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中华人民共和国出入境检验检疫行业标准

SN/T 1055—2002

出口坚果中氧化苯丁锡残留量检验方法 原子吸收分光光度法

Method for the determination of fenbutatin oxide residues in nuts for export—Atomic absorption spectrophotometric method

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前　　言

本标准是按照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》和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。

本标准的测定低限,是在参考若干国家及国际组织对商品中氧化苯丁锡最高残留限量(MRL)的规定和测定方法灵敏度的基础上而确定的。

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:中华人民共和国北京出入境检验检疫局。

本标准主要起草人:王嘉瑞、邹连生、刘岩、张世忠。

本标准首次发布。

中华人民共和国出入境检验检疫行业标准

出口坚果中氧化苯丁锡残留量检验方法 原子吸收分光光度法

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Method for the determination of fenbutatin oxide residues in nuts for export—Atomic absorption spectrophotometric method

1 范围

本标准规定了出口坚果中氧化苯丁锡残留量检验的抽样、制样和原子吸收分光光度测定方法。

本标准适用于出口栗子、核桃等坚果中氧化苯丁锡残留量的检验。

2 抽样和制样

2.1 抽样批次

以同种类、同等级为一抽样批次，每个抽样批次不得大于 50 t。

2.2 抽样数量

50 件及以下抽取 5 件。

51～100 件按件数的 10%抽取。

101～500 件以 100 件抽取 10 件为基础，其余件数按 6%抽取。

501～1 000 件以 500 件抽取 34 件为基础，其余件数按 3%抽取。

计算样件数量时，不足一件者按一件计。每件抽样数量应基本一致，核桃不得少于 20 颗，每批样品不得少于 500 颗；栗子每件抽样数量不得少于 500 g，每批样品不得少于 4 kg。

2.3 试样制备

将所取原始样品缩分出 1 kg，取可食部分，经样品均质器碎化，均分成 2 份，装入洁净容器内，作为试样，密封，并标明标记。

2.4 试样保存

将试样于－18℃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

以二氯甲烷-冰乙酸混合溶剂，提取试样中氧化苯丁锡残留，提取液经离心分离，浓缩至干后，残渣用湿法消化，溶解定容后，用配有氢化物发生系统、外焰加热石英管原子化器的原子吸收分光光度计测定，外标法定量。

3.2 试剂和材料

除另有注明外，试剂均为分析纯，水为去离子水。

3.2.1 提取剂：二氯甲烷-冰乙酸，6＋4(V/V)。

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3.2.2 浓硫酸:优级纯。

3.2.3 过氧化氢:30%,优级纯。

3.2.4 甲基紫指示剂:0.1 g 甲基紫溶于 100 mL 水中。

3.2.5 广范 pH 试纸。

3.2.6 10 mol/L 氢氧化钠溶液:40 g 氢氧化钠溶于 100 mL 水中。

3.2.7 碘化钾溶液:20 g 碘化钾溶于 100 mL 水中。

3.2.8 硫酸溶液:5 mL 浓硫酸溶于 1 000 mL 水中(氢化物发生系统用)。

3.2.9 0.025 mol/L 氢氧化钠溶液:1.0 g 氢氧化钠溶于 1 000 mL 水中。

3.2.10 硼氢化钠溶液:10.0 g 硼氢化钠溶于 1 000 mL 0.025 mol/L 的氢氧化钠溶液中(氢化物发生系统用)。

3.2.11 苯。

3.2.12 氧化苯丁锡标准品:纯度≥97%。

3.2.13 氧化苯丁锡标准溶液:根据标准品的纯度计算后,准确称取适量的氧化苯丁锡标准品,用苯配成浓度为 1.000 mg/mL 的标准储备溶液 100 mL,分装于 10 mL 的刻度试管中保存。再配制浓度为 0.0100 mg/mL 的标准工作溶液 100 mL,分装于 10 mL 的刻度试管中保存。

3.3 仪器和设备

3.3.1 原子吸收分光光度计:配有氢化物发生系统和外焰加热石英管原子化器。

3.3.2 容量瓶:100 mL。

3.3.3 刻度试管:具磨口塞,10 mL。

3.3.4 样品均质器。

3.3.5 振荡器。

3.3.6 离心瓶:具磨口塞,50 mL。

3.3.7 离心机:LD4-2 型医用离心机,或能与离心瓶相配合的其他类型。

3.3.8 凯氏烧瓶: 250 mL。

3.3.9 比色管:具磨口塞,25 mL。

3.3.10 玻璃珠:直径约 5 mm。

3.3.11 电热套:220 V,200 VA,容积 1 000 mL。

3.3.12 电调压器:0～220 V, 1 000 VA。

3.4 测定步骤

3.4.1 提取

称取试样约 5 g(精确到 0.1 g)于离心瓶内,加 25 mL 二氯甲烷-冰乙酸提取剂,振荡 45 min,振荡频率为 180 次/min。离心 5 min,转速为 2 000 r/min。将上清液转移至凯氏瓶中,转移时,用离心瓶塞挡住任何浮渣(果仁皮)流出。再用 25 mL 提取剂做第 2 次提取,用 20 mL 提取剂做第 3 次提取(第 3 次提取的振荡时间减为 20 min)。合并全部上清液于凯氏瓶中。

3.4.2 蒸发

加 4 粒玻璃珠于上述凯氏烧瓶中,在风力强劲的通风柜中,用电调压器和电热套,蒸发凯氏烧瓶中的溶剂。初始蒸发阶段,电压限定在 140 V,以防喷溅,蒸发到液量约为 10 mL 以下时,电压调定到 160 V继续加热蒸发,直至残留的油脂或者固体残渣的颜色变为棕黑色或黑色为止,取出,放冷约 2 min。为确保安全,整个蒸发期间,周围不得使用明火。

3.4.3 消化

3.4.3.1 炭化

于上述凯氏烧瓶中,加入 2 mL 浓硫酸(残渣为油脂时,加 2.5 mL 浓硫酸),再置凯氏瓶于通风柜里的电热套中,加热炭化残渣,电压限定在 160 V,以防过热,对油脂残渣而言,炭化到刚产生大量黑色泡

沫为止，取出，放冷 2 min。

3.4.3.2 消化

加入 10 mL 过氧化氢于上述凯氏瓶中，置凯氏瓶于通风柜里的电热套中，加热消化残渣，电压限定在 160 V，以防反应过于激烈，至过氧化氢作用完全时，立即取出（不得延误，延误将导致硫酸熬干），放冷 2 min。重复加过氧化氢，直至消化液变为无色透明时为止，取出，放冷 2 min。

注：对油脂残渣而言，在过氧化氢第一次作用完全时，出现激烈的炭化反应，同时产生大量的黑色泡沫，此时应立即取出凯氏瓶，放冷 2 min，然后再从加过氧化氢开始，重复消化操作。过氧化氢作用完全时，反应的激烈程度，一次比一次缓和，产生的黑色泡沫的量，一次比一次少。未消化掉的残液的颜色，也一次比一次浅，由最初的黑色，变为深棕色、浅棕色、黄色、浅黄色直至最后消化完全时，残液变为无色。

3.4.3.3 消化凯氏瓶颈上的残留物

加入 20 mL 过氧化氢于上述凯氏瓶中，凯氏瓶放回通风柜里的电热套中，电压调定到 200 V，消化至溶液冒白烟，呈透明无色，无任何微小气泡时为止，以确保过氧化氢被完全分解，并确保硫酸不被挥发至干。取出凯氏瓶，放冷后，供配制原子吸收测试溶液用。

3.4.4 标准工作溶液的消化

按 5 g 试样量计算，从浓度为 0.010 0 mg/mL 的氧化苯丁锡标准工作溶液中，准确吸取 0.00，0.10，0.20，0.30，0.40 mL（相当于试样中氧化苯丁锡含量为 0.00，0.20，0.40，0.60，0.80 mg/kg），分别放入一组编号的凯氏烧瓶中，各加入 4 粒玻璃珠，在风力强劲的通风柜中，用电热套和电调压器，电压调定在 140 V，加热挥发凯氏烧瓶中的溶剂至干，取出放冷，加入 0.5 mL 浓硫酸，20 mL 过氧化氢，电压调定在 200 V，加热消化，消化至溶液冒白烟，呈透明无色，无微小气泡时为止，以确保过氧化氢被完全分解，并确保硫酸不被挥发至干。取出凯氏瓶，放冷后，供配制原子吸收测试溶液用。

3.4.5 原子吸收测试溶液的配制

3.4.5.1 消化残液的溶解和定量转移

分 3 次，每次 5 mL 水，分别溶解、洗涤凯氏烧瓶中的标准和试样的消化残液，定量转移至比色管中，供降低酸度和定容使用。

3.4.5.2 降低测试溶液的酸度并定容

降低酸度操作，应逐一在比色管中进行，以防止指示剂过早加入而失效。往比色管中加入 6 滴甲基紫指示剂，摇匀，用 10 mol/L 的氢氧化钠溶液，分次加入，降低溶液的酸度，并不断用流水冷却比色管，溶液由黄色，变为黄绿色、绿色直至蓝色为止。降低酸度的操作，必须一次完成，中间不得中断，否则指示剂在低酸度条件下很快退色失效，使降低酸度的操作失败。为确保酸度不会降低过头显碱性，用广范 pH 试纸检验一下调酸后的溶液，试纸显示酸色即可。再加入 5 mL 碘化钾溶液，用水稀释至 25 mL 刻度线，摇匀，静置 5 min 后，进行原子吸收测定。

3.4.6 测定

3.4.6.1 原子吸收测定条件

配有氢化物发生系统、外焰加热石英管原子化器的原子吸收分光光度计的测定条件如下：

a）波长：224.6 nm；

b）狭缝宽度：1.30 nm；

c）灯电流：12.5 mA；

d）火焰：空气-乙炔焰；

e）燃气流量：2.0 L/min；

f）空气压强：160 kPa；

g）空气流量：15.0 L/min；

h）时间恒定值：2.00 s；

i）测量时间：15.0 s；

j) 延迟时间:5 s;

k) 测量次数:2;

l) 浓度单位:mg/kg(样品中氧化苯丁锡的含量单位);

m) 进样体积:5 mL。

3.4.6.2 原子吸收测定

按照设定好的仪器测试条件,测定每份标准的测试溶液和试样的测试溶液的原子吸收吸光度。

3.4.7 空白试验

除不加试样外,均按上述测定步骤进行。

3.4.8 结果计算与表述

用原子吸收分光光度计的数据处理机,或按式(1)计算试样中氧化苯丁锡的残留量:

$$X = \frac{A - A_0}{k} \qquad \cdots\cdots(1)$$

式中:X——试样中氧化苯丁锡残留量,mg/kg;

A——试样的原子吸收吸光度测定值;

A_0——空白试验原子吸收吸光度测定值;

k——标准曲线线性回归方程斜率,$(mg/kg)^{-1}$。

4 方法的测定低限、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

回收率的试验数据:试样中氧化苯丁锡的添加浓度在 0.10～0.80 mg/kg 范围时,回收率为 87.5%～110.0%。

Foreword

This standard was drafted in accordance with the requirement 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."

The method of determination of this standard was drafted by referring to relational domestic and foreign literature 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 limits for fenbutatin oxide residues in foodstuffs and the sensitivity of the method.

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

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

The main drafters of this standard are Wang Jiarui,Zou Liansheng,Liu Yan,Zhang Shizhong.

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 Entry-Exit Inspection and Quarantine

Method for determination of fenbutatin oxide residues in nuts for export— Atomic absorption spectrophotometric method

SN/T 1055—2002

1 Scope

This standard specifies the method of sampling, sample preparation and determination of fenbutatin oxide residues in nuts for export by the atomic absorption spectrophotometry.

This standard is applicable to the determination of fenbutatin oxide residues in nuts such as sweetnut, walnut for export.

2 Sampling and sample preparation

2.1 Inspection lot

An inspection lot should be same type and same grade, each lot should not be more than 50 tons.

2.2 Quantity of sample taken

Number of piece in each inspection lot	Minimum number of piece to be opened
≤50	5
51 ~ 100	10%
101 ~ 500	10 piece of 100 piece + 6% remainder piece.
501 ~ 1 000	34 piece of 500 piece + 3% remainder piece.

When calculate the number of piece, less than one piece should be assumed as one piece. The quantity taken from one piece should be same, for walnut, should not be less than 20 grains a piece; for sweetnut, should not be less than 500 grams a piece. For one inspection lot, the quantity of sample taken should not be less than 4 kg.

2.3 Preparation of test sample

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

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sealed and labeled.

2.4 Storage of test sample

The test samples should be stored at - 18℃.

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

3 Method of determination

3.1 Principle

After the sample is extracted by the dichloromethane-glacial acetic acid mixed solvent, the solution is centrifuged, separated, and concentrated to dryness, the dryness residue is digested by sulfuric acid and oxide reagents, then the digested solution is diluted to a definite volume. Finally, the solution is analyzed by the atomic absorption spectrophotometer equipped with a hydride formation system and an external flame heating quartz tube for the atomic vapor formation, using external standard method for the quantitative determination.

3.2 Reagents and materials

Unless otherwise specified, the purity of all of reagents is A.R., the water is distilled water.

3.2.1 Extraction reagent: dichloromethane-glacial acetic acid, 6 + 4(*V/V*).

3.2.2 Concentration sulfuric acid: High class purity.

3.2.3 Hydrogen peroxide: 30%, high class purity.

3.2.4 Methyl violet indicator solution: 0.1 gram methyl violet is dissolved in 100 mL water.

3.2.5 Universal indicator paper: pH 1 ~ 14.

3.2.6 10 mol/L solution of sodium hydroxide: 40 gram sodium hydroxide is dissolved in 100 mL water.

3.2.7 Potassium iodide solution: 20 grams potassium iodide is dissolved in 100 mL water.

3.2.8 Solution of sulfuric acid: 5 mL concentrated sulfuric acid dissolved in 1 000 mL water (to be used for hydride formation system).

3.2.9 0.025 mol/L solution of sodium hydroxide: 1.0 gram sodium hydroxide is dissolved in 1 000 mL water.

3.2.10 Sodium borohydried: 10.0 gram sodium borohydride is dissolved in 1 000 mL 0.025 mol/L solution of sodium hydroxide (to be used for hydride formation system).

3.2.11 Benzene.

3.2.12 Fenbutatin oxide standard material: Purity: ≥97%.

3.2.13 Fenbutatin oxide standard solution: After the calculation according to the purity of fenbutatin oxide, accurately weigh adequate amount of fenbutatin oxide standard material, dissolved in benzen and prepare 100 mL solution of the concentration as 1.000 mg/mL, divided into the 10 mL graduated test tubes as the standard stock solution. Then prepare again 100 mL solution of the concentration as 0.010 0 mg/mL, divided into the 10 mL graduated test tubes as the standard working solution.

3.3 Apparatus and equipment

3.3.1 The atomic absorption spectrophotometer: Equipped with a hydride formation system and an external flame heating quartz tube for atomic vapor formation.

3.3.2 Measuring flask: 100 mL.

3.3.3 Graduated test tube: 10 mL, with ground stopper.

3.3.4 Sample homogenizer.

3.3.5 Shaker.

3.3.6 Centrifuge bottle: 50 mL, with ground stopper.

3.3.7 Centrifuger: LD-4 model, or other model that can match with the centrifuge bottle.

3.3.8 Kjeldahl's flask: 250 mL.

3.3.9 Colorimetric test tube: 25 mL, with ground stopper.

3.3.10 Glass pearl: about 5 mm diameter.

3.3.11 Electrical heating mantle: 220 V, 200 VA, size 1 000 mL.

3.3.12 Voltage adjuster: 0 ~ 220 V, 1 000 VA.

3.4 Procedure

3.4.1 Extraction

Weigh ca 5 g of the test sample (accurate to 0.1 g) into a centrifuge bottle, add 25 mL dichloromethane-glacial acetic acid mixed extract reagent and stopper, the bottle is shaken for 45

min. by a shaker, shaking frequency is about 180 times/min. Then the centrifuge bottle is centrifuged for 5 min by a centrifuger, rotating speed is about 2 000 r/min, then the clean solution is decanted into the Kjeldahl's flask, during the decanting, the floating dregs (such as peel of kernel) is kept off by ground stopper of centrifuge bottle to prevent decanting out. Then the test sample is extracted second time by 25 mL, third time by 20 mL extract reagent (during third extracting, the shaken time is reduced as 20 min). All of the clean solutions are combined into the Kjeldahl's flask.

3.4.2 Evaporation

Add 4 pellets of glass pearls into the above Kjeldahl's flask, in the draught chamber with a strong wind power, heat the Kjeldahl's flask by the electrical heating mantle and the voltage adjuster to evaporate the extract solvent. During the initial evaporating, the voltage is kept at 140 V to prevent erupting. When the volume of solvent is less than 10 mL, the voltage is increased to 160 V to continue heating for evaporation, when the color of residue (oil or solid residue) is getting dark brown or dark, stop heating, take out the flask to cool about 2 min. For safety reason, guarantee no flame exist on the surrounding during the whole evaporating.

3.4.3 Digestion

3.4.3.1 Carbonization

Add 2 mL concentrated sulfuric acid (for oil residue, add 2.5 mL) into above Kjeldahl's flask, then put it back into the electrical heating mantle in the draught chamber, heat the flask to carbonize the residue, the voltage is adjusted as 160 V to prevent over heat. For oil residue, the carbonizing should not stop until a great number of dark foam just is occur, then the flask is taken out to cool about 2 min.

3.4.3.2 Digestion

Add 10 mL hydrogen peroxide into the above Kjeldahl's flask, then put it back into the electrical heating mantle in the draught chamber, heat and digest the content in flask, the voltage is adjusted as 160 V to prevent the reaction from too drastically. When the hydrogen peroxide is reacted out, take out the flask right away (should not delay, if delay, the sulfuric acid will be dried out) to cool about 2 min. Repeat adding the hydrogen peroxide operation and don't stop until the digested residue liquid has become clear and transparent, take out the flask to cool about 2 min.

Note: For oil residue, when the hydrogen peroxide is reacted out for the first time, the violent carbonizing reaction is occurred, at the same time a great number of dark foam is occurred, at this time the flask should be taken out to cool about 2 min, then repeat the digestion operation with adding the peroxide hydrogen. Whenever the hydrogen peroxide is reacted out, the reaction is more gentle than last time is, the amount of dark foam is less than last time is , the color of residue liquid is lighter than last time is, it is progressively getting from initial black, to dark brown, light brown, yellow, light yellow, and finally the color of residue liquid is colorless and transparent.

3.4.3.3 Digestion of residue on the inside wall of the neck of Kjeldahl's flask

Add 20 mL hydrogen peroxide into the above Kjeldahl's flask, then put it back into the electrical heating mantle in the draught chamber, the voltage is adjusted as 200 V, continue to digest till the solution has become clear and transparent, and produces the white smog, does not appear any micro-bubble, to ensure that residue hydrogen peroxide has decomposed out, but the sulfuric acid has not be dried out, then take out the flask to cool. The cool digested residue liquid will be prepared into testing solution for AAS.

3.4.4 Digestion of standard working solution

After calculate the each volume of standard working solution according to the amount of 5 g test sample, then accurately take out 0.00, 0.10, 0.20, 0.30, 0.40 mL (They are equivalent to fenbutatin oxide content in the test samples separately is 0.00, 0.20, 0.40, 0.60, 0.80 mg/kg.) from the 0.010 0 mg/mL standard working solution, separately put them into a group of numbered Kjeldahl's flasks, add 4 pellets of glass pearls into each flask, in the draught chamber with a strong wind power, heat the Kjeldahl's flask by the electrical heating mantle and voltage adjuster, the voltage is kept at 140 V, to evaporate the solvent to dryness, then take out flask to cool, and add 0.5 mL concentrated sulfuric acid and 20 mL hydrogen peroxide, the voltage is adjusted as 200 V, heat the flask for the digestion till the residue liquid has become clear and transparent, produces the white smog, does not appear any micro-bubble, to ensure that residue hydrogen peroxide has decomposed out, but the sulfuric acid has not be dried out. Then take out the flask to cool. The cool digested residue liquid will be prepared into testing solution for AAS.

3.4.5 Preparation of testing solution for AAS

3.4.5.1 Dissolve and quantitative transfer of the digested residue liquid

Divide 3 times, add 5 mL water each time, dissolve and rinse the digested residue liquid of the standard and the test sample in the Kjeldahl's flask, quantitatively transfer into a colorimetric test tube for preparing into testing solution for AAS.

3.4.5.2 Decrease the acidity of the testing solution

The operation of decreasing the acidity, should be proceeded in the colorimetric test tube one by one to prevent the indicator solution losing function due to it added into the tube too early. Add 6 drops of methyl violet indicator solution into the colorimetric test tube, shake to the uniformity, decrease the acidity of the solution with 10 mol/L sodium hydroxide, at the same time, cool the colorimetric test tube by cool flowing water, the color of the solution gradually is getting from yellow into yellow green, green to final blue, then stop adding alkaline solution. The operation of decreasing acidity in the every colorimetric test tube must be completed continually, should not be interrupted, otherwise the indicator will be faded quickly nuder the low acidity condition and lost its function, make the operation of decreasing acidity failed. To ensure the acidity does not decreases excessively to alkaline, test the solution after the decreasing acidity operation with universal indicator paper. If the paper is still kept red, the acidity of solution is correct. Then dilute the solution with water to the graduated level of 25 mL, shake to the uniformity, after staying 5 min, it is supplied for the determination of AAS.

3.4.6 Determination

3.4.6.1 AAS operating condition

The operating condition of the atomic absorption spectrophotometer equipped with a hydride formation system and an external flame heating quartz tube for atomic vapor formation is follows:

a) Wavelength: 224.6 nm;
b) Slit width: 1.30 nm;
c) Lamp current: 12.5 mA;
d) Flame: Air-C_2H_2;
e) Fuel flow: 2.0 L/min;
f) Air pressure: 160 kPa;
g) Air flow: 15.0 L/min;
h) Time constant: 2.00 s;
i) Measurement time: 15.0 s;
j) Delay time: 5 s;
k) Replicates: 2;
l) Concentration unit: mg/kg (content unit of fenbutatin oxide in sample);
m) Injection volume: 5 mL.

3.4.6.2 AAS determination

According to the operating condition that has set well, determine the absorption value of each testing solution of standard and sample.

3.4.7 Blank test

The blank test is proceeded according to the above procedure, but without adding of sample.

3.4.8 Calculation and expression of result

Calculate the residue content of the fenbutatin oxide in the test sample using the data processor of the atomic absorption spectrophotometer, or using the following formula:

$$X = \frac{A - A_0}{k} \qquad \cdots\cdots (1)$$

where

X—The residue content of fenbutatin oxide in test sample, mg/kg;
A—The atomic absorption value of test sample;
A_0—The atomic absorption value of blank;
k—The slope of standard curve, $(mg/kg)^{-1}$.

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

According to the experiment data, when the adding concentration of fenbutatin oxide in the test sample is in the range of 0.10 ~ 0.80 mg/kg, the recovery is 87.5% ~ 110.0%.

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