SN

中华人民共和国进出口商品检验行业标准

SN 0593－1996

出口肉及肉制品中辟哒酮残留量检验方法

Method for the determination of pyrazon 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》的要求而进行编写的。其中测定方法采用了美国食品药物管理局农药残留量分析手册(FDA，"Pesticide Analytical Manual"，Volume Ⅱ，Sec. 180.316)中辟哒酮残留量分析方法。但在技术内容上稍作改变，经验证后，按规定格式要求作了编辑性修改。在标准中同时制定了抽样和制样方法。

测定低限是根据国际上对肉及肉制品中辟哒酮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：中华人民共和国山东进出口商品检验局。

本标准主要起草人：沈志刚、李立、穆兆纯、张宁、王洪兵。

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及肉制品中辟哒酮检验的抽样、制样和气相色谱测定方法。

本标准适用于出口猪肉和牛肉中辟哒酮残留量的检验。

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。从每件中取一袋作为原始样品。总量不少于 2 kg。放入清洁容器内，加封后，标明标记，及时送交实验室。

如每件中无小包装或有小包装但每袋重量超过 2 kg 者，可用锋利刀(用酒精灭菌过)在抽出的包件中，每件割取不少于 100 g，混合后置于洁净容器内，作为混合原始样。混合原始样的重量不少于 2 kg。加封后，标明标记，及时送交实验室。

2.4 试样制备

从所取混合原始样中取出代表性样品约 1 kg，取可食部分，经组织捣碎机捣碎均匀，均分成两份，装入洁净容器内，作为试样。密封，标明标记。

2.5 试样保存

将试样于－18℃以下冷冻保存。

注：在抽样及制样的操作过程中，必须防止样品受到污染或发生残留物含量的变化。

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 测定方法

3.1 方法提要

样品中的辟哒酮经丙酮-乙腈溶液提取、冷冻过滤、净化，以正己烷为溶剂，用配有电子俘获检测器的气相色谱仪测定，外标法定量。

3.2 试剂和材料

除另有规定外，试剂均为分析纯，水为蒸馏水。

3.2.1 无水硫酸钠：经650℃灼烧4 h，置于干燥器内冷却备用。

3.2.2 乙腈：重蒸馏。

3.2.3 丙酮：重蒸馏。

3.2.4 丙酮-乙腈溶液：5+95。

3.2.5 正己烷：重蒸馏。

3.2.6 辟哒酮标准品：纯度≥99%。

3.2.7 辟哒酮标准溶液：准确称取适量的辟哒酮标准品，用正己烷配成浓度为100 μg/mL的标准储备溶液。使用时根据需要再用正己烷稀释成适当浓度的标准工作溶液。

3.3 仪器和设备

3.3.1 气相色谱仪：配有电子俘获检测器。

3.3.2 微量注射器：10 μL。

3.3.3 高速组织捣碎机。

3.3.4 恒温水浴。

3.3.5 布氏漏斗。

3.4 测定步骤

3.4.1 提取、净化

称取约20 g试样(精确至0.1 g)置于研钵中，加60 g无水硫酸钠，研磨均匀后，移于250 mL锥形瓶中，加入100 mL丙酮-乙腈(3.2.4)溶液，振荡30 min，通过布氏漏斗抽滤，用2×40 mL丙酮-乙腈溶液同上操作。合并滤液。

将滤液于－18℃冰箱中放置1 h，然后迅速过滤于200 mL容量瓶中，用丙酮定容。

准确移取10 mL净化液于具塞试管内，氮气吹干，准确加入1.00 mL正己烷，溶解残渣，供气相色谱测定。

3.4.2 测定

3.4.2.1 色谱条件

a) 毛细管色谱柱：HP-17，10 m×0.53 mm(id)，膜厚：2.0 μm，或相当者；

b) 柱温：240℃；

c) 进样口温度：270℃；

d) 检测器温度：300℃；

e) 载气：氮气，纯度≥99.99%，15 mL/min；

f) 进样量：1 μL；

g) 进样方式：无分流进样。

3.4.2.2 色谱测定：根据试样中辟哒酮含量情况，选定峰高相近的标准工作液，标准工作液和样液中辟哒酮响应值均应在仪器检测线性范围内。对标准工作液和样液等体积参插进样测定。在上述色谱条件下，辟哒酮的保留时间约为6 min。辟哒酮标准品的色谱图见附录A中图A1。

3.5 空白试验

除不称取试样外，均按上述测定步骤进行。

3.6 结果计算和表述

用色谱数据处理机或按式(1)计算试样中辟哒酮残留含量：

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

式中：X——试样中辟哒酮残留量含量，mg/kg；

A——样液中辟哒酮的色谱峰面积，mm^2；

A_s——标准工作液中辟哒酮的色谱峰面积，mm^2；

c——标准工作液中辟哒酮的浓度，μg/mL；

V——样液最终定容体积，mL；

m——最终样液相当的试样重量，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 0.01 mg/kg。

4.2 回收率

回收率的实验数据：

猪肉中辟哒酮添加浓度在 0.01 mg/kg 时，回收率为 80.0%；

猪肉中辟哒酮添加浓度在 0.10 mg/kg 时，回收率为 87.9%；

猪肉中辟哒酮添加浓度在 0.50 mg/kg 时，回收率为 98.7%；

牛肉中辟哒酮添加浓度在 0.01 mg/kg 时，回收率为 80.0%；

牛肉中辟哒酮添加浓度在 0.10 mg/kg 时，回收率为 93.6%；

牛肉中辟哒酮添加浓度在 0.50 mg/kg 时，回收率为 99.6%。

附 录 A
（提示的附录）
标准品色谱图

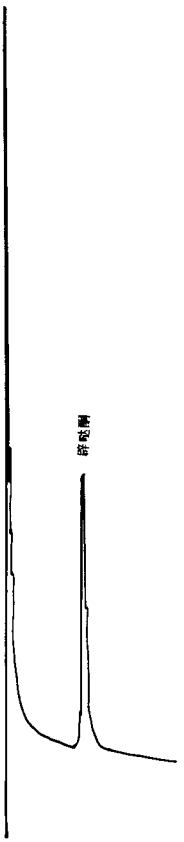


图 A1 辟哒酮标准品气相色谱图

Foreword

This standard was drafted in accordance with the requirements of 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". This standard adopts the relevant method for the determination of pyrazon residues in "Pesticide Analytical Manual,Vol. I ,Sec. 180.316",FDA of U.S.A. as the method of determination,which is technically equivalent to that of the original,but with slight modification. After going through verification,this standard was drafted according to the editorial requirement for stipulated format. Further,the methods of sampling and sample preparation are specified in the standard.

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

Annex A of this standard is an 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 Shandong Import and Export Commodity Inspection Bureau of the People's Republic of China.

The main drafters of the standard are Shen Zhigang,Li Li,Mu Zhaochun,Zhang Ning,Wang Hongbing.

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 0593—1996

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

1 Scope

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

This standard is applicable to the determination of pyrazon residues in pork and 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, specification and grade, should be the same.

2.2 Quantity of sample taken

Number of packages in each inspection lot	Minimum number of packages 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. From each, at least one bag shall be taken as the primary sample. The total weight of all the primary samples should not be less than 2 kg, which shall be placed in a clean container, sealed, labeled and sent to laboratory in time.

In case the frozen meat-pieces are not contained in small bags inside each package, or if there are small bags inside the package but the content of the bag exceeds 2 kg, cut out a part from the meat in each package of not less than 100 g with a sharp knife (disinfected with alcohol). Mix the parts of the meat as the mixed primary sample, which shall not be less than 2 kg. Place in a clean container, seal, label and send to the laboratory in time.

2.4 Preparation of test sample

The combined primary sample is reduced to 1 000 g, the edible portions are blended, and then di-

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, 199

vided into two equal portions. Each portion is placed in a clean container as the test sample, which is then sealed and labeled.

2.5 Storage of test sample

The test sample should be stored below −18℃.

Note: 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 contnt.

3 Method of determination

3.1 Principle

The pyrazon in the test sample is extracted with acetonitrile-acetone solution. Clean up by cooling and filtering. Determination is made by GC with electorn capture detector, using external standard method.

3.2 Reagents and materials

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

3.2.1 Anhydrous sodium sulfate: Ignite at 650℃ for 4 h, and store in a desiccator.

3.2.2 Acetonitrile: Redistill.

3.2.3 Acetone: Redistill.

3.2.4 Acetone-acetonitrile solution: (5+95).

3.2.5 n-Hexane: Redistill.

3.2.6 Pyrazon standard: Purity≥99%.

3.2.7 Pyrazon standard solution: Accurately weigh an adequate amount of pyrazon standard, dissolve in n-hexane and prepare a solution of 100 μg/mL in concentration as the standard stock solution. According to the requirement, prepare a standard working solution of appropriate concentration by diluting the stock solution with n-hexane.

3.3 Apparatus and equipment

3.3.1 Gas chromatograph: Equipped with election capture detector.

3.3.2 Micro-syringe: 10 μL.

3.3.3 High speed blender.

3.3.4 Water-bath, thermostatic.

3.3.5 Buchner funnel.

3.4 Procedure

3.4.1 Extraction and cleanup

Weigh ca 20 g (accurate to 0.1 g) of the test sample into a mortar, add 60 g of anhydrous sodium sulfate, grind to homogenize, and transfer into a 250 mL conical flask. Add 100 mL of acetone-acetonitrile solution (3.2.4), and extract on the shaker for 30 min. Filter by suction throngh a Buchner funnel. Repeat the process with 2×40 mL of acctone-acetonitrile solution (3.2.4). Combine the extracts the solution should be store below −18℃ for 1 h. Then filter promptly. Transfer the filtrate to a 200 mL volumetric flask and dilute to the mark with acetone. Pipet exactly 10 mL of the extract into a test tube with stopper, blow to dryness with a stream nitrogen. Then exactly 1.00 mL of n-hexane is added to dissolve the residue, the solution is used for GC determination.

3.4.2 Determination

3.4.2.1 GC operating condition

a) Capillary column: HP-17, 10 m×0.53 mm (id), film thickness 2.0 μm, or the equivalent;

b) Column temperature: 240℃;

c) Injection port temperature: 270℃;

d) Detector temperature: 300℃;

e) Carrier gas: Nitrogen, purity≥99.99%, 15 mL/min;

f) Injection volume: 1 μL;

g) Injection mode: Splitless.

3.4.2.2 GC determination

According to the approximate concentration of pyrazon in the sample solution, select the standard working solution with similar peak height to that of sample solution. The responses of pyrazon in the standard working solution and sample solution should be in the linear detecting range of the instrument. The standard working solution should be injected randomly in-between the injections of sample solution of equal volume. Under the above GC operating condition, the retention time of pyrazon is ca 6 min. For chromatogram of pyrazon standard, see fig. 1 in annex A.

3.5 Blank test

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

3.6 Calculation and expression of result

The calculation of the content of pyrazon in the test sample is carried out by GC data processor or according to formula(1):

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

where

X——Content of pyrazon in test sample, mg/kg;

A——Peak area of pyrazon in sample solution, mm^2;

A_s——Peak area of pyrazon in standard working solution, mm^2;

c——Concentration of pyrazon in the standard working solution, μg/mL;

V——Final volumn of the sample solution, mL;

m——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 0.01 mg/kg.

4.2 Recovery

According to the experimental data, the fortifying concentrations of pyrazon and its corresponding recoveries are:

In pork, 0.01 mg/kg, the recovery 80.0%;

0.10 mg/kg, the recovery 87.9%;

0.50 mg/kg, the recovery 98.7%.

In beef, 0.01 mg/kg, the recovery 80.0%;

0.10 mg/kg, the recovery 93.6%;

0.50 mg/kg, the recovery 99.6%.

Annex A

(informative)

Chromatogram of the standard

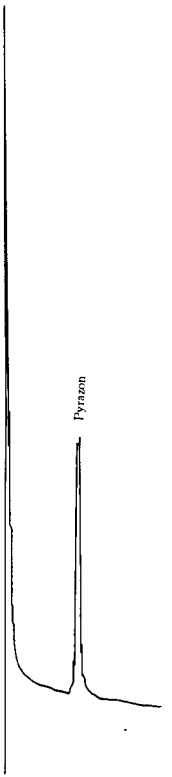


Fig. A1 GC chromatogram of pyrazon standard