of acetonitrile. Combine the supernatants to the funel, add 25 g NaCl and 60 mL phosphate buffer(3.12), shake for 15 min, lay separation , wipe off water, then purify from fats by extraction with 20 mL acetonitrile saturated hexane, shake for 10 min, wipe off hexane. Acetonitrile through anhydrous sodium sulfate is evaporate to nearly dry in a water bath under 40℃ . Then the residue is redissolved in 2.0 mL of ethyl acetate-*n*-hexane(1+4).

6.1.4 Laver: 3 g (accurating to 0.01 g) of test sample is weighed and put into a 250 mL conical flask. 10 mL water is added into the conical flasklet standed for 15 min. 30 mL of acetonitrile is added into the conical flask and shaked for 20 min, let standed 10 min and filtered into a 250 mL separatory funnel . The extraction is repeated with 20 mL of acetonitrile. Combine the supernatants to the funnel, add 25 g NaCl and 60 mL phosphate buffer (3.12), shake 15 min, lay separation. And acetonitrile through anhydrous sodium sulfate is evaporated to nearly dry in a water bath under 40℃ . Then the residue is redissolved in 2.0 mL of ethyl acetate-*n*-hexane(1+4).

6.2 Clean up

6.2.1 Extraction of sample from 6.1.1, 6.1.2 and 6.1.3 is cleaned up by Envi-Carb and PSA. Fiest, which are washed with 10 mL and 4 mL ethyl acetate-*n*-hexane(1+4), tandem 2 column(Envi-Carb

up,PSA down). Pass the sample extract solution through the tandem column at a rate of ≤2 mL/min. Rinse the column with 3 × 2 mL ethyl acetate-*n*-hexane(1 + 4). Collect elution in a tube. Nitrogen evaporator to dry in a water bath under 40℃ ,add 1.0 mL acetonitrile and ready for GC-MS determination.

6.2.2 Extraction of sample from 6.1.4 is cleaned up by Envi-Carb and SCX. Envi-Carb is washed with 10 mL ethyl acetate-*n*-hexane (1+4). Pass the sample extract solution through the column at a rate of ≤2 mL/min. Rinse the column with 3 × 2 mL ethyl acetate-*n*-hexane(1 + 4). Collect elution in a tube. Nitrogen evaporator to dryness, add 1 mL *n*-hexane solution. SCX is activated with 2 mL acetone and 2 mL dichloromethane, pass the solution through the column. Rinse the column with 2 × 1 mL *n*-hexane, 2 mL dichloromethane, 2 × 2 mL acetone in turn, elution by 2 mL ammoniated methanol(3.14). Collect elution in a separatory funnel, add 10 mL phosphate buffer(3.12) and shake up. Reextract thrice with 10 mL, 10 mL, 5 mL dichloromethane. Dichloromethane through anhydrous sodium sulfate. Nitrogen evaporator to dryness, add 1.0 mL acetonitrile and ready for GC-MS determination.

6.3 Determination

6.3.1 GC-MS operating conditions:

a) Column: DB-5MS, 30 m × 0.25 mm(i.d.) × 0.25 μm, or to some tune;

b) Column temperature: 70℃ (1 min) $\xrightarrow{25℃/min}$ 180℃ $\xrightarrow{5℃/min}$ 220℃ $\xrightarrow{15℃/min}$ 280℃ (3 min);

c) Carrier gas: He, Purity≥99.999%;

d) Injection temperature: 260℃;

e) Flow rate: 1.0 mL/min;

f) Injection mode: Splitless;

g) Injection volume: 1.0 μL;

h) Ion source: EI, 70 eV;

i) Source temperature: 230℃;

j) Quadrupole temperature: 150℃;

k) Interface temperature: 280℃;

l) Selected ions(m/z):241,184,226,199.

6.3.2 GC-MS determination

According to the approximate concentration of prometryne in sample solution, select the standard working solution with similar peak area to that of sample solution. The responses of the analyte in the standard working solution and the sample solution should be within the linear range of the instrument detection. The retention time of prometryne is about 11.5 min under the above conditions. The Choromatogram of total ion current of the standard, see annex A.

Qualitative ion of prometryne is 241,184,226,199. The quantitation ion is 184. For the same analysis batch and the same compound, the variation range of the ion ratio at the similar concentration can not be out of range of table 1, then the corresponding analyte must be present in the sample.

Table 1—Qualitative ion and relative ion ratio

Compound	Qualitative ion (m/z)	Stand ion ratio/%	Permitted tolerances/%
Prometryne	241.00	100	—
	184.00	70~80	10
	226.00	50~60	10
	199.00	20~30	15

6.4 Blank test

The operation of blank test is the same as above, except without sample.

7 Calculation and expresssion of result

The content of prometryne is calculated by the following formula (1):

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

where

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

A—peak area of prometryne in sample solution;

A_s—peak area of prometryne in standard solution;

c_s—concentration of prometryne in standard solution, μg/mL;

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

m—mass of test sample, g.

Note: The blank value shall be subtracted from the above result of calculation.

8 Limit of determination and recovery

8.1 Limit of determination

The limit of determination of this method is 0.01 mg/kg.

8.2 Add concentration and recovery

Data are shown in Table 2.

Table 2—Add concentration and recovery

Sample	Add concentration/(mg/kg)	Recovery/%
rice	0.01	102.06～109.76
	0.02	98.18～109.47
	0.05	101.74～108.78
peanut	0.01	82.23～95.34
	0.02	90.16～102.71
	0.05	86.73～95.86
carrot	0.01	74.19～87.12
	0.02	70.21～90.12
	0.05	82.73～88.30
broccoli	0.01	90.74～115.38
	0.02	96.44～109.8
	0.05	84.08～108.86
tomato	0.01	75.54～87.44
	0.02	86.67～95.00
	0.05	85.04～91.99
mushroom	0.01	74.54～86.83
	0.02	82.67～96.88
	0.05	85.04～91.38
onion	0.01	77.55～96.05
	0.02	86.44～104.62
	0.05	88.53～102.24

Table 2(Continued)

Sample	Add concentration/(mg/kg)	Recovery/%
apple	0.01	81.40～102.17
	0.02	82.52～100.83
	0.05	86.85～102.20
citrus	0.01	82.36～104.18
	0.02	85.83～108.60
	0.05	82.16～104.79
castanea mollissima	0.01	74.19～88.20
	0.02	85.04～96.26
	0.05	80.16～100.18
chicken	0.01	81.62～97.62
	0.02	83.54～97.61
	0.05	88.58～102.36
beef	0.01	82.16～102.48
	0.02	86.80～108.33
	0.05	92.58～107.13
chicken kidney	0.01	88.40～102.84
	0.02	84.68～102.55
	0.05	79.73～87.80
laver	0.01	75.54～87.44
	0.02	86.67～93.94
	0.05	85.73～91.99

Annex A
(informative annex)
Choromatogram of total ion current (TIC) and mass spectrum

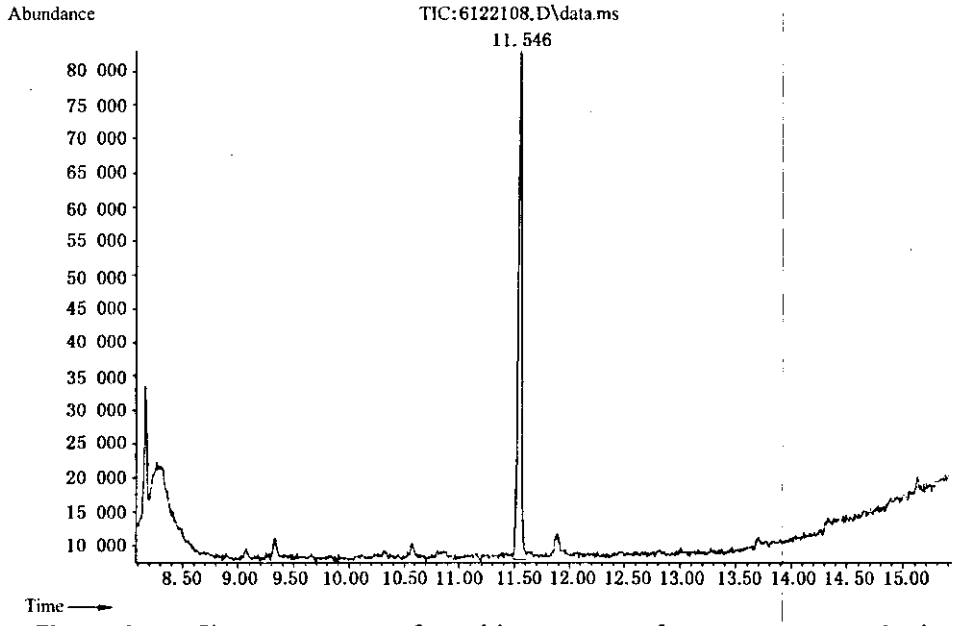

Figure A. 1—Choromatogram of total ion current of prometryne standard

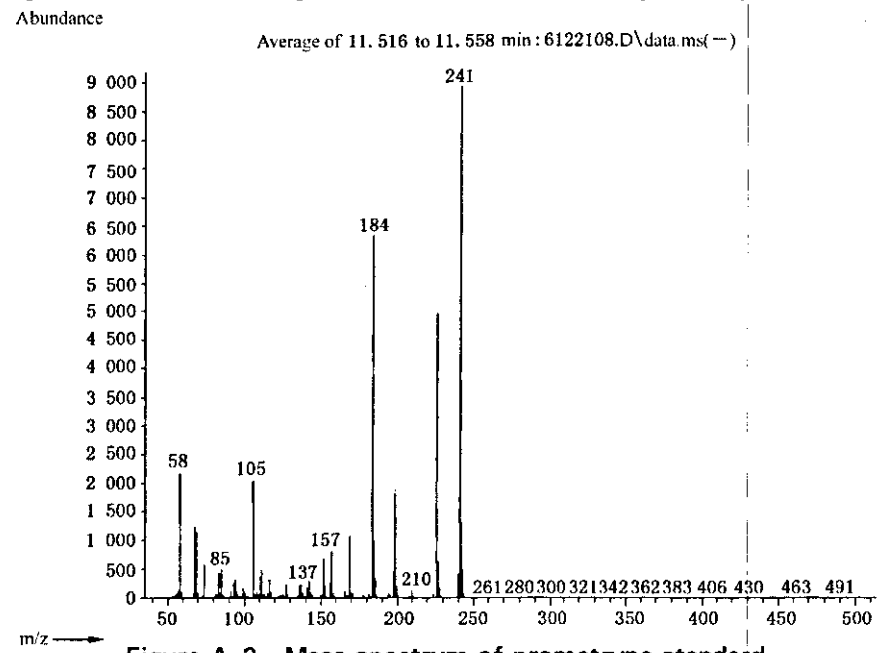

Figure A. 2—Mass spectrum of prometryne standard

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