SN

中华人民共和国进出口商品检验行业标准

SN 0591—1996

出口粮谷及油籽中二嗯硫磷残留量检验方法

Method for the determination of dioxathion residues in cereals and oil seeds 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. 171)中二嗪硫磷残留量分析方法。技术内容与原方法相同，经验证后，按规定格式要求仅作了编辑性修改。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粮谷及油籽中二嗪硫磷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：陈冰、刘桂荣、穆兆纯、沈志刚、张宁。

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

本标准规定了出口粮谷及油籽中二嗯硫磷残留量检验的抽样、制样和气相色谱测定方法。

本标准适用于出口豌豆和花生仁中二嗯硫磷残留量的检验。

2 抽样和制样

2.1 检验批

以不超过 200 t 为一检验批。200 t 袋装豌豆约 2 200 袋；袋装花生仁约 2 400 袋。

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

2.2.1 袋装货品

按式(1)计算抽样袋数：

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

式中：N—— 全批袋数；

a—— 抽样袋数。

注：a 值取整数，小数部分向前进位为整数。

2.2.2 散积货品(豌豆)

货堆高度不超过 2 m。按货堆面积划区设点。以 50 m^2 为一个取样区，每区设中心及四角(距边线 1 m处)5 个点。每增加一个取样区，增设 3 个点。

2.3 抽样工具

对豌豆或花生仁不用单管取样品。

2.3.1 金属双套管取样器：全长 1 m，2 m(包括手柄)两种。内、外管同部位分段开几个槽口，每个槽口长 15～20 cm，口宽 2.0～2.5 cm。内管的内径为 2.5～3.0 cm；取样器的探头长约 7 cm。

2.3.2 取样铲或取样勺。

2.3.3 分样板。

2.3.4 盛样器：筒或袋，可密封。

2.3.5 分样布或适用铺垫物。

2.4 抽样方法

2.4.1 袋装抽样

2.4.1.1 倒包抽样：从堆垛的各部位随机抽取 2.2.1 规定的应抽样件数的 10%(每批一般不少于 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

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

角，倒拖约 1 m，使袋内货物全部倒出。查看袋内和袋间品质是否均匀。确认情况正常后，用取样铲随机在各部位抽取样品，并立即将样品倒入盛样器内。每袋抽取样品数量应基本一致。

2.4.1.2 袋内抽样：按 2.2.1 规定的应抽样件数（扣除倒包抽样件数），在堆垛四周上、中、下各层以曲线形走向随机抽取。然后按豌豆、花生仁，用下述方法进行取样：

对豌豆，用 1 m 长的金属双套管取样器（2.3.1），关闭槽口，从每袋一角依斜对角方向插入袋内，然后旋转内管以开启槽口，待样品流满内管后，再旋转内管以关闭槽口。抽出取样器，立即将样品倒入盛样器内。

对花生仁，将应抽各件拆开袋口缝线 3～5 针，用取样勺从开口处抽取样品。立即缝好袋口，并将所取样品倒入盛样器内。

每袋所取样品的量应与 2.4.1.1 基本一致。每批所抽取的样品总量应不少于 4 kg。

2.4.2 散积抽样：按 2.2.2 规定的取样点，逐点抽取样品。将取样器（2.3.1）槽口关闭，以倾斜 45°角度插入货堆至相应深度，旋转取样器内管以开启槽口，待样品流满内管后，再旋转内管以关闭槽口。抽出取样器，立即将样品倒入盛样器内。从各点所抽取的样品量应基本一致。

每批所抽取的样品总量应不少于 4 kg。

2.4.3 大样缩分

袋装样品：合并从袋内和倒包抽样所取全部样品，倒于分样布上，用分样板按四分法缩分出样品不少于 2 kg，盛于样品筒内，加封后标明标记并及时送交实验室。

散积样品：将抽取的全部样品，倒于分样布上，以下按上述袋装样品方法进行。

2.5 试样制备

2.5.1 制样工具

2.5.1.1 磨碎机。

2.5.1.2 粉碎机。

2.5.1.3 筛子：20 目筛，2.0 mm 圆孔筛（用于花生）。

2.5.1.4 分样板。

2.5.1.5 盛样瓶：具塞广口瓶。

2.5.2 制样方法

2.5.2.1 豌豆试样制备：将样品按四分法缩分至 1 kg，用磨碎机（2.5.1.1）全部磨碎并通过 20 目筛。混匀，均分成两份作为试样。分装入洁净的盛样器内，密封，标明标记。

2.5.2.2 花生仁试样制备：将样品按四分法缩分至 500 g，用样品粉碎机（2.5.1.2）粉碎，使全部通过 2.0 mm 圆孔筛。混匀，均分成两份作为试样。分装于洁净的盛样器内，密闭，标明标记。

2.6 试样保存

将试样于－5℃以下避光保存。

注：在抽样及制样操作过程中，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

豌豆或花生仁中残留的二噻硫磷采用乙腈提取，提取液经液液分配转入二氯甲烷中，再经活性炭柱净化，用配有火焰光度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，水为蒸馏水或相应的去离子水。

3.2.1 二氯甲烷：重蒸时收集 39℃～41℃馏分。

3.2.2 乙酸乙酯：重蒸馏。

3.2.3 乙腈：重蒸馏。

3.2.4 乙腈-水(2+1)溶液。

3.2.5 活性炭:于 1 L 烧杯中加入 100 g 粉末状活性炭、250 mL 浓盐酸,调成浆状,盖上表面皿,在磁力搅拌下煮沸 1 h。加 250 mL 水搅拌,再煮沸 30 min。经布氏漏斗过滤,并用水洗至中性,在 130℃烘箱中烘干,冷却后贮存于密闭容器中备用。

3.2.6 氧化镁:取 500 g 左右,用水调至浆状,在蒸汽浴上加热 30 min 后用布氏漏斗抽滤。在 105℃～130℃干燥过夜,冷却后密闭保存。

3.2.7 硅藻土:Celite 545。

3.2.8 混合吸附剂:将 1 份酸处理过的活性炭,2 份氧化镁和 4 份酸洗处理的硅藻土混匀,密闭保存。

3.2.9 二嗪硫磷标准品:纯度≥95%。

3.2.10 二嗪硫磷标准溶液:准确称取适量的二嗪硫磷标准品,用乙酸乙酯配制成浓度为 100 mg/L 储备液,4℃下保存。根据需要稀释成适当浓度的标准工作液。

3.2.11 洗脱溶液:乙腈-苯(1+1)。

3.3 仪器和设备

3.3.1 气相色谱仪并配有火焰光度检测器,磷滤光片 526 nm。

3.3.2 均质器。

3.3.3 旋转蒸发器。

3.3.4 活性炭柱:220 mm×18 mm(内径)层析柱,配有连接管连接真空滤器。将 1 g 硅藻土装入层析柱底部(轻击),加入 6 g 混合吸附剂(轻击),然后再装入 2 cm 的玻璃棉。

3.4 测定步骤

3.4.1 提取

称取约 10 g 试样(精确至 0.1 g),置于 250 mL 均质杯中,加入 30 mL 乙腈水溶液,高速均质3 min。抽滤,滤液转入 250 mL 分液漏斗,用 20 mL 乙腈水溶液洗均质杯及残渣,滤液并入分液漏斗。向分液漏斗中加入 50 mL 二氯甲烷,振荡 1 min,静置分层,将二氯甲烷层放入 100 mL 容量瓶,用 20,10 mL 二氯甲烷洗涤水相,合并二氯甲烷,并用二氯甲烷定容至刻度。

3.4.2 净化

用 100 mL 洗脱溶液预洗活性炭柱,弃去预洗液。从容量瓶中准确移取 20 mL 二氯甲烷液过柱,调节流量为 5 mL/min,然后用 120 mL 洗脱液洗脱,用数毫升乙酸乙酯淋洗柱端及真空滤器连接管。收集全部流出液,在 70℃水浴中浓缩至近干,用乙酸乙酯定容至 1.0 mL,供气相色谱测定。

3.4.3 测定

3.4.3.1 气相色谱条件

a) 色谱柱:填充柱,1.85 m×2 mm(内径),10%(m/m)OV-101 涂于 WHP(100～120 目)上;

b) 色谱柱温度:175℃;

c) 进样口温度:220℃;

d) 检测器温度:220℃;

e) 载气:高纯氮,纯度≥99.99%,30 mL/min;

f) 氢气:45 mL/min;

g) 空气:50 mL/min;

h) 氧气:10 mL/min;

i) 进样量:2 μL。

3.4.3.2 气相色谱测定

根据样液中被测农药含量情况,选定峰面积相近的标准工作液。标准工作液和待测样液中农药的响应值均应在仪器检测的线性范围内。对标准工作液与样液等体积参插进样测定。在上述色谱条件下,二嗪硫磷的保留时间约为 9 min。标准品色谱图见附录 A 中图 A1。

3.4.4 空白试验

除不称取试样外，均按上述测定步骤进行。

3.5 结果计算和表述

用色谱数据处理机或按式(2)计算试样中二嗪硫磷的残留含量：

$$X = \frac{A \cdot c \cdot V}{A_s \cdot m} \quad \cdots\cdots(2)$$

式中：X ——试样中二嗪硫磷含量，mg/kg；

A ——样液中二嗪硫磷的色谱峰面积，mm^2；

A_s——标准工作液中二嗪硫磷的色谱峰面积，mm^2；

c ——标准工作液中二嗪硫磷的浓度，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：计算结果需将空白值扣除。

4 测定低限、回收率

4.1 测定低限

本方法的测定低限为 0.05 mg/kg。

4.2 回收率

4.2.1 豌豆中二嗪硫磷的回收率实验数据：

二嗪硫磷添加浓度在 0.05 mg/kg 时，回收率为 81.0%；

二嗪硫磷添加浓度在 0.10 mg/kg 时，回收率为 92.6%；

二嗪硫磷添加浓度在 0.25 mg/kg 时，回收率为 97.1%。

4.2.2 花生仁中二嗪硫磷的回收率实验数据：

二嗪硫磷添加浓度在 0.05 mg/kg 时，回收率为 76.6%；

二嗪硫磷添加浓度在 0.10 mg/kg 时，回收率为 94.4%；

二嗪硫磷添加浓度在 0.25 mg/kg 时，回收率为 102.7%。

附 录 A
（提示的附录）
标准品气相色谱图

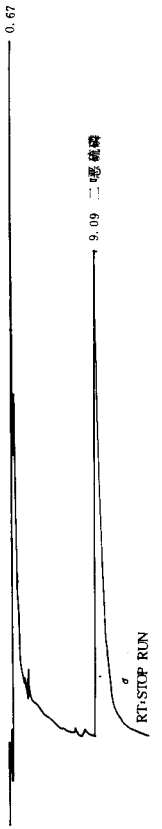


图 A1 二嗯硫磷标准品气相色谱图

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". In this standard, the testing method for dioxathion residues in (FDA, "Pesticide Analytical Manual," Volume I, Sec. 180.171) is adopted as the method of determination, which is thus technically identical with that of FDA; only minor editorial modification required for the stipulated format was made after going through verification. In addition, methods of sampling and sample preparation are also specified in this standard.

The limit of determination is defined in the standard on the basis of the current international maximum limit for dioxathion residues in cereals and oil seeds and the sensitivity of the method.

Annex A of this standard is an informative annex.

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 Chen Bing, Liu Guirong, Mu Zhaochun, Shen Zhigang, Zhang Ning.

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 0591—1996

Method for the determination of dioxathion residues in cereals and oil seeds for export

1 Scope

This standard specifies the method of sampling, sample preparation and determination of dioxathion residues by gas chromatography in cereals and oil seeds for export.

This standard is applicable to the determination of dioxathion residues contant in peas and peanuts for export.

2 Sampling and sample preparation

2.1 Inspection lot

Each inspection lot should not exceed 200 tonnes. An inspection lot of 200 tonnes for peas in bags shall be ca 2 200 bags, and for peanuts in bags shall be ca 2 400 bags.

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

2.2 Quantity of sample taken

2.2.1 Cargo in bags

Calculate the number of bags to be taken by formula(1):

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

where

N —total number of bags in a lot;

a —number of bags to be taken.

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

2.2.2 Cargo in bulk(peas)

The height of the cargo pile should not exceed 2 m. Set up areas and spots for sampling on the pile surface. 50 m^2 is considered as an area, in which 5 spots shall be fixed, one in the centre and four at four corners(1 m from the margins)of the area. For an additional area, three more sampling spots shall be fixed.

2.3 Sampling tools

For peas or peanuts don't use the metallic sampler.

2.3.1 Metallic double-casing sampler: Length 1 m, 2 m(both including handle)with some slots on different sections and respectively at the same heights for both inner and outer casings; length of slots: 15—20 cm, width of the slots: 2.0—2.5 cm; inside diameter of the inner casing: 2.5—3.0 cm; the probe length of the sampler: ca 7 cm.

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, 1996

2.3.2 Sampling shovel or ladle.

2.3.3 Plate for quartering.

2.3.4 Sample container: Can or bag, which can be seald.

2.3.5 Cloth (or other suitable material) sheet: For sample dividing (quartering).

2.4 Sampling procedure

2.4.1 For cargo in bags

2.4.1.1 Sampling by emptying out: Draw 10 percent of the number of bags specified in 2.2.1 (not less than 3 bags) at random. Unseam and open the bag, and lay it 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. Check whether the quality of goods is uniform within and between the bags. After confirming the goods are in normal condition, scoop up the sample from different parts of the out-poured content at random, and promptly place in a sample container. The quantity of sample drawn from each bag should be basically the same.

2.4.1.2 Sampling from inside the bags: Draw the samples from the number of bags specified in 2.2.1 (by deducting the number of bags drawn in 2.4.1.1) as follows: Along the sine wave of the pile, draw the samples from the bags of upper, middle and lower parts around the pile at random. For peas or peanuts proceed respectively as follows:

For peas, using the metallic double-casing sampler (2.3.1, length 1m) (the slots should be closed while inserting in) diagonally into each bag. Turn the inner casing to open the slots so that the sample may fill up the inner tube. Again turn the inner casing to close the slots and draw out the sampler. Promptly pour the sample into a sample container.

For peanuts, partially unseam the bag mouth (3—5 stitches), scoop out the sample from each bag (with a ladle) at the bag opening. Promptly close the bag by seaming, and pour the sample into a sample container.

The amount of sample drawn from each bag should be basically the same as in (2.4.1.1). The total weight of the sample of each lot should be not less than 4 kg.

2.4.2 Sampling from the cargo in bulk (for peas):

Insert the double-casing sampler (2.3.1) successively into the pile at the spots specified in 2.2.2, to appropriate depth at 45° (the slots should be closed while inserting in). Turn the inner casing to open the slots so that the sample may fill up the inner tube. Again turn the inner casing to close the slots and draw out the sampler. Promptly pour the sample into a sample container. The sample drawn from all the spots shall be basically the same.

The total weight of the sample of each lot should be not less than 4 kg.

2.4.3 Reduction of gross sample

For cargo in bags: Pour all the samples (from both 2.4.1.1 and 2.4.1.2) on a clean sheet. Reduce to not less than 2 kg by quartering with a plate. Place in a sample container, seal, label and send to the laboratory in time.

For cargo in bulk: Pour all the drawn sample onto a clean sheet and proceed as for cargo in bags described above.

2.5 Preparation of test sample

2.5.1 Tools

2.5.1.1 Grinder.

2.5.1.2 Pulverizer.

2.5.1.3 Sieve: 20-mesh sieve; 2.0 mm round-hole seive(for peanuts).

2.5.1.4 Plate for quartering.

2.5.1.5 Sample container: Wide-mouth bottle with ground stopper.

2.5.2 procedure

2.5.2.1 For pea: Reduce by quartering to ca 1 kg. Grind with a grinder(2.5.1.1)to let all pass through a 20-mesh sieve. Mix thoroughly and divide into two equal portions, place in clean sample containers as the test sample, seal and label.

2.5.2.2 For peanuts: Reduce by quartering to ca 500 g, pulverize with a pulverizer(2.5.1.2)to let all pass through a 2.0 mm round-hole seive. Mix thoroughly and divide into two equal portions as the test samples. Place in clean containers, seal and label.

2.6 Storage of test sample

The test samples should be stored below −5℃ and kept away from light.

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

3 Method of determination

3.1 Principle

The dioxathion residues in peas or peanuts are extracted with acetonitrile, the extract is treated with methylene chloride for liquid-liquid partitioning. The extract is then cleaned up by charcoal column. Determination is made by means of a gas chromatograph equipped with flame photometric detector, using external standard method.

3.2 Reagents and materials

Unless otherwise specified, the reagents should be analytically pure. "Water"is distilled water or corresponding de-ionized water.

3.2.1 Methylene chloride: Redistill, collect the distillate of 39℃—41℃.

3.2.2 Ethyl acetate: Redistill.

3.2.3 Acetonitrile: Redistill.

3.2.4 Acetonitrile solution: Acetonitrile-water(2+1).

3.2.5 Activated: Charcoal: Slurry 100 g of activated charcoal powder with 250 mL concentrated HCL in a baker(1 L)and stir with magnetic bar while boiling for 1 h, covering with watch glass while heating. Add 250 mL of water, stir, and boil an additional 30 min. Filte and collect the charcoal in a Buchner funnel and wash with water until washing are neutral. Dry at 130℃ in an oven and store in a closed container after cooled.

3.2.6 Magnesium oxide: Slurry 500 g of MgO with water. After heating for 30 min on a boiling bath, filter with Buchner funnel by suction. Then dry at 105℃—130℃ in an oven over night and store in a closed container.

3.2.7 Celite 545.

3.2.8 Adsorbent mixture: Mix in following weight proportions, 1 part of acid-treated activated charcoal, 2 parts of magnesium oxide, and 4 parts of acid-treated Celite 545. keep sealed.

3.2.9 Dioxathion standards: Purity≥95%.

3.2.10 Dioxathion standards solution: Accurately weigh an adequate amount of standard dioxathion, dissolve in a small volume of ethyl acetate. Dilute with ethyl acetate to form a standard stock solution with concentration of 100 mg/L (stored below 4℃). Then dilute the stock solution with ethyl acetate to the re-

quired concentration as the standard working solution.

3.2.11 Eluting solution: Acetonitrile-benzene(1+1).

3.3 Apparatus and equipment

3.3.1 Gas chromatograph, equipped with flame photometric ditector and phosphorous filter(526 nm).

3.3.2 Blender.

3.3.3 Rotary evaporator.

3.3.4 Activated charcoal column: 220 mm×18 mm(id)column, with a side-arm connecting to a vacuum filtering flask. Place 1 g of Celite 545 in column, tamp, add 6 g of adsorbent mixture, and tamp again. Finally add 2 cm glass wool plug on the top of adsorbent.

3.4 Procedure

3.4.1 Extraction

Weigh ca 10 g(accurate to 0.1 g)of the test sample into a 250 mL blender cup, add 30 mLof aqueous acetonitrile solution, then blend 3 min at high speed. Filter with suction through funnel. Transfer the filtrate to a 250 mL separatory funnel, wash the blender cup and residue with 20 mLof aqueous acetonitrile solution, combine the filtrate liquid into the separatory funnel. Add 50 mL of methylene chloride into the separatory funnel, shake for 1 min, and set aside to let separate clearly. Drain the methylene chloride layer into a 100-mL volumetric flask, wash the water phase with 20, 10 mL of methylene chloride. Combine the methylene chloride into the same volumetric flask, make up to volume with methylene chloride.

3.4.2 Cleanup

Pre-wash the activated charcoal column with 100 mL of eluting solution. Discard the washings. Accurately pipet 20 mL of methylene chloride solution from the volumetric flask onto the charcoal column, adjust the flow rate to 5 mL/min. Elute the column with 120 mL of eluting solution. Rinse the column tip and vacuum adapter with several mL of ethyl acetate. Collect all eluates in a evaporating bottle, evaporate it nearly to dryness in a water bath at 70℃ with the ratary evaporater. And then exactly dilute the concentrated solution to 1.0 mL with ethyl acetate for GC determination.

3.4.3 Determination

3.4.3.1 GC operating condition

a) GC column: Packed column. 1.85 m×2 mm(id), 10%(*m*/*m*) OV-101 on WHP(100—120 mesh);

b) Column temperature: 175℃;

c) Injection port temperature: 220℃;

d) Detector temperature: 220℃;

e) Carrier gas: Nitrogen, purity≥99.99%; 30 mL/min;

f) Hydrogen: 45 mL/min;

g) Air: 50 mL/min;

h) Oxygen: 10 mL/min;

i) Injection volume: 2 μL.

3.4.3.2 GC determination

According to the approximate concentration of dioxathion in the sample solution, select a standard working solution with similar peak area to that of the sample solution. The responses values of dioxathion in the standard working solution and sample solution should be within the linear range of the instrumental detection. The standard working solution should be randomly injected in-between the injections of the sample solution of equal volume. Under the above chromatographic condition, the retention time of dioxathion is about 9 min. For gas chromatogram of the standard, see figure 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 with omission of sample addition.

3.5 Calculation and expression of the result

Calculate the content of dioxathion residues in the test sample by GC data processor or according to formula(2):

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

where

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

A—the peak area of dioxathion in the sample solution, mm^2;

A_s—the peak area of dioxathion in the standard working solution, mm^2;

c—the concentration of dioxathion in the standard working solution, $\mu g/mL$;

V—the final volume of the 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 0.05 mg/kg.

4.2 Recovery

4.2.1 Recovery experimental data of dioxathion in peas

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

0.05 mg/kg, the recovery 81.0%;

0.10 mg/kg, the recovery 92.6%;

0.25 mg/kg, the recovery 97.1%.

4.2.2 Recovery experimental data of dioxathion in peanuts

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

0.05 mg/kg, the recovery 76.6%;

0.10 mg/kg, the recovery 94.4%;

0.25 mg/kg, the recovery 102.7%.

Annex A

(informative)

Chromatogram of the standard



Fig. A1 GC chromatogram of dioxathion stardard