SN

中华人民共和国进出口商品检验行业标准

SN 0594—1996

出口肉及肉制品中西玛津残留量检验方法

Method for the determination of simazine residues in meat and meat products for export

1996-11-15发布　　1997-05-01实施

中华人民共和国国家进出口商品检验局　发布

前　言

本标准是根据GB/T 1.1—1993《标准化工作导则　第1单元：标准的起草与表述规则　第1部分：标准编写的基本规定》及SN/T 0001—1995《出口商品中农药、兽药残留量及生物毒素检验方法标准编写的基本规定》的要求进行编写的。其中测定方法是参考国内外有关文献，经研究、改进和验证后而制定的。本标准同时制定了抽样和制样方法。

测定低限是根据国际上对肉和肉制品中西玛津残留量的最高限量和测定方法的灵敏度而制定的。

本标准的附录A是提示的附录。

本标准由中华人民共和国国家进出口商品检验局提出并归口。

本标准起草单位：中华人民共和国辽宁进出口商品检验局和沈阳农业大学。

本标准主要起草人：姜莉、牛森、周艳明、宫英姿、宋文斌。

本标准系首次发布的行业标准。

中华人民共和国进出口商品检验行业标准

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1 范围

本标准规定了出口肉和肉制品中西玛津残留量的抽样、制样和气相色谱测定方法。

本标准适用于出口牛肉中西玛津残留量的检验。

2 抽样和制样

2.1 检验批

以不超过 2 500 件为一检验批。

同一检验批的商品应具有相同的特征，如包装、标记、产地、规格和等级等。

2.2 抽样数量

批量，件	最低抽样数，件
1～25	1
26～100	5
101～250	10
251～500	15
501～1 000	17
1 001～2 500	20

2.3 抽样方法

按 2.2 规定的抽样件数随机抽取，逐件开启。从每件内至少取 100 g 作为原始样品，原始样品总量不少于 2 kg。加封后，标明标记，及时送交实验室。

2.4 试样制备

将所取原始样品缩分出 1 kg，经均质机搅碎，混匀，均分成两份，分别装入洁净容器内，作为试样。密封并标明标记。

2.5 试样保存

将试样于－18℃以下冷冻保存。

注：在抽样和制样的操作过程中，必须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

试样经用二氯甲烷-甲醇混合液提取，提取液经弗罗里硅土柱净化，用配有氮磷检测器的气相色谱仪测定，外标法定量。

中华人民共和国国家进出口商品检验局 1996-11-15 批准　　1997-05-01 实施

3.2 试剂和材料

除另有规定外，所用试剂均为分析纯，水为重蒸馏水。

3.2.1 丙酮-石油醚混合液：(15+85)溶液。

3.2.2 二氯甲烷-甲醇混合液：(9+1)溶液。

3.2.3 无水硫酸钠：于650℃灼烧4 h，用前于105℃烘4 h，冷却后贮于密闭容器中，备用。

3.2.4 弗罗里硅土：层析用，100～200目，在650℃灼烧4 h，用前于130℃烘5 h，冷却后贮于密闭容器中，备用。

3.2.5 西玛津标准品：纯度≥98%。

3.2.6 西玛津标准溶液：准确称取适量的西玛津标准品，用丙酮配制成浓度为0.10 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 气相色谱仪：配有氮磷检测器。

3.3.2 旋转蒸发器。

3.3.3 超声波水浴。

3.3.4 弗罗里硅土柱：玻璃柱，40 cm×1.5 cm(内径)，自上而下依次填装2 cm高无水硫酸钠，10 g弗罗里硅土，2 cm高无水硫酸钠。使用前用丙酮-石油醚混合液(15+85)预淋洗。

3.4 测定步骤

3.4.1 提取

称取试样约30 g(精确至0.1 g)，加30 g无水硫酸钠(3.2.3)，研磨成粉状，转入锥形瓶中。加入100 mL二氯甲烷-甲醇(9+1)混合液，超声振荡20 min。用布氏漏斗抽滤，保留滤液。残渣再用100 mL二氯甲烷-甲醇混合液(9+1)，如上述操作。合并滤液于旋转蒸发器的蒸发瓶中，于50℃水浴中减压蒸至近干。

3.4.2 净化

用20 mL丙酮-石油醚(15+85)分三次将上述残渣溶解，溶液注入弗罗里硅土柱中净化，用100 mL丙酮-石油醚(15+85)洗脱，控制流速每秒2～3滴，流出液接收于旋转蒸发器的蒸发瓶中。于60℃水浴中减压蒸至近干，用丙酮定容至5.0 mL，供气相色谱法测定。

注：浓缩时避免蒸干。

3.4.3 测定

3.4.3.1 色谱条件

a) 色谱柱HP-1，5 m×0.53 mm(id)，膜厚2.65 μm；

b) 色谱柱温度：155℃；

c) 进样口温度：240℃；

d) 检测器温度：250℃；

e) 氮气：纯度≥99.99%，40 mL/min；

f) 氢气：2 mL/min；

g) 空气：140 mL/min；

h) 进样量：2 μL；

i) 进样方式：无分流进样。

3.4.3.2 色谱测定

根据样液中西玛津含量情况，选定峰高相近的标准工作溶液。标准工作溶液和样液中西玛津响应值均应在仪器检测线性范围内。对标准工作溶液和样液等体积参插进样测定。在上述色谱条件下，西玛津色谱峰保留时间约为2.2 min。西玛津标准品色谱图见附录A中图A1。

3.4.4 空白试验

除不加试样外，均按上述测定步骤进行。

3.4.5 结果计算和表述

用色谱数据处理机或按式(1)计算：

$$X = \frac{h \cdot c \cdot V}{h_s \cdot m} \quad \cdots\cdots(1)$$

式中：X——试样中西玛津残留含量，mg/kg；

h——样液中西玛津的色谱峰高，mm；

h_s——标准工作溶液中西玛津的峰高，mm；

c——标准工作溶液中西玛津的浓度，μg/mL；

V——样液最终定容体积，mL；

m——最终样液所代表的试样量，g。

注：计算结果需扣除空白值。

4 测定低限、回收率

4.1 测定低限

本方法的测定低限为 0.02 mg/kg。

4.2 回收率

回收率的实验数据：

西玛津的添加浓度在 0.02 mg/kg 时，回收率为 89.6%；

西玛津的添加浓度在 0.05 mg/kg 时，回收率为 91.42%；

西玛津的添加浓度在 0.1 mg/kg 时，回收率为 89.87%。

附 录 A
（提示的附录）
标准品色谱图

图 A1 西玛津标准品色谱图

Foreword

This standard was drafted in accordance with the requirements of the GB/T 1.1—1993 "Directives for the work of standardization—Unit 1:Drafting and presentation of standards—Part 1:General rules for drafting standards"and SN/T 0001—1995"General rules for drafting the standard methods for the determination of pesticide,veterinary drug residues and biotoxins in commodities for export". The method of determination was drafted by referring to relevant domestic and foreign literatures through research,modification and verification. In addition,methods of sampling and sample preparation are also specified in this standard.

The limit of determination in this standard is defined on the basis of the current international maximum limit for simazine residues in meat and meat products and the sensitivity of the method.

Annex A of this standard is the informative annex.

This standard was proposed by and is under the charge of the State Administration of Import and Export Commodity Inspection of the People's Republic of China.

This standard was drafted by Liaoning Import and Export Commodity Inspection Bureau of the People's Republic of China and Shenyang Agriculture University.

The main drafters of this standard are Jiang Li,Nu Shen,Zhou Yanming,Gong Yingzi,Song Wenbin.

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.

Professional Standard of the People's Republic of China for Import and Export Commodity Inspection

SN 0594—1996

Method for the determination of simazine residues in meat and meat products for export

1 Scope

This standard specifies the methods of sampling, sample preparation and determination of simazine residues by gas chromatography in meat and meat products for export.

This standard is applicable to the determination of simazine residues in beef for export.

2 Sampling and sample preparation

2.1 Inspection lot

The quantity of an inspection lot should not be more than 2 500 packages.

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

2.2 Quantity of sample taken

Number of packages in each inspection lot	Minimum number of package to be taken
1—25	1
26—100	5
101—250	10
251—500	15
501—1 000	17
1 001—2 500	20

2.3 Sampling Procedure

A number of packages specified in 2.2 are taken at random and opened one by one. The sample weight taken as the primary sample from each package should be at least 100 grams. The total weight of all primary samples should not be less than 2 kg, which shall be sealed, labeled and sent to laboratory in time.

2.4 Preparation of test sample

The combined primary sample is reduced to 1 kg, which is blended, homogenized thoroughly, and then divided into two equal portions. Each portion is placed in a clean container as the test sample, which is sealed and labeled.

2.5 Storage of test sample

The test sample should be stored below −18℃.

Approved by the State Administration of Import and Export Commodity Inspection of the People's Republic of China on Nov. 15, 1996

Implemented from May 1, 1997

Note: In the course of samping and sample preparation, precaution must be taken to avoid the contamination or any factors which may cause the change of residue content.

3 Method of determination

3.1 Principle

The simazine in test sample is extracted with dichloromethane-methanol mixed solution. The extract is cleaned up through florisil column and the solution is analyzed by GC with nitrogen-phosphorus detector, using external standard method.

3.2 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade, "Water" is distilled water.

3.2.1 Acetone-petroleum ether mixed solution: (15+85) solution.

3.2.2 Dichloromethane-methanol mixed solution: (9+1) solution.

3.2.3 Anhydrous sodium sulfate: Ignite at 650℃ for 4 h. Heat at 105℃ for 4 h before use. Store in a desiccator, ready for use.

3.2.4 Florisil: For chromatographic use, 100—200 mesh, ignite at 650℃ for 4 h. Heat at 130℃ for 5 h before use. Store in a desiccator, ready for use.

3.2.5 Simazine standard: Purity≥98%.

3.2.6 Simazine standard solution: Accurately weigh an adequate amount of simazine standard, dissolve with acetone to form the standard stock solutions of 0.10 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 Chromatograph: Equipped with nitrogen phosphorus detector.

3.3.2 Rotary evaporator.

3.3.3 Supersonic water-bath.

3.3.4 Florisil column: 40 cm×1.5 cm(id), glass column, filled successivly with 2 cm height of anhydrous sodium sulfate, 10 grams of florisil and 2 cm height of anhydrous sodium sulfate. Before use, prewash with acetone-petroleum ether mixed solution(15+85).

3.4 Procedure

3.4.1 Extraction

Weigh ca 30 g of the test sample (accurate to 0.1 g), add 30 g of anhydrous sodium sulfate (3.2.3), grind to dry powder, then transfer into a conical flask. Add 100 mL of dichloromethane-methanol (9+1) mixed solution, vibrate for 20 min by supersonic means. Filter the extract with a Buchner funnel, reserve the filtrate.

Extract the residue once more with 100 mL of dichloromethane-methanol mixed solution. Combine the filtrates in an evaporating bottle of rotary evaporator, and the solution is concentrated to near dryness in a 50℃ water-bath under reduced pressure.

3.4.2 Cleanup

Dissolve the residue three times with 20 mL in total of acetone-petroleum ether(15+85). Remove onto florisil column and elute with 100 mL of acetone-petroleum ether (15+85), controlling the flowing speed(2—3 drops/sec). Receive the eluate in an evaporating bottle of rotary evaporator, and the solution is concentrated to near dryness in a 60℃ water-bath under reduced pressure, and exactly diluted to 5 mL with acetone. The solution is used for chromatographic determination.

Note: In the course of concentration, avoid evaporating to dryness.

3.4.3 Determination

3.4.3.1 GC operating condition

a) GC column: HP-1, 5 m×0.53 mm(id), film thickness: 2.65 μm;

b) Column temperature: 155℃;

c) Injection port temperature: 240℃;

d) Detector temperature: 250℃;

e) Nitrogen: Purity≥99.99%, 40 mL/min;

f) Hydrogen: 2 mL/min;

g) Air: 140 mL/min;

h) Injection volume: 2 μL;

i) Injection mode: Splitless.

3.4.3.2 GC determination

According to the approximate concentration of simazine in the sample solution, select the standard working solution with similar peak height to that of sample solution. The responses of simazine in the standard solution and 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 sample solution of equal volume. Under the above chromatographic condition, the retention time of simazine is about 2.2 min. For chromatogram of simazine standard, see fig. A1 in annex A.

3.4.4 Blank test

The operation of the blank test is the same as that described in the method of determination but without addition of sample.

3.4.5 Calculation and expression of result

The calculation of result is carried out by GC data processor or according to formula(1):

$$X = \frac{h \cdot c \cdot V}{h_s \cdot m} \qquad \cdots\cdots(1)$$

where

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

h—the peak height of simazine in the sample solution, mm;

h_s—the peak height of simazine in the standard working solution, mm;

c—the concentration of simazine in the standard working solution, μg/mL;

V—the final volume of sample solution, mL;

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

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

4 Limit of determination and recovery

4.1 Limit of determination

The limit of determination of this method is 0.02 mg/kg.

4.2 Recovery

According to the experimental data, the fortified concentrations of simazine and its corresponding recoveries are as follows:

0.02 mg/kg, the recovery 89.6%;

0.05 mg/kg, the recovery 91.42%;

0.1 mg/kg, the recovery 89.87%.

Annex A

(informative)

Chromatogram of the standard

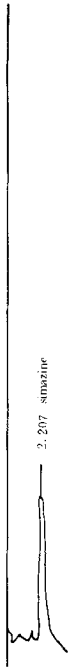

Fig. A1 Chromatogram of simazine standard