SN

中华人民共和国进出口商品检验行业标准

SN 0201—93

出口水产品中多氯联苯残留量检验方法

Method for determination of polychlorinated biphenyl residues in aquatic products for export

1993-06-04 发布　　1993-08-01 实施

中华人民共和国国家进出口商品检验局　发布

(京)新登字 023 号

中华人民共和国进出口商品检验行业标准

出口水产品中多氯联苯残留量检验方法

SN 0201—93

Method for determination of polychlorinated biphenyl residues in aquatic products for export

1 主题内容与适用范围

本标准规定了使用大口径(0.53 mm)毛细管柱和配有电子俘获检测器的气相色谱仪检测特定的六个单一的氯联苯作为水产品中多氯联苯残留量的检验方法及抽样、制样方法。

本标准适用于出口鱼、虾、贝中多氯联苯残留量的检验，同时也适用于猪肉中多氯联苯残留量的检验。

2 抽样和制样

2.1 检验批

以不超过 10 000 箱为一检验批，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(箱)		最低抽样数(箱)
冷冻品	活品、盐藏品	
150 及以下	90 及以下	3
151～3 200	91～500	8
3 201～10 000	501～1 200	13
	1 201～10 000	20

2.3 抽样方法

按 2.2 规定的抽样箱数随机抽取，逐件开启。每箱至少取 500 g 作为原始样品，原始样品总量不得少于 2 kg。加封后，标明标记，及时送实验室。

2.4 试样制备

将抽取的样品去鳞、去骨后，将所有可食部分充分搅碎和混匀，用四分法缩分出 1 kg，然后一分为二份，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℃冷冻保存。

注：① 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。

② 猪肉样品的抽取和制备按 SN 0195《出口肉及肉制品中 2,4-滴残留量检验方法》中规定的抽样、制样方法进行。

3 测定方法

3.1 方法提要

样品同无水硫酸钠研磨干燥后，于索氏提取器内，用石油醚加丙酮(8＋2)提取。提取液经碱皂化、无

中华人民共和国国家进出口商品检验局 1993-06-04 批准　　1993-08-01 实施

水硫酸钠溶液洗涤、浓硫酸净化后以气相色谱仪电子俘获检测器进行检测，灭蚁灵作内标进行定量。

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 石油醚：沸程 30～60℃。经氧化铝柱净化过滤后用全玻璃系统蒸馏器重蒸馏，收集 60℃以前馏分。

3.2.2 丙酮：重蒸馏。

3.2.3 无水硫酸钠：650℃灼烧 4 h，冷却后，储于密闭容器中。

3.2.4 硫酸钠溶液：20 g/L。称 20 g 无水硫酸钠(见 3.2.3)溶于 1 000 mL 水中。

3.2.5 氢氧化钾乙醇溶液：1 mol/L。称氢氧化钾 56 g 溶于 1 000 mL 乙醇中。

3.2.6 乙醇。

3.2.7 浓硫酸：优级纯。

3.2.8 氧化铝：层析用，中性，650℃灼烧 4 h，冷却至室温储于密闭容器中。

3.2.9 氯联苯标准溶液和灭蚁灵(内标物)标准溶液。

3.2.9.1 灭蚁灵标准品：纯度大于 99%。

3.2.9.2 氯联苯标准储备溶液：准确称取多氯联苯 K-400(相当于 Aroclor 1248)、K-500(相当于 Aroclor 1254)各 0.020 0±0.000 1 g 于 200 mL 容量瓶中，加少许石油醚溶解后，用石油醚稀释到刻度(溶液浓度为 200 μg/mL)。

3.2.9.3 灭蚁灵标准溶液：准确称取灭蚁灵 0.010 0±0.000 1 g 于 100 mL 容量瓶中，加少量石油醚溶解后，用石油醚稀释到刻度(溶液浓度为 100 μg/mL)。

3.2.9.4 氯联苯标准工作溶液：取氯联苯标准储备液 2 mL、1 mL、0.5 mL、250 μL 分别置于四个 100 mL 容量瓶中，各加入 0.05 μg 灭蚁灵(即 50 μL 灭蚁灵标准溶液)后用石油醚稀释到刻度，摇匀。其浓度如下：

编号	相当于多氯联苯(mg/L)	灭蚁灵(mg/L)
1	4	0.05
2	2	0.05
3	1	0.05
4	0.5	0.05

注：配制更低浓度氯联苯标准液时，需相应降低内标物浓度。

测定前，取上述与样液浓度相近的标准溶液，按 3.4.3.1 条仪器操作条件，将仪器调好，待仪器稳定后，进样 1 μL，按归一化法(扣去灭蚁灵峰高)计算特定的六个单一的氯联苯的百分含量，再分别计算六个单一氯联苯的浓度(mg/L)。六个特定的单一氯联苯峰顺序参见多氯联苯色谱图中 28 号，52 号，101 号，153 号，138 号，180 号峰。

3.2.9.5 乙醇加石油醚溶液(1+1)。

3.3 仪器和设备

3.3.1 气相色谱仪并配有电子俘获检测器。

3.3.2 绞肉机。

3.3.3 全玻璃系统重蒸馏装置。

3.3.4 索氏提取器：250 mL。

3.3.5 分液漏斗：500 mL。

3.3.6 无水硫酸钠柱：在斗径 20 mm，筒身 70 mm，柄外径 8 mm 的筒形漏斗内，下部放玻璃棉，上部放约 15 g 无水硫酸钠。

3.3.7 旋转蒸发器。

3.3.8 高速捣碎器。

3.3.9 微量注射器:1 μL、10 μL、50 μL、100 μL、250 μL。

3.3.10 氧化铝柱:玻璃制,高 30 cm、内径 5 cm 色谱柱,出口有活塞开关,柱底部有烧结玻璃片,内装氧化铝(见 3.2.8),装填高度为 25 cm。

3.4 测定步骤

3.4.1 提取

称取试样约 10 g(精确至 0.1 g),于高速捣碎机中,加 40 g 无水硫酸钠捣碎几分钟,将样品制成干松粉末,装于滤纸筒内,然后放入索氏提取器中。在提取器的瓶中加入 50 mL 1 mol/L 氢氧化钾乙醇溶液和石油醚加丙酮(8+2)混合液 130 mL,在水浴上提取 6 h(回流速度每小时 10～12 次)。将提取液移于 500 mL 分液漏斗中,用 20 mL 乙醇加石油醚(1+1)溶液洗涤提取器的瓶,洗液并入上述分液漏斗中。加 100 mL 20 g/L 硫酸钠水溶液,振摇 1 min,静置分层。将水层放入原提取器的瓶中,上层提取液从分液漏斗上口倒入另一干净的分液漏斗中。再将水层倒回原分液漏斗中。然后每次用 30 mL 石油醚再提取水层三次。每次的石油醚提取液合并到第一次的提取液中。加 150 mL 20 g/L 硫酸钠溶液于合并的提取液中,振摇,静置分层。弃去水层。

3.4.2 净化

于提取液中加入浓硫酸(提取液和浓硫酸的比例为 10∶1,以体积计)。轻轻振摇后,静置分层,弃去酸层。再按上述操作重复净化 1～2 次,每次振摇半分钟,净化至酸液呈无色或淡黄色。然后加 20 g/L 硫酸钠溶液 100 mL,振摇,静置分层,弃去水层。再如上重复洗涤一次。将石油醚液通过无水硫酸钠柱,再用石油醚洗涤分液漏斗及无水硫酸钠柱。

收集石油醚液及洗液于旋转蒸发器中浓缩至约 10 mL,定量加入灭蚁灵(内标物)标准溶液,用石油醚稀释至刻度,摇匀,进行气相色谱测定。

3.4.3 测定

3.4.3.1 色谱条件

a. 载气:氮气(纯度≥99.99%),4 mL/min;

b. 辅助气:氮气(纯度≥99.99%),30 mL/min;

c. 毛细管柱:材料:熔融石英,长 25 m,内径 0.53 mm;固定相:BD-5,交联的,膜厚 5.0 μm;

d. 柱温:238℃;

e. 进样口温度:270℃;

f. 检测器温度:300℃;

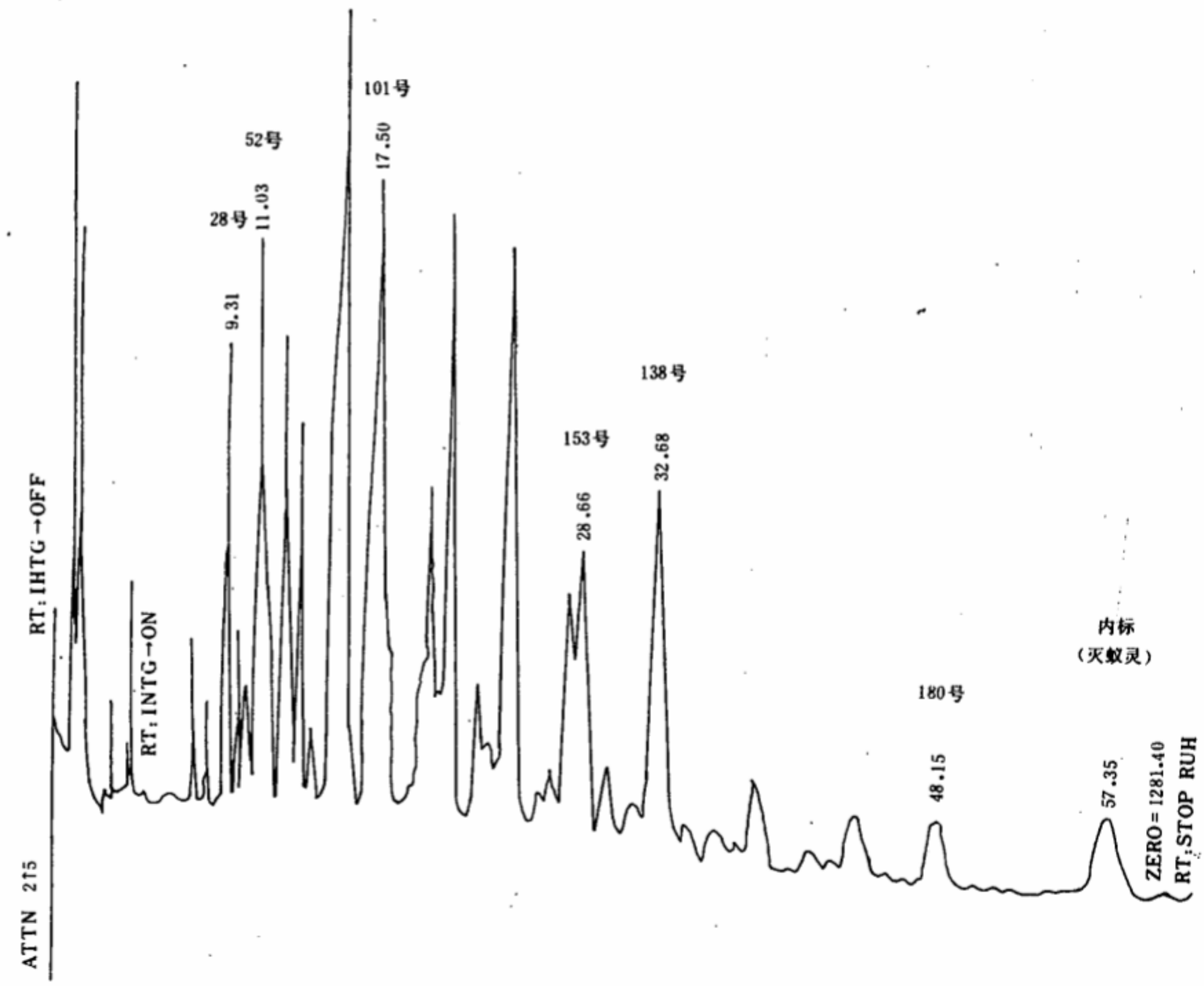
g. 进样方式:柱头进样方式。

3.4.3.2 色谱测定

待上述条件稳定后取与样液浓度相近的氯联苯标准工作溶液 1 μL 进行色谱测定。六个特定的单一氯联苯,灭蚁灵出峰顺序和保留时间如下(参见多氯联苯色谱图):

编号 (IUPAC No.)	结构	保留时间
28(三氯联苯)	2,4-4′	9.31 min
52(四氯联苯)	2,5-2′,5′	11.03 min
101(五氯联苯)	2,4,5-2′,5′	17.50 min
153(六氯联苯)	2,4,5-2′,4′,5′	28.66 min
138(六氯联苯)	2,3,4-2′,4′,5′	32.68 min
180(七氯联苯)	2,3,4,5-2′,4′,5′	48.15 min
内标(灭蚁灵)		57.35 min

取上述净化样液 1～5 μL 按内标法进行色谱测定。实际应用的标准工作液及待测液中各氯联苯组分的响应值均应在检测器的线性范围内。



多氯联苯色谱图

3.4.4 空白试验

除不加试样外，按上述条件及步骤进行空白试验。

3.4.5 结果的计算和表述

应用数据处理系统适当程序计算六个特定的单一氯联苯残留量，或按下列公式分别进行计算。

$$X = \frac{h \cdot h_{is} \cdot c_s \cdot m_i}{h_s \cdot h_i \cdot c_{is} \cdot m}$$

式中：X ——试样中氯联苯残留量，mg/kg；

h ——样液中氯联苯组分峰高，mm；

h_s ——标准工作液中氯联苯组分峰高，mm；

h_{is} ——标准工作液中内标物峰高，mm；

h_i ——样液中内标物峰高，mm；

c_s ——标准工作液中氯联苯浓度，ng/μL；

c_{is} ——标准工作液中内标物浓度，ng/μL；

m_i ——样液中加入内标物的量，μg；

m ——称取样品量，g。

注：计算结果需扣除空白值。

本方法的测定结果分别按六个特定的单一氯联苯实际检测含量(mg/kg)报出。

4 测定低限、回收率

4.1 测定低限

本方法测定低限为 0.005 mg/kg。

4.2 回收率

回收率的实验数据：

28 号三氯联苯(2,4-4′)浓度在 0.023～0.235 mg/kg 范围，回收率为 89.3%～101.3%。

52 号四氯联苯(2,5-2′,5′)浓度在 0.064～0.370 mg/kg 范围，回收率为 85.9%～101.1%。

101 号五氯联苯(2,4,5-2′,5′)浓度在 0.068～0.434 mg/kg 范围，回收率为 86.8%～110.4%。

153 号六氯联苯(2,4,5-2′,4′,5′)浓度在 0.023～0.174 mg/kg 范围，回收率为 100%～117.8%。

138 号六氯联苯(2,3,4-2′,4′,5′)浓度在 0.032～0.215 mg/kg 范围，回收率为 90.6%～108.4%。

180 号七氯联苯(2,3,4,5-2′,4′,5′)浓度在 0.009～0.043 5 mg/kg 范围，回收率为 100%～114.0%。

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山东进出口商品检验局负责起草。

本标准主要起草人刘学悌、刘淑贞、李戈。

主要参考文献：

Quantitative Determination of Specified Chlorobiphenyls in Fish with Capillary Gas Chromatography and its Use for Monitoring and Tolerance Purpose, Inter. J. Environ. Anal. Chem. , Vol. 14, pp 147-157.

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

SN 0201—93

Method for determination of polychlorinated biphenyl residues in aquatic products for export

1 Scope and field of application

This standard specifies the methods of sampling, sample preparation and determination of the six specified individual polychlorinated biphenyls as the polychlorinated biphenyl residues in aquatic products for export by wide-bore capillary column (0.53 mm) gas chromatography (GC) with electron capture detector.

This standard is applicable to the determination of polychlorinated biphenyl residues in aquatic products, such as fish, prawn and shell fish for export, and also in pork for export.

2 Sampling and sample preparation

2.1 Inspection lot

The quantity of an inspection lot should not be more than 10 000 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 packages to be taken
Frozen	Live, salted	
≤150	≤90	3
151～3 200	91～500	8
3 201～1 000	501～1 200	13
	1 201～10 000	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 500 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 scaled and deboned. The edible portion is blended and reduced by quartering to 1 kg. Then divided into two equal portions, each portion is placed in a clean container as test sample, which is sealed and labeled.

2.5 Storage of sample

Approved by the State Administration of Import and Export Commodity Inspection of the People's Republic of China on Jun. 4, 1993

Implemented from Aug. 1, 1993

The test samples should be stored at −18℃.

Note 1: 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.

Note 2: The sampling and sample preparation of the pork should be carried out as that indicated in "Method for determination of 2,4-D residues in meat and meat products for export", SN 0195.

3 Method of determination

3.1 Principle

After grinding and drying by blending with anhydrous sodium sulphate, transfer the sample into a Soxhlet extractor, and extract the chlorobiphenyls with petroleum ether-acetone(8+2). The extract is saponified with alkali and washed with sodium sulphate solution and then cleaned up with concentrated sulphuric acid. Determine the residues by GC with electron capture detector using Mirex as internal standard.

3.2 Reagents and materials

Unless otherwise specified, all reagents shall be analytically pure; "water" means distilled water.

3.2.1 Petroleum ether: Distillation range 30—60℃, after clean up by filtering through alumina column, redistill the reagent in the all-glass apparatus, and collect the fraction below 60℃.

3.2.2 Acetone: Redistilled.

3.2.3 Anhydrous sodium sulphate: Ignite at 650℃ for 4 h, and keep in a tightly closed container after cooling.

3.2.4 Sodium sulphate solution: 20 g/L. Weigh 20 g of anhydrous sodium sulphate(3.2.3)and dissolve in 1 000 mL of water.

3.2.5 Potassium hydroxide alcoholic solution: 1 mol/L. Weigh 56 g of potassium hydroxide and dissolve in 1 000 mL of alcohol.

3.2.6 Alcohol.

3.2.7 Concentrated sulphuric acid: G.R..

3.2.8 Alumina: For chromatography, neutral, ignite at 650℃ for 4 h, and keep in a tightly closed container after cooling to the room temperature.

3.2.9 Chlorinated biphenyls standard solution and Mirex(internal standard)standard solution.

3.2.9.1 Mirex standard: Purity>99%.

3.2.9.2 Chlorinated biphenyls standard stock solution: Accurately weigh 0.020 0±0.000 1 g each of polychlorinated biphenyl K-400 (equivalent to Aroclor 1248)and K-500(equivalent to Aroclor 1254), place them in 200 mL volumetric flask, add a small amount of petroleum ether to dissolve them and dilute to mark(concentration being 200 μg/mL).

3.2.9.3 Mirex standard solution: Accurately weigh 0.010 0±0.000 1 g of Mirex, transfer into a 100 mL volumetric flask, add a small amount of petroleum ether to dissolve them and dilute to mark(concentration being 100 μg/mL).

3.2.9.4 Chlorinated biphenyls standard working solution: Transfer 2 mL, 1 mL, 0.5 mL, and 250 μL of chlorinated biphenyls standard solution into four 100 mL volumetric flasks respectively. Add 0.05 μg of Mirex(i.e. 50 μL of Mirex standard solution)to each flask, and dilute to mark with petroleum ether. Their concentrations are as follows.

No.	Equivalent to polychlorinated biphenyl(mg/L)	Mirex (mg/L)
1	4	0.05
2	2	0.05
3	1	0.05
4	0.5	0.05

Note: When prepare the chlorobiphenyl solution of lower concentration, the concentration of the internal standard should also be reduced accordingly.

Before determination, adjust GC properly according to the conditions stated in 3.4.3.1. When it becomes stable, inject 1 μL of the above standard solution of suitable concentration to GC. Calculate the six specified contents of individual chlorobiphenyls (as peak height by the normalization method with the peak height of Mirex deducted therefrom). Then calculate the concentrations of them separately (in mg/L). The peaks in sequence are No. 28, No. 52, No. 101, No. 153, No. 138 and No. 180 (See "Chromatogram of polychlorinated biphenyls").

3.2.9.5 Alcohol-petroleum ether (1+1).

3.3 Apparatus and equipment

3.3.1 Gas chromatograph (equipped with ECD).

3.3.2 Meat chopper.

3.3.3 All-glass distillation apparatus.

3.3.4 Soxhlet extractor: 250 mL.

3.3.5 Separatory funnel: 500 mL.

3.3.6 Anhydrous sodium sulphate column: In a funnel tube of 20 mm (id) × 70 mm with a neck of 8 mm (od), fill with glass wool at the lower part and add ca 15 g of anhydrous sodium sulphate above it.

3.3.7 Rotary vacuum evaporator.

3.3.8 High speed blender.

3.3.9 Micro-syringe: 1 μL, 10 μL, 50 μL, 100 μL, 250 μL.

3.3.10 Alumina column: Glass chromatographic column 5 cm (id) × 30 cm with coarse fritted plate and with a stopcock at the outlet. The column is filled to a height of 25 cm with alumina (3.2.8).

3.4 Procedure

3.4.1 Extraction

Weigh ca 10 g of the test sample (accurate to 0.1 g) and transfer into a high speed blender. Add 40 g of anhydrous sodium sulphate and blend for several minutes and turn the sample into dry powder, then fill it in a filter-paper tube and place in a Soxhlet extractor. Add 50 mL of 1 mol/L KOH-Alcohol solution and 130 mL of petroleum ether-acetone (8+2) solution into the bottle of the extractor and extract for 6 h on a water bath (the reflux speed is 10—12 times per hour). The extract is transferred to a 500 mL separatory funnel. Rinse the bottle with 20 mL of alcohol-petroleum ether (1+1) solution, combine the washings in the same funnel. Then add 100 mL of 20 g/L sodium sulphate solution into the separatory funnel, shake for 1 min, and let stand to separate.

Drain the water layer into original bottle. The upper layer extract is poured from the upper mouth of the separatory funnel into another clean separatory funnel. Pour the water layer into original separatory funnel. Then reextract the water layer three times with 30 mL of petroleum ether each. Combine all the petroleum ether extracts with the first extract. Add 150 mL of 20 g/L sodium sulphate solution to the combined extract, shake and let stand to separate. Discard the water layer.

3.4.2 Clean up

Add concentrated sulphuric acid to the extract(the ratio of the extract and the concentrated sulphuric acid is 10 : 1 by volume)and shake gently. Let stand to separate. Discard the sulphuric acid layer. Repeat the procedure (usually 1 or 2 times)by shaking for half a minute each time,until the sulphuric acid layer is colourless of light yellow. Then add 100 mL of 20 g/L sodium sulphate solution and shake,let stand to separate. Discard the water layer. Repeat the above procedure once more,and let the petroleum ether layer pass through the anhydrous sodium sulphate column. Rinse the separatory funnel and the column with petroleum ether. Collect the clean up solution in rotary vacuum evaporator and concentrate the solution to ca 10 mL. Add the standard solution of Mirex quantitatively as internal standard,dilute it to mark with petroleum ether and mix well for GC determination.

3.4.3 Determination

3.4.3.1 GC operating conditions

a. Carrier gas:Nitrogen(purity≥99.99%),4 mL/min;

b. Make-up gas:Nitrogen(purity≥99.99%),30 mL/min;

c. Capillary column:

Material:Fused silica;0.53 mm(id),25 m(length);

Stationary phase:BD-5,cross-linked,film thickness 5.0 μm;

d. Column temperature:238℃;

e. Injection port temperature:270℃;

f. Detector temperature:300℃;

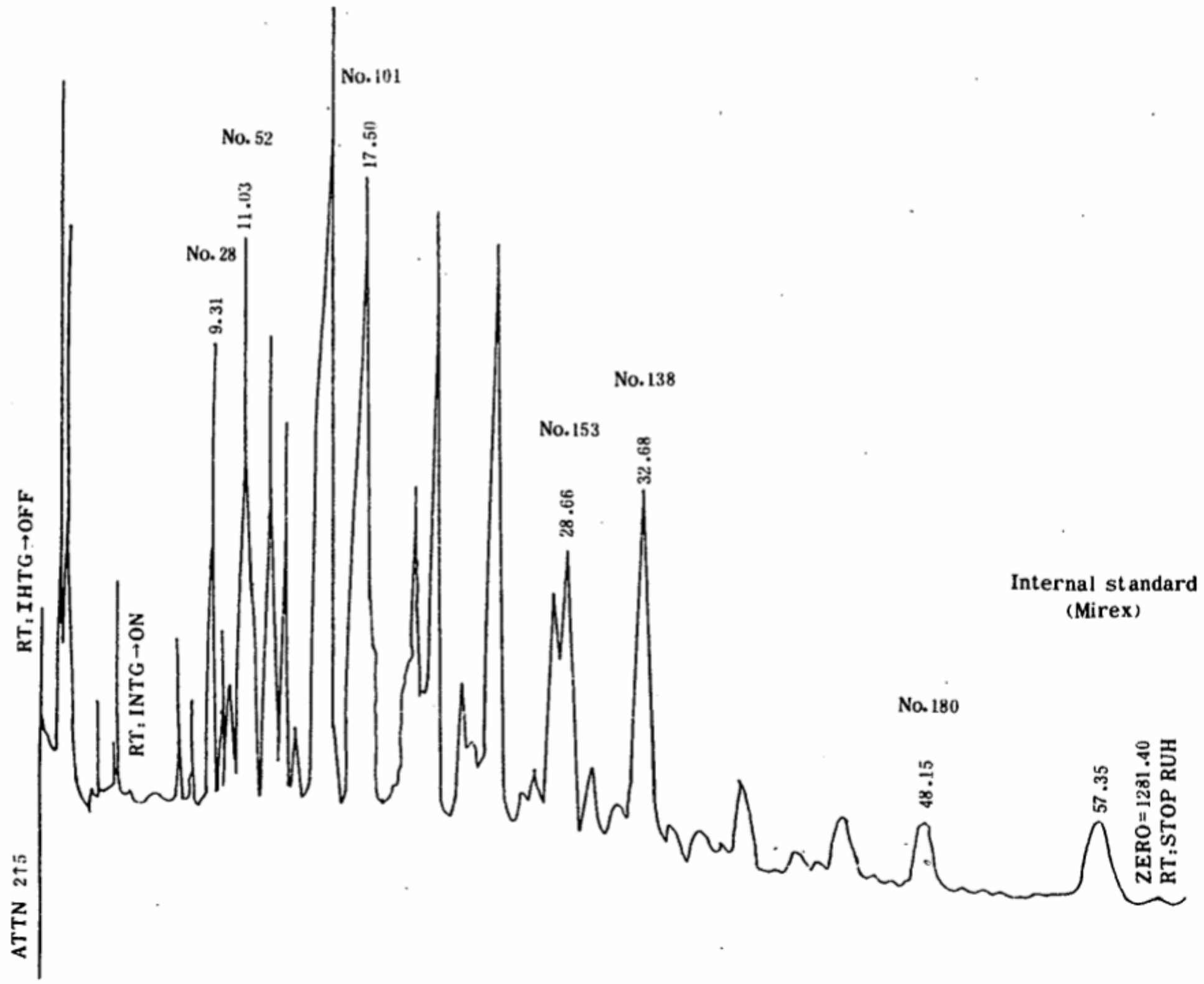
g. Inspection mode:On-column method.

3.4.3.2 GC operation

When the above condition becomes stable,take 1 μL of the chlorinated biphenyls standard working solution which has the similar concentration with the test solution. The peaks in sequence and the retention times of the six specified individual chlorobiphenyls and Mirex are as follows(See "Chromatogram of polychlorinated biphenyls"):

IUPAC No.	Constitution	Retention time
28(Trichlorobiphenyl)	2,4-4′	9.31 min
52(Tetrachlorobiphenyl)	2,5-2′,5′	11.03 min
101(Pentachlorobiphenyl)	2,4,5-2′,5′	17.50 min
153(Hexachlorobiphenyl)	2,4,5-2′,4′,5′	28.66 min
138(Hexachlorobiphenyl)	2,3,4-2′,4′,5′	32.68 min
180(Heptachlorobiphenyl)	2,3,4,5-2′,4′,5′	48.15 min
Internal standard(Mirex)		57.35 min

Inject suitable volume of the cleaned up sample solution(e.g. 1—5 μL) for GC determination,using internal standard method. The response of the chlorinated biphenyls in the standard working solutions and the sample solutions must be within the linear range of the detector.



Chromatogram of polychlorinated biphenyls

3.4.4 Blank test

To be carried out according to the above mentioned condition and procedure without the test sample.

3.4.5 Calculation and expression of results

Calculate the contents of the six specified individual chlorobiphenyls residues by using the appropriate program of a data system or using the following equation:

$$X = \frac{h \cdot h_{is} \cdot c_s \cdot m_i}{h_s \cdot h_i \cdot c_{is} \cdot m}$$

where:

X—chlorobiphenyl content in test sample, mg/kg;

h —peak height of chlorobiphenyl in sample solution, mm;

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

h_{is} —peak height of internal standard in standard working solution, mm;

h_i —peak height of internal standard in sample solution, mm;

c_s —concentration of chlorobiphenyl in standard working solution, ng/μL;

c_{is} —concentration of internal standard in standard working solution, ng/μL;

m_i —mass of internal standard added in sample solution, μg;

m —mass of weighed sample, g.

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

The results are expressed in mg/kg, and reported separately according to the contents of six specified individual chlorobiphenyls.

4 Limit of determination and recovery

4.1 Limit of determination

The limit of determination is 0.005 mg/kg.

4.2 Recovery

According to the experimental data, the recoveries are as follows:

No. 28 Trichlorobiphenyl (2, 4-4′) at the concentration of 0.028—0.235 mg/kg, the recovery is 89.3%—101.3%.

No. 52 Tetrachlorobiphenyl (2, 5-2′, 5′) at the concentration of 0.064—0.370 mg/kg, the recovery is 85.9%—101.1%.

No. 101 Pentachlorobiphenyl (2, 4, 5-2′, 5′) at the concentration of 0.068—0.434 mg/kg, the recovery is 86.8%—110.4%.

No. 153 Hexachlorobiphenyl (2, 4, 5-2′, 4′, 5′) at the concentration of 0.023—0.174 mg/kg, the recovery is 100%～117.8%.

No. 138 Hexachlorobiphenyl (2, 3, 4-2′, 4′, 5′) at the concentration of 0.032—0.215 mg/kg, the recovery is 90.6%—108.4%.

No. 180 Heptachlorobiphenyl (2, 3, 4, 5-2′, 4′, 5′) at the concentration of 0.009—0.043 mg/kg, the recovery is 100%—114.0%.

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 Shandong Import and Export Commodity Inspection Bureau of the People's Republic of China.

This standard was mainly drafted by Liu Xuedi, Liu Shuzheng and Li Ge.

Reference:

Quantitative Determination of Specified Chlorobiphenyls in Fish with Capillary Gas Chromatography and its Use for Monitoring and Tolerance Purpose, Inter. J. Environ. Anal. Chem., Vol. 14, pp 147-157.

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

SN 0201—93

中国标准出版社出版　中国标准出版社北京印刷厂印刷

1994 年 1 月第一版　1994 年 1 月第一次印刷　书号:155066・2-9169