SN

B04

中华人民共和国进出口商品检验行业标准

SN 0340—95

出口粮谷、蔬菜中百草枯残留量检验方法 紫外分光光度法

Method for the determination of paraquat residues in cereals, vegetables for export —UV-spectrophotometric method

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中华人民共和国国家进出口商品检验局　发布

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1 主题内容与适用范围

本标准规定了出口粮谷、蔬菜中百草枯残留量检验的抽样、制样和紫外分光光度测定方法。

本标准适用于出口大米、白菜中百草枯残留量的检验。

2 抽样和制样

2.1 大米抽样

2.1.1 检验批

以不超过 4 000 袋为一检验批。

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。

2.1.2 抽样数量

按一批总袋数的平方根抽取：

$$a = \sqrt{N} \qquad \cdots\cdots(1)$$

式中：a ——抽样袋数；

N ——全批袋数。

注：a 值取整数，小数部分向前进位为整数。

2.1.3 抽样工具

2.1.3.1 单管取样器：不锈钢管，全长 55 cm(包括手柄)，直径 1.5 cm，沟槽长度应超过袋对角线长度的一半。

2.1.3.2 取样铲。

2.1.3.3 分样板。

2.1.3.4 样品筒(袋)：可密封。

2.1.3.5 分样布(分样)。

2.1.4 抽样方法

2.1.4.1 袋内抽样：按 2.1.2 规定计算抽样袋数(扣除倒包抽样袋数)，在堆垛四周上、中、下各部位以曲线形走向，随机抽取。将扦槽朝下，从每袋一角依斜对角方向插入袋内，将扦槽朝上旋转 180°，抽出扦样器立即倒入盛样容器内。每袋扦取样品数量应基本一致。

2.1.4.2 倒包抽样：从堆垛的各部位随机抽取 2.1.2 规定的应抽样件数的 10%(每批不少于 3 袋)，将袋口缝线全部拆开，平置于分样布或其他洁净的铺垫物上，双手紧握袋底两角提起约成 45°倾斜角，倒拖 1 m 以上，使袋内货物全部倒出后，用取样铲在各部位扦取样品约 100 g，立即倒入盛样容器内。

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2.1.4.3 大样缩分：集中袋内和倒包所取样品，倒于清洁分样布上，使用分样板，按四分法缩分样品不少于 4 kg，加封后，标明标记并及时送实验室。

2.1.5 试样制备

将样品缩分至 1 kg，全部磨碎，通过 20 目筛，混匀，均分成两份，装入洁净容器内，标明标记。

2.1.6 试样保存

将试样于－5℃以下避光保存。

2.2 蔬菜抽样

2.2.1 检验批

以不超过 1 000 件为一个检验批。

同一检验批内商品应具有同一特征，如包装、标记、产地、规格、等级等。

2.2.2 抽样数量

批量，件	最低抽样数，件
1～25	1
26～100	5
101～250	10
251～1 000	15

2.2.3 抽样工具

2.2.3.1 取样刀：不锈钢菜刀。

2.2.3.2 样品袋：聚乙烯塑料食品袋。

2.2.4 抽样方法

按 2.2.2 规定的抽样件数，在不同部位随机抽取，逐件开启。每件抽取样品不少于 500 g 为原始样品，原始样品总量不得少于 2 kg，将所取样品装入样品袋内，加封后，标明标记，及时送实验室。

2.2.5 试样制备

将所取原始样品混匀，取可食部分，切碎，按四分法缩分出 500 g，经组织匀浆机匀浆成均匀的样品，均分成两份，装入洁净的广口瓶中，密封，标明标记，作为实验室样品。

对于不易均匀的菜类样品，先将缩分过的样品切碎精确称重。然后将切碎的样品倒入组织匀浆机内，按样品重量的 20%（m/m）加入蒸馏水，匀浆。注意：后加入的水分在称样时要扣除。上述操作的每一步都应详细记录。

2.2.6 试样保存

将试样于－18℃冷冻保存，备用。

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变化。

3 测定方法

3.1 方法提要

试样中百草枯用硫酸溶液煮沸回流加以提取，提取液经阳离子交换树脂柱净化，百草枯被吸附在树脂上，然后，以饱和氯化铵溶液洗脱。于流出液中加入连二亚硫酸钠溶液，百草枯被还原为蓝色化合物，用紫外分光光度计进行定量。

3.2 试剂和材料

除特殊规定外，试剂为分析纯，水为蒸馏水或相应的去离子水。

3.2.1 硫酸（比重 1.84）。

3.2.2 氢氧化钠。

3.2.3 氯化钠。

3.2.4 盐酸（比重 1.18）。

3.2.5 氯化铵。

3.2.6 乙二胺四乙酸二钠(EDTA)。

3.2.7 苯。

3.2.8 连二亚硫酸钠。

3.2.9 硫酸溶液:9 mol/L。

3.2.10 盐酸溶液:2 mol/L。

3.2.11 氢氧化钠溶液:12.5 mol/L。

3.2.12 氢氧化钠溶液:10 mol/L。

3.2.13 氢氧化钠溶液:0.3 mol/L。

3.2.14 饱和氯化钠溶液:溶解 360 g 氯化钠于 1 L 水中,搅拌溶解,澄清备用。

3.2.15 饱和氯化铵溶液:溶解 370 g 氯化铵于 1 L 水中,搅拌溶解,过滤备用。

3.2.16 稀氯化铵溶液:1/10 饱和溶液。量取 1 份饱和氯化铵溶液加入 9 份水,混匀。

3.2.17 连二亚硫酸钠溶液:0.2%于 0.3 mol/L 氢氧化钠溶液中。溶解 0.20 g 连二亚硫酸钠于少量 0.3 mol/L的氢氧化钠溶液中,于棕色容量瓶内以该氢氧化钠溶液定容至 100 mL,混匀。此溶液必须在临使用前新鲜配制,超过 1.5 h 后不宜使用。

3.2.18 离子交换树脂:AG 50WX-8,100～200 目,在水中浸泡。

3.2.19 百草枯标准品:百草枯二氯化物含量大于 99%。

3.2.20 百草枯标准溶液:准确溶解 0.025 0±0.000 1 g 百草枯标准品于少量饱和氯化铵溶液中,转移至 250 mL 棕色容量瓶中并以饱和氯化铵溶液准确定容,摇匀,作为标准贮备液,此溶液百草枯二氯化物的浓度为 100 μg/mL。根据需要再配成适用浓度的标准工作液。

3.3 仪器和设备

3.3.1 紫外分光光度计:具有连续波长与吸收扫描功能,配备 5 cm 比色皿。

3.3.2 粮谷粉碎机:筛板孔径 1 mm。

3.3.3 组织匀浆机。

3.3.4 减压抽滤装置:配备 1 000 mL 抽滤瓶及 φ10 cm 平底漏斗。

3.3.5 加热回流装置:1 000 mL 圆底烧瓶及配套的球形冷凝管。

3.3.6 净化柱:50 mL 酸式滴定管。在玻璃柱的下部塞入一小团玻璃棉,高度约 0.5～1 cm,垂直固定好柱子,加入 10 mL 在水中浸泡并沉降的树脂,分别用 50 mL 饱和氯化钠溶液和 50 mL 水洗柱。注意保持液面略高于树脂层。每个试样测定均须使用一根新制备的柱子。

3.4 测定步骤

3.4.1 提取

称取粮谷试样约 50.0 g 或蔬菜试样 200.0 g(精确至 0.1 g)置于 1 000 mL 圆底烧瓶中,根据试样的含水量加入适量的水和 9 mol/L 的硫酸溶液,使瓶内溶液的总体积约为 200～300 mL(包括试样的含水量)、硫酸的浓度为 2.5 mol/L。加入数粒小玻璃珠,连接回流冷凝管。加热至沸(如产生大量气泡可加入几滴正辛醇)并使之回流 5 h 以上。取下烧瓶,冷却,加入 500 mL 水。将提取液倒入已铺垫好双层快速滤纸(含油试样可多垫几层滤纸)的平底漏斗上,用抽滤装置过滤,用少量水分数次洗涤。对非油类试样,将滤液倒入 1 000 mL 的烧杯中。对含油试样可将滤液倒入 1 000 mL 分液漏斗中,然后用 100 mL 苯分三次萃取,收集水相于 1 000 mL 烧杯中。于烧杯中的溶液中加入 12.5 mol/L 的氢氧化钠溶液,其体积相当于回流前所加 9 mol/L 硫酸溶液的量,加入 5 gEDTA,搅拌至完全溶解,加水至溶液总体积约 900 mL,然后再用 10 mol/L 的氢氧化钠溶液调节 pH 至 9。冷却至室温,将溶液全部倒入 1 000 mL 的分液漏斗中备用。

3.4.2 净化和洗脱

3.4.2.1 净化

将盛有提取液的分液漏斗固定在净化柱的上方(可用洁净的胶管将二者连接),调节活塞,使溶液以10～12 mL/min 的速度过柱。过柱后移开分液漏斗。然后以 5 mL/min 的速度依次用 50 mL 水、50 mL 2 mol/L 的盐酸溶液、50 mL 水和 50 mL 稀氯化铵溶液淋洗柱子,弃去所有流出液。

3.4.2.2 洗脱

将上述处理后的柱子用饱和氯化铵溶液洗脱柱上百草枯,洗脱速度为 0.5～1 mL/min,收集 50 mL 洗脱液于 50 mL 容量瓶中。

注:洗脱时柱温(环境温度)不应低于 20℃。

3.4.3 测定

3.4.3.1 测定波长的选择

吸取 10 mL 含百草枯二氯化物为 1.0 μg/mL 的百草枯标准工作液于 50 mL 比色管中,加入 2 mL 连二亚硫酸钠溶液,摇匀后立即倒入 5 cm 比色皿中,置于紫外分光光度计比色皿中进行测定,设定波长从 410 至 380nm 进行吸光度扫描,选择最大吸收峰处为测定波长 λ_m,然后分别选择 $\lambda_m \pm 4$nm 处为校正波长 λ_h 和 λ_l。

3.4.3.2 工作曲线的绘制

分别吸取百草枯标准贮备溶液 0.00 mL、0.05 mL、0.10 mL、0.25 mL、0.50 mL、0.75 mL、1.00 mL和 1.50 mL 于 8 只100 mL容量瓶中,各加入饱和氯化铵溶液至刻度。此工作液百草枯的浓度分别为 0.00 μg/mL、0.05 μg/mL、0.10 μg/mL、0.25 μg/mL、0.50 μg/mL、0.75 μg/mL、1.00 μg/mL和1.50 μg/mL。分别吸取上述工作液 10 mL 于 8 支 50 mL 比色管中,各加入 2 mL 连二亚硫酸钠溶液,混匀后立即用紫外分光光度计,在 λ_m 处以空白液对照测定各工作液的吸光度,以吸光度为纵坐标,相应浓度为横坐标绘制标准曲线。

同时于 λ_h 和 λ_l 处(与空白液对照)分别测定 1.00 μg/mL 百草枯工作液的吸光度。

3.4.3.3 样品测定

吸取 10 mL 洗脱液于 50 mL 比色管中,加入 2 mL 连二亚硫酸钠溶液,混匀后立即于 λ_m、λ_h 和 λ_l 处以空白液对照分别测定吸光度。

3.4.4 空白试验

除不称取样品外,按上述测定步骤进行。

3.5 结果计算和表述

按下式计算试样中百草枯(以百草枯二氯化物计)的残留量:

$$X = \frac{c \cdot V}{m} \qquad \cdots\cdots(2)$$

式中:X ——试样中百草枯二氯化物的残留量,mg/kg;

c ——从标准曲线中以样品的校正吸光度值($A_{校}$)查出对应的百草枯二氯化物的浓度,μg/mL;

V ——洗脱液最终定容体积,mL;

m ——称取的试样量,g。

试样的校正吸光度值按下式计算:

$$A_{校} = \frac{A_m^p}{2A_m^p - (A_h^p + A_l^p)}[2A_m - (A_h + A_l)] \qquad \cdots\cdots(3)$$

式中:$A_{校}$——试样的校正吸光度;

A_m^p ——λ_m 处百草枯二氯化物浓度为 1 μg/mL 时测得的吸光度;

A_h^p ——λ_h 处百草枯二氯化物浓度为 1 μg/mL 时测得的吸光度;

A_l^p ——λ_l 处百草枯二氯化物浓度为 1 μg/mL 时测得的吸光度;

A_m ——λ_m 处测得试样的吸光度;

A_h ——λ_h 处测得试样的吸光度;

A_1 ——λ_1 处测得试样的吸光度。

注：计算结果需扣除空白值。

4 方法的测定低限、回收率

4.1 测定低限

本方法测定低限为 0.02 mg/kg。

4.2 回收率

回收率实验数据，百草枯添加浓度在 0.02～10 mg/kg 范围内，回收率为 70.2%～108.0%。

附加说明：

本标准由中华人民共和国国家进出口商品检验局提出。

本标准由中华人民共和国海南进出口商品检验局负责起草。

本标准主要起草人欧伟、陈如娅、卓海华、张惠。

主要参考文献：

FDA-Pesticide Analytical Manual, Vol. Ⅱ, Pesticide Reg, Sec. 180.205, 1985.

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

SN 0340—95

Method for the determination of paraquat residues in cereals, vegetables for export —UV-spectrophotometric method

1 Scope and field of application

This standard specifies the method of sampling, sample preparation and determination by UV-spectrophotometry of paraquat residues in cereals and vegetables for export.

This standard is applicable to the determination of paraquat residues in rice and Chinese cabbage for export.

2 Sampling and samlpe preparation

2.1 Sampling of rice

2.1.1 Inspection lot

Each inspection lot should not exceed 4 000 bags.

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

2.1.2 Quantity of sample taken

The square root of the total number of bags in a lot shall be taken as the number of bags to be sampled:

$$a = \sqrt{N} \qquad \cdots\cdots(1)$$

where

a —Number of bags to be taken;

N —Total number of bags in a lot.

Note: If value a is with decimal, round off the decimal part, which is added as unity to the integral part of a.

2.1.3 Sampling tools

2.1.3.1 Sampler: Stainless steel tube. Length (including handle) 55 cm; diameter 1.5 cm; groove length is more than half of the bag's diagonal length.

2.1.3.2 Sampling shovel.

2.1.3.3 Plate for quartering.

2.1.3.4 Sample can (bag): Which can be sealed.

2.1.3.5 Cloth sheet:. For sample dividing (quartering).

2.1.4 Sampling procedure

2.1.4.1 Sampling from the bag

Approved by the State Administration of Import and Export Commodity Inspection of the People's Republic of China on May. 29, 1995

Implemented from Nov. 1, 1995

Draw the samples from a number of bags specified in 2.1.2 (minus the number of sampled bags by emptying out) as follows: Along the sine wave of the pile, draw samples from the bags of the upper, middle and lower parts of the pile at random. Insert the sampler, with its groove facing downward, diagonally into each bag, then turn the sampler by 180°, draw out the sampler, and promptly pour the sample into a container. The quantity of the sample drawn from each bag shall be basically the same.

2.1.4.2 Sampling by emptying out

Draw 10% of the number of bags specified in 2.1.2 (not less than 3 bags) at any part of the pile at random. Unseam and open the bag, and lay the bag on a clean cloth sheet or other clean sheet. Grasp tight two corners of the bag bottom and raise up to an angle of 45°, tug backward for ca 1 m until all content of the bag is emptied out. Scoop up the sample from different parts of the out-poured content with a shovel, totalling ca 100 g, and place in sample container promptly. Mix all the samples from different bags to form a gross sample.

2.1.4.3 Reduction of gross sample

Pour all the samples (from both 2.1.4.1 and 2.1.4.2) on a clean sheet for quartering, reduce to not less than 4 kg with a plate by quartering, place in a sample container, seal, label and send to the laboratory in time.

2.1.5 Preparation of test sample

Reduce the sample to ca 1 kg, grind thoroughly and let pass through a 20 mesh sieve, mix, divide into 2 equal portions, place in clean containers, seal and label.

2.1.6 Storage of test sample

The test sample shall be stored below −5℃ and kept away from light.

2.2 Sampling of vegetables

2.2.1 Inspection lot

The quantity of an inspection lot should not be more than 1 000 packages.

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

2.2.2 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—1 000	15

2.2.3 Sampling tools

2.2.3.1 Sampling knife: Stainless steel knife.

2.2.3.2 Sample bag: Polyethylene bag for foodstuff.

2.2.4 Sample procedure

A number of packages specified in 2.2.2 are taken at random and opened one by one. The sample weight taken as the primary sample from each package should be at least 500 g. The total weight of all primary samples should be not less than 2 kg, which shall be sealed, labeled and sent to laboratory in time.

2.2.5 Preparation of test sample

The edible portions of the combined primary sample is chopped and reduced to 500 g by quarter-

ing, then blended with a tissue blender and divided into two equal portions. Each portion is placed in a clean container as test sample, which is then sealed and labeled.

For the vegetable sample which is hard to blend, chop the sample first, then weigh accuratly. Transfer the chopped samples to a blender and according to the weight of sample, add 20% (*m/m*) distilled water. Blend it at high speed. The added water must be deducted from the weight of the sample for determination. Each step mentioned above must be recorded in detail.

2.2.6 Storage of test sample

The test samples should be stored at −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 content.

3 Method of determination

3.1 Principle

The paraquat test sample is extracted with sulfuric acid by reflux, the extract is purified with cationic exchange resin, then the paraquat is eluted with saturated ammonium chloride solution. Add sodium dithionite solution to the eluate, the paraquat is reduced resulting in a compound of blue coloration, which is determined by the UV-spectrophotometer.

3.2 Reagents and materials

Unless otherwise specified, all the reagents should be analytical pure, water is distilled water or corresponding de-ionized water.

3.2.1 Sulfuric acid (specific gravity 1.84).

3.2.2 Sodium hydroxide.

3.2.3 Sodium chloride.

3.2.4 Hydrochloric acid (specific gravity 1.18).

3.2.5 Ammonium chloride.

3.2.6 Ethylenediaminetetracetic acid disodium salt (EDTA).

3.2.7 Benzene.

3.2.8 Sodium dithionite.

3.2.9 Sulfuric acid: 9 mol/L.

3.2.10 Hydrochloric acid: 2 mol/L.

3.2.11 Sodium hydroxide solution: 12.5 mol/L.

3.2.12 Sodium hydroxide solution: 10 mol/L.

3.2.13 Sodium hydroxide solution: 0.3 mol/L.

3.2.14 Saturated sodium chloride solution: Dissolve 360 g sodium chloride in 1 litre of water, stir to dissolve and the clear solution is ready for use.

3.2.15 Saturated ammonium chloride solution: Dissolve 370 g ammonium chloride in 1 litre of water, stir to dissolve and use after filtration.

3.2.16 Dilute ammonium chloride solution: 1/10 saturated solution. Add 1 portion of the saturated ammonium chloride solution to 9 portions of water and mix well.

3.2.17 Sodium dithionite solution: 0.2% in 0.3 mol/L sodium hydroxide solution. Dissolve 0.20 g of sodium dithionite in a small partion of 0.3 mol/L sodium hydroxide solution and transfer to a 100 mL brown volumetric flask, dilute to mark with the same sodium hydroxide solution, mix thoroughly. Prepare just prior to use and no use is permissible after keeping more than 1.5 h.

3.2.18 Cationic exchange resin: AG 50 W X-8, 100—200 mesh, soaked in water.

3.2.19 Paraquat standard: Purity of paraquat dichloride is more than 99%.

3.2.20 Paraquat standard solution: Dissolve 0.025 0±0.000 1 g of paraquat standard in a small portion of saturated ammonium chloride solution, transfer into a 250 mL brown volumetric flask, dilute to mark with the same solution, mix thoroughly to be used as a standard stock solution, which contains 100 μg/mL of paraquat dichloride. Then dilute the solution to the required concentrations as the standard working solutions.

3.3 Apparatus and equipment

3.3.1 UV-spectrophotometer: With continuous scan function for wavelength and absorption, equipped with 5 cm colorimetric cells.

3.3.2 Pulverizer: The diameter of sieve aperture is 1 mm.

3.3.3 High-speed blender.

3.3.4 Vacuum filter device: With 1 000 mL vacuum filter flask and ϕ 10 cm Buchner funnel.

3.3.5 Heating and reflux device: With 1 000 mL boiling flasks and reflux condenser.

3.3.6 Cleanup column: 50 mL buret. Put a plug of glass wool at the bottom of column, about 0.5—1 cm in height, fix the column vertically, then add 10 mL of resin soaked and settled in water, pass 50 mL of saturated sodium chloride solution and then 50 mL of water through the column for washing, keep the surface of solution a little higher than the surface of resin. Use a freshly prepared column for each sample.

3.4 Procedure

3.4.1 Extraction

Weigh a representative sample of 50.0 g of the cereal or 200.0 g of vegetable (all accurate to 0.1 g) into a 1 000 mL boiling flask. In accordance with the water content of the sample, add adeguate amount of water and 9 mol/L H_2SO_4 to make up the total volume of solution in the flask to about 200—300 mL (including the content of sample water), concentration of H_2SO_4 solution being 2.5 mol/L. Heat under reflux (with glass beads added) to boiling (if excessive foaming occurs, add a few drops of n-capryl alcohol) and keep boiling for more than 5 hours, then remove the flask and let cool, then dilute with 500 mL H_2O. Filter the solution through a Buchner funnel with double layers of fast filter paper (more layer for oily sample) by vacunm filter device, then wash the funnel with water several times. Transfer the filtrate of the non-oily sample to a 1 000 mL beaker. For oil sample, transfer the filtrate to a 1 000 mL separatory funnel, extract three times with 100 mL in total of benzene, then transfer the aqueous phase to a 1 000 mL beaker, to the aqueous solution add a volume of 12.5 mol/L NaOH equivalent to the volume of 9 mol/L H_2SO_4 added prior to reflux. Add 5 g of EDTA, stir untill dissolved, then adjust the pH to 9 with 10 mol/L NaOH. Cool the solution to room temperature, then transfer it to a 1 000 mL separatory funnel for the next process.

3.4.2 Cleanup and elution

3.4.2.1 Cleanup

Set the separatory funnel containing the solution above the column for cleanup (both may connected with a clean rubber tube). Let the extract flow at a rate of 10—12 mL/min by adjusting the stop-cock. Remove away the separatory funnel. Rinse the column successively with 50 mL of water, 50 mL of 2 mol/L HCl, 50 mL of water and 50 mL of dilute NH_4Cl solution at the rate of 5 mL/min. Discard all the effluents.

3.4.2.2 Elution

Elute the paraquat on the column with saturated NH_4Cl with a flow rate of 0.5—1 mL/min, collect 50 mL of the eluate in a 50 mL volumetric flask.

Note: Column temperature (room temperature) should be not less than 20℃ during elution.

3.4.3 Determination

3.4.3.1 Selection of measuring wavelength

Pipet 10 mL of the standard solution containing 1.0 μg/mL paraquat dichloride into a 50 mL colorimetric tube, add 2 mL of sodium dithionite solution, shake well and immediately transfer the test solution into a 5 cm colorimetric cell, place it in the UV-spectrophotometer, scan for absorbances. at the wavelength range from 410 nm to 380 nm. Select the wavelength of maximum absorbance for measuring (λ_m) then select the $\lambda_m \pm 4$ nm as corrected wavelengths (λ_h and λ_l) respectively.

3.4.3.2 Preparation of the working curve

Respectively pipet 0.00 mL, 0.05 mL, 0.10 mL, 0.25 mL, 0.50 mL, 0.75 mL, 1.00 mL, 1.50 mL of paraquat standard stock solution into eight 100 mL volumetric flasks, and dilute to the mark with saturated ammonium chloride solution, mix well. The concentration of these paraquat standard working solution are 0.00 μg/mL, 0.05 μg/mL, 0.10 μg/mL, 0.25 μg/mL, 0.50 μg/mL, 0.75 μg/mL, 1.00 μg/mL, 1.50 μg/mL. Respectively pipet 10 mL standard working solutions into eight 50 mL colorimetric tubes, add 2 mL of sodium dithionite solution into each tube and shake. Immediately measure the absorbance at λ_m against the blank solution. Plot a working curve of absorbances (λ_m) (ordinate versus concentrations of paraquat dichloride (abscissa). Then measure the absorbance of 1.00 μg/mL paraquat working solution at λ_h and λ_l against the blank solution.

3.4.3.3 Determination of the test sample

Pipet 10 mL of the eluate aliquot into a colorimetric tube, add 2 mL of sodium dithionite solution, mix and immediately read the absorbances at λ_m, λ_h and λ_l against the blank solution.

3.4.4 Blank test

The operation of the blank test is the same as that described in the method of determination, but with omission of sample addition.

3.5 Calculation and expression of result

The content of paraquat (calculated as paraquat dichloride) in test the sample is calculated according to the following formula:

$$X = \frac{c \cdot V}{m} \qquad \cdots\cdots(2)$$

where

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

c —the concentration of paraquat dichloride in test sample obtained versus corrected absorbance ($A_{corr.}$) from the calibration curve, μg/mL;

V —the final volume of the eluate, mL;

m —the mass of the test sample, g.

Note: The corrected absorbance of the test sample is calculated according to the following formula:

$$A_{corr.} = \frac{A_m^p}{2A_m^p - (A_h^p + A_l^p)}[2A_m - (A_h + A_l)] \qquad \cdots\cdots(3)$$

where

$A_{corr.}$ —the corrected absorbance of the test sample;

A_m^p —the absorbance for a standard containing 1 μg/mL paraquat dichloride at λ_m;

A_h^p —the absorbance for a standard containing 1 μg/mL paraquat dichloride at λ_h;

A_l^p —the absorbance for a standard containing 1 μg/mL paraquat dichloride at λ_l;

A_m —the observed absorbance of test sample at λ_m;

A_h —the observed absorbance of test sample at λ_h;

A_l —the observed absorbance of test sample at λ_l.

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, when the fortifying concentration of paraquat dichloride is in the range of 0.02—10 mg/kg, the recovery is 70.2%—108.0%.

Additional explanations:

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

This standard was drafted by the Hainan Import and Export Commodity Inspection Bureau of the People's Republic of China.

This standard was mainly drafted by Ou Wei, Chen Ruya, Zhuo Haihua, Zhang Hui.

Reference:

FDA-Pesticide Analytical Manual, Vol. Ⅱ, Pesticide Reg, Sec. 180.205, 1985.

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