



中华人民共和国出入境检验检疫行业标准

SN/T 0712—2010
代替 SN 0712—1997

进出口粮谷和大豆中 11 种除草剂 残留量的测定 气相色谱-质谱法

Determination of 11 herbicide residues in cereals and soybean for import
and export—GC-MS method

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前 言

本标准代替 SN 0712—1997《出口粮谷中戊草丹、二甲戊灵、丙草胺、氟酰胺、灭锈胺、苯噻酰草胺残留量检验方法》。

本标准与 SN 0712—1997 相比,主要变化如下:

- 修改了标准的名称;
- 对原标准的结构进行了修改;
- 标准的适用范围由大米增加为小麦、玉米和大豆;
- 增加了乙草胺、甲草胺、异丙甲草胺、丁草胺和吡氟酰草胺残留量的测定;
- 按照主要贸易国家对酰胺类除草剂的限量要求重新制定了检测低限;
- 采用气相色谱质谱法替代了原标准中的气相色谱法;
- 增加了确证方法。

本标准的附录 A 为规范性附录,附录 B 和附录 C 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:中华人民共和国黑龙江出入境检验检疫局。

本标准主要起草人:康庆贺、吴岩、杨长志。

本标准于 1997 年 12 月 1 日首次发布,本次为第一次修订。

进出口粮谷和大豆中 11 种除草剂 残留量的测定 气相色谱-质谱法

1 范围

本标准规定了进出口粮谷和大豆中 11 种除草剂残留量的气相色谱-质谱检测方法。

本标准适用于进出口大米、玉米、小麦和大豆中乙草胺、戊草丹、甲草胺、异丙甲草胺、二甲戊灵、丁草胺、氟酰胺、丙草胺、灭锈胺、吡氟酰草胺和苯噻酰草胺残留量的测定和确证。

2 方法提要

试样中残留的除草剂用正己烷和丙酮提取,提取液浓缩后,经中性氧化铝固相萃取柱或氟罗里硅土固相萃取柱净化,用气相色谱-质谱仪检测和确证,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,水为蒸馏水或相当纯度的水。

- 3.1 丙酮:色谱纯。
- 3.2 正己烷:色谱纯。
- 3.3 乙腈:色谱纯。
- 3.4 氯化钠。
- 3.5 无水硫酸钠:经 650 °C 灼烧 4 h,置于干燥器内备用。
- 3.6 正己烷-丙酮溶液(2+1,体积比)。
- 3.7 正己烷-丙酮溶液(9+1,体积比)。
- 3.8 正己烷-丙酮溶液(8+2,体积比)。
- 3.9 11 种除草剂标准品:纯度均大于等于 95.0%。
- 3.10 标准储备溶液:分别准确称取农药标准品(见附录 A),用正己烷分别配制成浓度为 100 µg/mL~1 000 µg/mL 的标准储备液。标准储备液于 -18 °C 条件下储存;根据需要再用正己烷稀释成适当浓度的标准工作液,于 0 °C~4 °C 条件下储存。
- 3.11 中性氧化铝固相萃取柱:1 000 mg,6 mL。使用前预先依次用 3 mL 丙酮、5 mL 正己烷活化,备用。
- 3.12 氟罗里硅土固相萃取柱:500 mg,6 mL。使用前预先依次用 3 mL 丙酮、5 mL 正己烷活化,备用。

4 仪器和设备

- 4.1 气相色谱-质谱联用仪(GC-MSD):配有电子轰击电离离子源(EI)。
- 4.2 电子天平:感量为 0.000 1 g 和 0.01 g。
- 4.3 固相萃取装置。
- 4.4 粉碎机。
- 4.5 离心机,5 000 r/min。
- 4.6 旋涡混合器。
- 4.7 旋转蒸发器。
- 4.8 均质器,20 000 r/min。

4.9 浓缩瓶:100 mL。

4.10 具塞离心管:15 mL、50 mL。

4.11 无水硫酸钠柱:8.0 cm×1.5 cm(内径)玻璃柱,内装 5 cm 高无水硫酸钠。

5 试样制备与保存

5.1 试样制备

将样品按四分法缩分至 500 g,用粉碎机(4.4)全部磨碎,并通过 2.0 mm 的圆孔筛,混匀,均分成两份作为试样,分装至洁净的盛样瓶内,密封并标明标记。

5.2 试样保存

试样于 0℃~4℃ 以下保存。在制样的操作过程中,应防止样品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 大米、玉米和小麦

称取试样约 5 g(精确到 0.01 g)于 50 mL 离心管中,加入 3 g 氯化钠、10 mL 水和 20 mL 正己烷-丙酮(3.6),用均质器以 10 000 r/min 均质 2 min,在 4 000 r/min 下离心 5 min。将上清液通过无水硫酸钠柱脱水于 100 mL 浓缩瓶中。再用 20 mL 正己烷-丙酮(3.6)重复提取一次,合并提取液于同一 100 mL 浓缩瓶中,在 35℃ 水浴中用旋转蒸发器浓缩至近干,再用氮气流吹干。加入 2 mL 正己烷溶解残渣待净化。

6.1.2 大豆

称取试样约 5 g(精确到 0.01 g)于 50 mL 离心管中,加入 5 g 无水硫酸钠和 20 mL 乙腈,用均质器以 10 000 r/min 均质 2 min,在 4 000 r/min 下离心 5 min。将上清液转移至 100 mL 浓缩瓶中。再用 20 mL 乙腈重复提取一次,合并提取液于同一 100 mL 浓缩瓶中,在 35℃ 水浴中用旋转蒸发器浓缩至近干,再用氮气流吹干。加入 2 mL 正己烷溶解残渣待净化。

6.2 净化

6.2.1 玉米

将上述样液全部转移到中性氧化铝固相萃取柱(3.12)中,控制流速在 1 mL/min~2 mL/min,弃去流出液。加入 10 mL 正己烷-丙酮(3.8)溶液洗脱,控制流速在 1 mL/min~2 mL/min,收集洗脱液。洗脱液经 60℃ 氮吹仪吹干后,用 5 mL 正己烷溶解,供气相色谱-质谱仪测定。

6.2.2 大米、大豆和小麦

将上述样液全部转移到氟罗里硅土固相萃取柱(3.13)中,控制流速在 1 mL/min~2 mL/min,弃去流出液。加入 13 mL 正己烷-丙酮(3.7)溶液洗脱,控制流速在 1 mL/min~2 mL/min,收集洗脱液。洗脱液经 60℃ 氮吹仪吹干后,用 5 mL 正己烷溶解,供气相色谱和气相色谱-质谱仪测定。

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),膜厚 0.25 μm 或性能相当者;
- b) 色谱柱温度:初始温度 100℃ 保持 1 min,以 15℃/min 升至 260℃,保持 15 min;
- c) 进样口温度:250℃;
- d) 色谱-质谱接口温度:280℃;
- e) 载气:氦气,纯度大于 99.999%,恒流模式,流速 1.0 mL/min;
- f) 进样量:1 μL;
- g) 进样方式:不分流进样,0.75 min 后开阀;
- h) 电离方式:EI;

- i) 电离能量:70 eV;
- j) 离子源温度:230 ℃;
- k) 四极杆温度:150 ℃;
- l) 测定方式:选择离子监测方式;
- m) 选择监测离子(m/z):见表 1 和参见附录 B;
- n) 溶剂延迟:5.0 min。

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)	驻留时间/ms
1	5.00	223,269,160,188,237,222,151,265,162,238,240,146	40
2	9.20	252,162,253,281,176,311,237,160,173,145,323,262,238	30
3	10.60	119,91,269,120,266,394,267,245,192,136,148,298	40

6.3.2 气相色谱-质谱检测和确证

根据样液中被测物的含量情况,选定浓度与样液相近的标准工作溶液。标准工作溶液和样液中每种除草剂的响应值均应在仪器检测线性范围内。标准工作溶液和样液等体积参插进样测定。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 中每种除草剂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,样品中待测物质保留时间与标准工作溶液中对应的保留时间的偏差在 $\pm 2.5\%$,且样品中被测物质的相对离子丰度与浓度相当标准工作溶液的相对离子丰度进行比较,相对丰度允许相对偏差不得超过表 2 规定的范围,则可确定样品中存在对应的被测物。11 种除草剂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 中的图 C.1。

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/%	± 10	± 15	± 20	± 50

6.4 空白试验

除不加试样外,均按上述步骤进行。

6.5 结果计算和表述

试样中每种除草剂残留量按式(1)计算:

$$X_i = \frac{A_i \times c_{is} \times V \times 1\,000}{A_{is} \times m \times 1\,000} \dots\dots\dots (1)$$

式中:

X_i ——试样中每种除草剂残留量,单位为毫克每千克(mg/kg);

A_i ——样液中每种除草剂的峰面积;

c_{is} ——标准工作液中每种除草剂的浓度,单位为微克每毫升($\mu\text{g/mL}$);

A_{is} ——标准工作液中每种除草剂的峰面积;

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)。

7 测定低限、回收率

7.1 测定低限

采用本方法对大米、小麦、玉米和大豆中 11 种除草剂残留进行测定,各种农药的测定低限均为 0.01 mg/kg。

7.2 添加浓度范围及回收率

本方法添加浓度及回收率范围见表 3。

表 3 11 种除草剂在大米、小麦、玉米和大豆中的添加回收率

序 号	中文通用名称	添加回收率(添加水平为 0.01 mg/kg~2.0 mg/kg)/%			
		大米	小麦	玉米	大豆
1	乙草胺	87.1~98.6	79.4~100.7	82.5~108.7	87.1~105.2
2	甲草胺	85.3~106.5	83.1~111.0	80.0~108.3	75.3~101.2
3	戊草丹	80.5~102.1	76.5~104.0	84.3~101.6	81.6~108.1
4	异丙甲草胺	85.3~111.8	85.6~110.6	84.7~100.9	88.0~111.1
5	二甲戊灵	86.6~104.1	77.3~117.2	84.2~107.1	85.6~110.6
6	丁草胺	80.5~94.7	85.3~106.7	86.8~104.2	80.0~101.2
7	氟酰胺	84.9~102.5	88.2~107.6	82.4~101.2	82.7~105.3
8	丙草胺	85.9~110.6	82.1~106.3	87.3~107.1	79.5~105.3
9	灭锈胺	80.5~99.7	89.4~101.7	80.3~101.1	75.3~103.2
10	吡氟酰草胺	76.7~104.9	73.8~102.5	72.4~107.1	78.1~102.7
11	苯噻酰草胺	85.3~107.2	79.3~112.5	75.8~100.3	85.3~107.2

附 录 A
(规范性附录)
基 本 信 息

表 A.1 11 种除草剂标准物质基本信息

序 号	除草剂名称	英文名称	CAS 编号	化学分子式
1	乙草胺	acetochlor	34256-82-1	$C_{14}H_{20}ClNO_2$
2	甲草胺	alachlor	15972-60-8	$C_{14}H_{20}ClNO_2$
3	戊草丹	esprocarb	85785-20-2	$C_{15}H_{23}NOS$
4	异丙甲草胺	metolachlor	51218-45-2	$C_{15}H_{22}ClNO_2$
5	二甲戊灵	pendimethalin	40487-42-1	$C_{13}H_{19}N_3O_4$
6	丁草胺	butachlor	23184-66-9	$C_{17}H_{26}ClNO_2$
7	氟酰胺	flutolanil	66332-96-5	$C_{17}H_{16}F_3NO_2$
8	丙草胺	pretilachlor	51218-49-6	$C_{17}H_{26}ClNO_2$
9	灭锈胺	mepronil	55814-41-0	$C_{17}H_{19}NO_2$
10	吡氟酰草胺	diflufenican	83164-33-4	$C_{19}H_{11}F_5N_2O_2$
11	苯噻酰草胺	mefenacet	73250-68-7	$C_{16}H_{14}N_2O_2S$

附 录 B
(资料性附录)

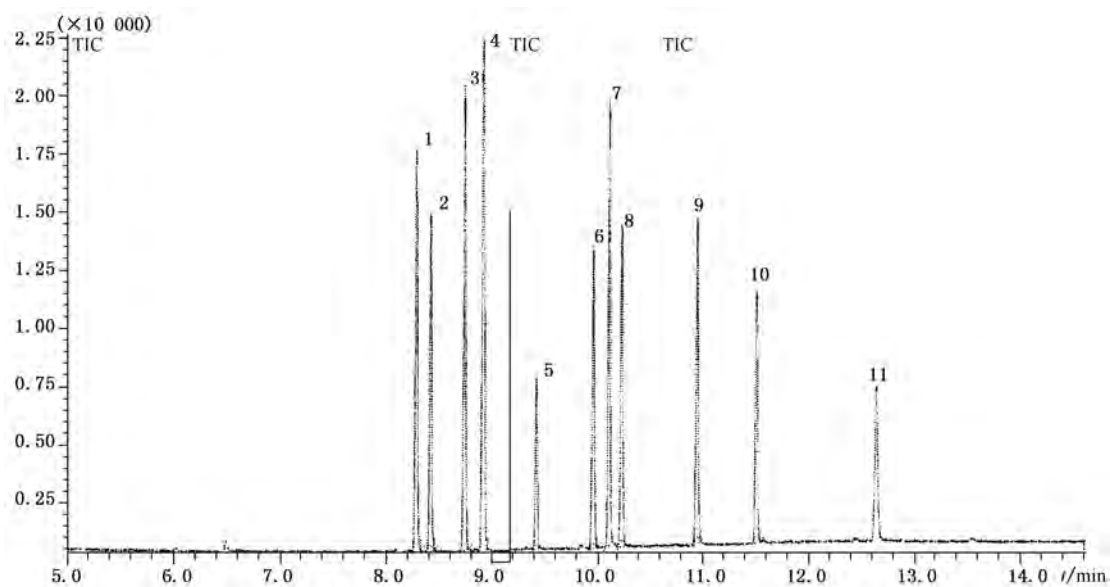
11 种除草剂的保留时间、定量和定性选择离子及测定低限表

表 B.1 11 种除草剂的保留时间、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)		
			定量	定性	丰度比
1	乙草胺	8.268	223	146、162、269	63 : 100 : 75 : 9
2	甲草胺	8.405	160	188、237、269	100 : 84 : 24 : 9
3	戊草丹	8.731	222	162、151、265	100 : 67 : 25 : 12
4	异丙甲草胺	8.910	162	238、240、146	100 : 45 : 14 : 11
5	二甲戊灵	9.408	252	162、253、281	100 : 22 : 10 : 14
6	丁草胺	9.945	176	311、237、160	90 : 100 : 24 : 7
7	氟酰胺	10.098	173	145、281、323	100 : 27 : 26 : 13
8	丙草胺	10.220	262	162、176、238	46 : 100 : 92 : 73
9	灭锈胺	10.937	119	91、269、120	100 : 26 : 18 : 9
10	吡氟酰草胺	11.493	266	394、267、245	100 : 21 : 16 : 13
11	苯噻酰草胺	12.629	192	136、148、298	100 : 25 : 19 : 8

附录 C
(资料性附录)

11 种除草剂标准物气相色谱-质谱选择离子色谱图



- 1——乙草胺；
- 2——戊草丹；
- 3——甲草胺；
- 4——异丙甲草胺；
- 5——二甲戊灵；
- 6——丁草胺；
- 7——氟酰胺；
- 8——丙草胺；
- 9——灭锈胺；
- 10——吡氟酰胺；
- 11——苯噻酰胺。

图 C.1 11 种除草剂标准物气相色谱-质谱选择离子色谱图

Foreword

This standard replaces SN 0712—1997 “Method for the determination of esprocarb, pendimethalin, pretilachlor, flutolanil, mepronil and mefenacet residues in cereals for export”.

This standard and SN 0712—1997 compare the main revision to be as follows:

- Revised the standard name;
- The structure of the original standard was revised;
- Add maize, soybean and wheat beside rice for the standard scope of application;
- Add the determination of acetochlor, alachlor, metolachlor, butachlor and diflufenican residuals;
- In accordance with the major trading country on the type of acetanilide herbicide-limited re-development of the detection limit;
- Using gas chromatography mass spectrometry to replace the original standards in the gas chromatography;
- Add the conformation to the method.

Annex A of this standard is an normative annex, annex B and annex C of this standard is an informative annex.

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

This standard was drafted by Heilongjiang Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

This main drafters of this standard are Kang Qinghe, Wu Yan, Yang Changzhi.

This standard was presented at December 1, 1997 for the first time, the amended for the first time.

Determination of 11 herbicide residues in cereals and soybean for import and export—GC-MS method

1 Scope

This standard specifies the method of sample preparation, determination of 11 herbicide residues in cereals and soybean for import and export by GC-MS.

This standard is applicable to the determination and confirmation of 11 herbicide residues in rice, maize, soybean and wheat for import and export.

2 Principle

The 11 herbicides residues in the test sample are extracted with *n*-hexane-acetone (2+1, V/V). After the extracts is evaporated, the residues is cleaned up by solid phase extraction (SPE) with a neutral aluminum oxide cartridge or florisil cartridge. The residues is determined and confirmed by GC-MS, using external standard method.

3 Reagents and materials

Unless otherwise specified, all regents are analytically pure, “water” is distilled water.

3.1 Acetone; LC grade.

3.2 *n*-Hexane; LC grade.

3.3 Acetonitrile; LC grade.

3.4 Sodium chloride.

3.5 Anhydrous sodium sulfate; Ignite at 650 °C for 4 h and store in a desiccators.

3.6 *n*-Hexane-acetone solution (2+1, Volume ratio).

3.7 *n*-Hexane-acetone solution (9+1, Volume ratio).

3.8 *n*-Hexane-acetone solution (8+2, Volume ratio).

3.9 11 herbicides standards; Purity $\geq 95\%$.

3.10 Standard stock solution: Accurately weigh an adequate amount of 11 herbicides standards, Standard reserve liquid in storage under the conditions of $-18\text{ }^{\circ}\text{C}$. Dissolve in *n*-hexane and prepare a solution of $100\text{ }\mu\text{g/mL} \sim 1\,000\text{ }\mu\text{g/mL}$ as the standards stock solution. At $0\text{ }^{\circ}\text{C} \sim 4\text{ }^{\circ}\text{C}$ under the conditions of storage.

3.11 Neutral aluminum oxide cartridge: 1 000 mg, 6 mL, or equivalent. Wash cartridge with 3 mL acetone, 5 mL *n*-hexane in sequence before use.

3.12 Florisil cartridge: 500 mg, 6 mL, or equivalent. Wash cartridge with 3 mL acetone, 5 mL *n*-hexane in sequence before use.

4 Apparatus and equipment

4.1 Gas chromatography equipped with mass selective detector (MSD).

4.2 Electronic balance; the sense of 0.000 1 g and 0.01 g.

4.3 SPE-12G Column Processor.

4.4 Grinder.

4.5 Centrifuge; 5 000 r/min.

4.6 Vortex mixer.

4.7 Rotary evaporator.

4.8 High speed homogenizer; 20 000 r/min.

4.9 Evaporated flask; 100 mL.

4.10 Centrifuge tubes with ground stopper; 15 mL, 50 mL.

4.11 Column of anhydrous sodium sulfate; $8.0\text{ cm} \times 1.5\text{ cm}$ (i. d.), prepacked with 5 cm Height of anhydrous sodium sulfate.

5 Preparation and storage of sample

5.1 Preparation of test sample

Reduce by quartering to ca 500 g, pulverize with a pulverizer (4.4) to let all pass a 2.0 mm round hole sieve. Mix thoroughly and divide into two equal portions as the test sample. Place in clean sample containers.

5.2 Storage of test sample

The test samples should be stored at 0 °C ~ 4 °C refrigerator. In the course of sampling and sample preparation, precautions should be taken to avoid contamination or any factors which may cause the change of residue content.

6 Procedure

6.1 Extraction

6.1.1 Rice, maize and wheat

Weigh ca. 5 g (accurate to 0.01 g) of the test sample into a 50 mL centrifuge tube, add 3 g of anhydrous sodium sulfate and 10 mL water and 20 mL *n*-Hexane-acetone solution (2 + 1, V/V) (3.6) in sequence. Homogenize the tube at 10 000 r/min for 2 min on high speed homogenizer, and then centrifuge at 4 000 r/min for 5 min. Transfer the supernatant layer into a 100 mL evaporated flask. The remaining solution is extracted again with 20 mL of *n*-Hexane-acetone solution (2 + 1, V/V). Combine the supernatant layer into the same 100 mL evaporated flask. Evaporate the solution to near dryness with rotary evaporator in a water-bath below 35 °C, then blow to dryness under a nitrogen flow. Dissolve the residues by adding 2 mL of *n*-hexane for cleaning up.

6.1.2 Soybean

Weigh ca. 5 g (accurate to 0.01 g) of the test sample into a 50 mL centrifuge tube, add 5 g of anhydrous sodium sulfate and 20 mL of acetonitrile in sequence. Homogenize the tube at 10 000 r/min for 2 min on high speed homogenizer, and then centrifuge at 4 000 r/min for 5 min. Transfer the supernatant layer into a 100 mL evaporated flask. The remaining solution is extracted again with 20 mL of acetonitrile. Combine the supernatant layer into the same 100 mL evaporated flask. Evaporate the solution to near dryness with rotary evaporator in a water-bath below 35 °C, then blow to dryness under a nitrogen flow. Dissolve the residues by adding 2 mL of *n*-hexane for cleaning up.

6.2 Clean

6.2.1 Maize

Transfer the sample solution into neutral aluminum oxide cartridge (3.12) at a rate of 1 mL/min~2 mL/min, discard the effluent. Then elute the cartridge with 10 mL of *n*-hexane-acetone (3.8). Collect all eluates and blow to dryness in nitrogen evaporator below 60 °C. The residue is dissolved by adding 5 mL of *n*-hexane for GC-MS determination.

6.2.2 Rice, soybean and wheat

Transfer the sample solution into florisil cartridge (3.13) at a rate of 1 mL/min~2 mL/min, discard the effluent. Then elute the cartridge with 13 mL of *n*-hexane-acetone (3.7). Collect all eluates and blow to dryness in nitrogen evaporator below 60 °C. The residue is dissolved by adding 5 mL of *n*-hexane for GC-MS determination.

6.3 Determination

6.3.1 GC-MSD operation conditions

- a) Column: DB-5ms fused quartz capillary column, 30 m × 0.25 mm (i. d.), film thickness 0.25 μm, or the equivalent of Performance;
- b) Column temperature: Initial temperature 110 °C hold for 1 min, ramp at 15 °C/min to 260 °C, hold for 15 min;
- c) Inlets temperature: 250 °C;
- d) Interface temperature: 280 °C;
- e) Carrier gas: Helium, purity ≥99.999%, flow rate = 1.0 mL/min;
- f) Injection volume: 1 μL;
- g) Injection mode: splitless, purge after 0.75 min;
- h) Ionization mode: EI;
- i) Ionization energy: 70 eV;
- j) Ionization source temperature: 230 °C;
- k) Quadropole temperature: 150 °C;

- l) Acquisition mode: Selected Ion Monitoring (SIM) mode;
- m) Selected monitoring ions (m/z): See table 1 and annex B;
- n) Solvent delay time: 5.0 min.

Table 1—Parameter of selected monitoring ion in MS table

Channels	Time/min	Selected monitoring ion (m/z)	Dwell/ms
1	5.00	223, 269, 160, 188, 237, 222, 151, 265, 162, 238, 240, 146	40
2	9.20	252, 162, 253, 281, 176, 311, 237, 160, 173, 145, 323, 262, 238	30
3	10.60	119, 91, 269, 120, 266, 394, 267, 245, 192, 136, 148, 298	40

6.3.2 GC-MS determination and confirmation

According to the estimated approximate concentration of per herbicide in the sample solution, select the standard working solution of similar concentration to that of sample solution. The responses of herbicide in the standard working solution and the sample solution should be in the linear range of the instrumental detection. The standard working solution should be injected randomly in-between the injections of the sample solution of equal volume. If there any peak of any peak of sample solution appeared at the same retention as such peak of the standard solution, the ratio of the chromatographic retention time of analyte shall correspond to that of the calibration solution at a tolerance of $\pm 2.5\%$. The relative intensities of the detected ions of each analyte shall correspond to those of the calibration standard solution at comparable concentrations, the allowed relative deviation of relative intensity is less than within the table 2 tolerance, it must be confirmed by selected monitoring ion (m/z) of species and abundance ratio see annex B. Under the above GC-MS condition, the retention time of 11 herbicide for GC-MS chromatogram (TIC) of the standards, see annex B and figure C. 1 in annex C.

Table 2—Maximum permitted tolerances for relative ion intensities while confirmation

Relative ion intensities/%	>50	>20~50	>10~20	≤ 10
Permitted relative tolerances/%	± 10	± 15	± 20	± 50

6.4 Blank test

The operation of the blank test is the same as that described in the method of determination, but without addition of sample. Calculation and expression of the result.

6.5 Calculation and expression of result

Calculate the content of per herbicide residue in the test sample by GC-MSD data processor or using the followed formula (1).

$$X_i = \frac{A_i \times c_{is} \times V \times 1\,000}{A_{is} \times m \times 1\,000} \dots\dots\dots (1)$$

Where

X_i —the residue content of per herbicide in the test samples,mg/kg;

A_i —the peak area of per herbicide in the test sample solution;

c_{is} —the concentration of per herbicide in the standard working solution,μg/mL;

A_{is} —the peak area of per herbicide in the standard working solution;

V —the final volume of sample solution,mL;

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

7 Limit of determination and recovery

7.1 Limit of determination

Use the method to determination 11 herbicide residues in rice,wheat,maize and soybean,and LQD is 0.01 mg/kg.

7.2 Recovery

Range for recovery of this method see table 3.

Table 3—Recovery range of 11 herbicide in rice,wheat maize and soybean

No.	Compound name	Range of recovery (Spiked level is 0.01 mg/kg~2.0 mg/kg)/%			
		rice	wheat	maize	soybean
1	Acetochlor	87.1~98.6	79.4~100.7	82.5~108.7	87.1~105.2
2	Alachlor	85.3~106.5	83.1~111.0	80.0~108.3	75.3~101.2
3	Esprocarb	80.5~102.1	76.5~104.0	84.3~101.6	81.6~108.1
4	Metolachlor	85.3~111.8	85.6~110.6	84.7~100.9	88.0~111.1
5	Pendimethalin	86.6~104.1	77.3~117.2	84.2~107.1	85.6~110.6
6	Butachlor	80.5~94.7	85.3~106.7	86.8~104.2	80.0~101.2
7	Flutolanil	84.9~102.5	88.2~107.6	82.4~101.2	82.7~105.3
8	Pretilachlor	85.9~110.6	82.1~106.3	87.3~107.1	79.5~105.3
9	Mepronil	80.5~99.7	89.4~101.7	80.3~101.1	75.3~103.2
10	Diflufenican	76.7~104.9	73.8~102.5	72.4~107.1	78.1~102.7
11	Mefenacet	85.3~107.2	79.3~112.5	75.8~100.3	85.3~107.2

Annex A
(normative)
Basic Information

Table A. 1—Species of 11 herbicides

No.	Pesticides (Chinese)	Pesticides (English)	CAS No.	Molecular formula
1	乙草胺	acetochlor	34256-82-1	$C_{14}H_{20}ClNO_2$
2	甲草胺	alachlor	15972-60-8	$C_{14}H_{20}ClNO_2$
3	戊草丹	esprocarb	85785-20-2	$C_{15}H_{23}NOS$
4	异丙甲草胺	metolachlor	51218-45-2	$C_{15}H_{22}ClNO_2$
5	二甲戊灵	pendimethalin	40487-42-1	$C_{13}H_{19}N_3O_4$
6	丁草胺	butachlor	23184-66-9	$C_{17}H_{26}ClNO_2$
7	氟酰胺	flutolanil	66332-96-5	$C_{17}H_{16}F_3NO_2$
8	丙草胺	pretilachlor	51218-49-6	$C_{17}H_{26}ClNO_2$
9	灭锈胺	mepronil	55814-41-0	$C_{17}H_{19}NO_2$
10	吡氟酰草胺	diflufenican	83164-33-4	$C_{19}H_{11}F_5N_2O_2$
11	苯噻酰草胺	mefenacet	73250-68-7	$C_{16}H_{14}N_2O_2S$

Annex B

(informative)

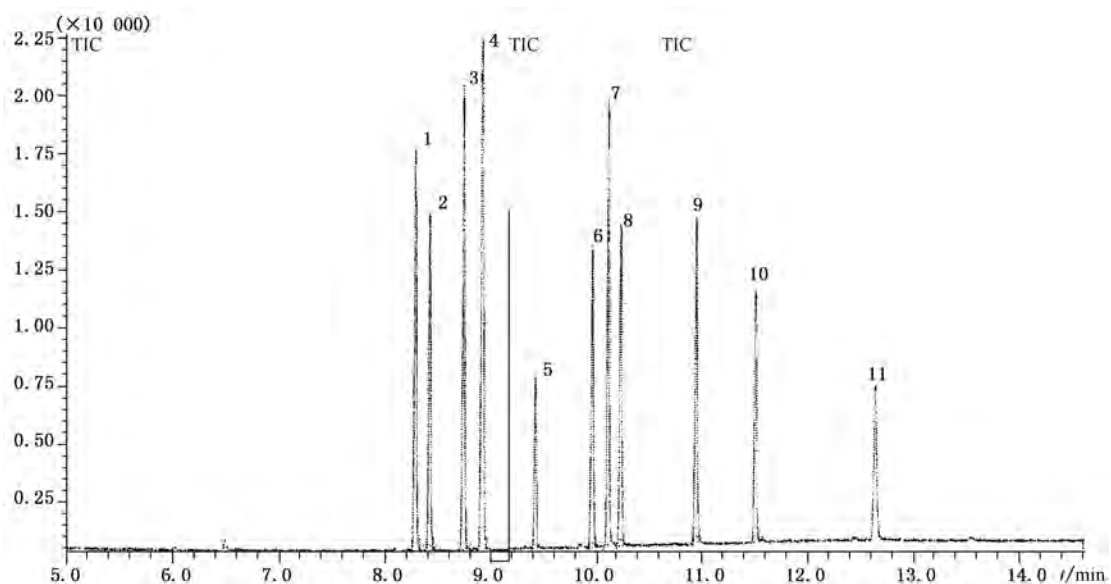
The retention time, determination and confirmation selected monitoring ion and
limit of determination of 11 herbicide

Table B. 1—Retention time, determination and confirmation selected monitoring ion and
limit of determination of 11 herbicide

No.	Pesticides	time/min	Characteristic fragment ion (amu)		
			determination	confirmation	Abundance ratio
1	Acetochlor	8.268	223	146, 162, 269	63 : 100 : 75 : 9
2	Alachlor	8.405	160	188, 237, 269	100 : 84 : 24 : 9
3	Esprocarb	8.731	222	162, 151, 265	100 : 67 : 25 : 12
4	Metolachlor	8.910	162	238, 240, 146	100 : 45 : 14 : 11
5	Pendimethalin	9.408	252	162, 253, 281	100 : 22 : 10 : 14
6	Butachlor	9.945	176	311, 237, 160	90 : 100 : 24 : 7
7	Flutolanil	10.098	173	145, 281, 323	100 : 27 : 26 : 13
8	Pretilachlor	10.220	262	162, 176, 238	46 : 100 : 92 : 73
9	Mepronil	10.937	119	91, 269, 120	100 : 26 : 18 : 9
10	Diflufenican	11.493	266	394, 267, 245	100 : 21 : 16 : 13
11	Mefenacet	12.629	192	136, 148, 298	100 : 25 : 19 : 8

Annex C
(informative)

GC-MS chromatography (TIC) of the 11 herbicide standard



- 1—Acetochlor;
- 2—Alachlor;
- 3—Esprocarb;
- 4—Metolachlor;
- 5—Pendimethalin;
- 6—Butachlor;
- 7—Flutolanil;
- 8—Pretilachlor;
- 9—Mepronil;
- 10—Diflufenican;
- 11—Mefenacet.

Figure C. 1—GC-MS chromatography (TIC) of the 11 herbicide standard

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