



中华人民共和国出入境检验检疫行业标准

SN/T 0192—2017
代替 SN 0192—1993

出口水果中溴螨酯残留量的检测方法

Determination of bromopropylate residue in fruits for export

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中 华 人 民 共 和 国
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前 言

本标准按照 GB/T 1.1—2009 给出的规则起草。

本标准代替 SN 0192—1993《出口水果中溴螨酯残留量检验方法》。

与 SN 0192—1993 相比,除编辑性修改外,主要技术修改如下:

- 增加了样品基质;
- 修改了样品提取步骤;
- 修改了样品净化步骤;
- 将样品测定填充色谱柱改成毛细管色谱柱;
- 增加了气相色谱质谱确证法;
- 略去了抽样步骤。

请注意本文件的某些内容可能涉及专利。本文件的发布机构不承担识别这些专利的责任。

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:中华人民共和国上海出入境检验检疫局。

本标准主要起草人:时逸吟、杨惠琴、程甲、樊彦莉、周瑶、伊雄海、邓晓军、朱坚。

本标准所代替标准的历次版本发布情况为:

- SN 0192—1993。

出口水果中溴螨酯残留量的检测方法

1 范围

本标准规定了水果中溴螨酯残留量的检测和确证方法。

本标准适用于苹果、樱桃、柠檬、草莓、菠萝、葡萄、西瓜中溴螨酯残留量的检测和确证。

2 规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件,仅注日期的版本适用于本文件。凡是不注日期的引用文件,其最新版本(包括所有的修改单)适用于本文件。

GB/T 6682 分析实验室用水规格和试验方法

3 方法提要

试样中残留的溴螨酯用乙腈匀浆提取,盐析离心后,上清液经固相萃取柱净化,用带有电子捕获检测器气相色谱测定,外标法定量,如有必要用气相色谱-质谱联用仪进行确证。

4 试剂材料

除非另有说明,所用试剂均为分析纯。

4.1 乙腈:色谱纯。

4.2 甲苯:色谱纯。

4.3 正己烷:色谱纯。

4.4 氯化钠。

4.5 无水硫酸钠:用前在 650 °C 灼烧 4 h,冷却后贮于干燥器中。

4.6 乙腈-甲苯溶液(3+1,体积比):取 200 mL 甲苯,加入 600 mL 乙腈,摇匀备用。

4.7 溴螨酯(bromopropylate, $C_{17}H_{16}Br_2O_3$, CAS 号 8181-80-1)标准品:纯度大于或等于 98%。

4.8 溴螨酯标准储备溶液:准确称取 0.01 g(精确至 0.000 1 g)溴螨酯标准品于 10 mL 的容量瓶中,用正己烷溶解配成浓度为 1.0 mg/mL 的标准储备溶液,于 4 °C 保存,保存期限为 3 个月。

4.9 溴螨酯标准中间溶液:准确移取 1 mL 标准储备液至 10 mL 容量瓶中,用正己烷稀释至刻度配成浓度为 100 μ g/mL 的标准溶液,现配现用。

4.10 基质标准工作溶液:将一定体积的溴螨酯标准溶液分别加到适量样品空白基质提取液中,混匀,配成基质标准工作溶液。基质标准工作溶液应现用现配。

4.11 石墨化炭-氨基固相萃取柱:石墨化炭和氨基填料各 0.5 g,6 mL,或相当者。

4.12 无水硫酸钠柱:7.5 cm \times 1.5 cm(i.d.)玻璃柱,内装 5 cm 高无水硫酸钠。

4.13 有机相滤膜:0.45 μ m。

5 仪器与设备

5.1 气相色谱仪:配有电子捕获(ECD)检测器。

5.2 气相色谱-质谱联用仪:配有电子轰击源(EI)。

5.3 天平:感量 0.000 1 g、0.01 g 各一台。

5.4 食品粉碎机。

5.5 均质器。

5.6 离心机:最大转速为 4 000 r/min。

5.7 旋转蒸发器。

5.8 固相萃取装置。

5.9 氮吹仪。

5.10 涡旋混匀器。

6 试样的制备与保存

6.1 试样制备

取有代表性样品约 500 g,将其可食部分切碎后,用组织捣碎机将样品加工成浆状,装入洁净容器作为试样,密封并标明标记。

6.2 试样保存

试样于-18℃状态下保存。在制样的操作过程中,应防止样品受到污染或发生残留物含量的变化。

7 分析步骤

7.1 提取

称取试样 10 g(精确至 0.01 g)于 50 mL 塑料离心管中,加入 10 mL 乙腈,高速均质 1 min 进行提取,4 000 r/min 离心 5 min 取上清液。残渣再加入 10 mL 乙腈重复提取一次。合并提取液,加入 2 g 氯化钠,振摇 10 min,4 000 r/min 离心 5 min 后,上清液过无水硫酸钠柱,后将提取液收集于圆底烧瓶中,在 40℃以下旋转蒸发至近干。残留物中加入 2 mL 乙腈-甲苯溶液溶解待净化。

7.2 净化

用 10 mL 乙腈-甲苯混合溶液活化石墨化炭-氨基固相萃取柱,弃去淋洗液。将上述的待净化液(7.1)转移至小柱中过柱,用 6 mL 乙腈-甲苯溶液分三次洗涤样液瓶,并将洗涤液移入柱中,收集全部流出液(在整个活化、上样和洗脱过程中应避免 SPE 柱干涸),过程中控制流速在 1 mL/min。收集洗脱液于 15 mL 刻度试管中并于 40℃下氮吹浓缩至近干,准确加入 1.0 mL 正己烷溶液后供气相色谱或气相色谱-质谱仪测定。

7.3 测定

7.3.1 气相色谱参考条件

色谱测定条件如下:

- 色谱柱:DB-5 石英毛细管柱,长 30 m,内径 0.32 mm,膜厚 0.25 μm,或相当者;
- 进样口温度:280℃;
- 进样量:1.0 μL;不分流进样,0.6 min 开阀;
- 柱温:初始温度 100℃,保持 1 min,以 10℃/min 升温至 230℃,保持 5 min,再以 30℃/min

升温至 280 °C,保持 10 min;

- e) 载气:氮气,纯度大于或等于 99.999%;恒流模式;流速:1.0 mL/min;
- f) 检测器温度:300 °C;
- g) 尾吹气:氮气,60 mL/min。

7.3.2 气相色谱-质谱参考条件

7.3.2.1 色谱测定条件如下:

- a) 色谱柱:DB-5MS 石英毛细管柱,长 30 m,内径 0.25 mm,膜厚 0.25 μ m,或相当者;
- b) 进样口温度:280 °C;
- c) 进样量:1.0 μ L,不分流进样,0.6 min 开阀;
- d) 柱温:初始温度 100 °C,保持 1 min,以 10 °C/min 升温至 230 °C,保持 5 min,再以 30 °C/min 升温至 280 °C,保持 10 min;
- e) 载气:氮气,纯度大于或等于 99.999%;恒流模式;流速:1.0 mL/min。

7.3.2.2 质谱测定条件如下:

- a) GC-MS 接口温度:280 °C;
- b) 电离方式:EI;
- c) 离子源温度:150 °C;
- d) 溶剂延迟:10 min;
- e) 测定方式:选择离子监测模式(SIM),详见表 1。

表 1 溴螨酯的监测离子及其丰度比

名 称	监测离子(m/z)amu	监测离子丰度比/%
溴螨酯	341 *、339、343、183、157	100 : 50 : 50 : 60 : 30
注:* 表示定量离子。		

7.3.3 气相色谱测定

7.3.3.1 标准曲线:采用标准工作液,分别配制成 0.00 μ g/mL、0.01 μ g/mL、0.05 μ g/mL、0.10 μ g/mL、0.20 μ g/mL、0.50 μ g/mL、1.00 μ g/mL 标准溶液,直接经气相色谱测定或配制成 0.00 μ g/mL、0.01 μ g/mL、0.05 μ g/mL、0.10 μ g/mL、0.20 μ g/mL、0.50 μ g/mL、1.00 μ g/mL 标准基质工作溶液供气相色谱-质谱测定,峰面积对浓度作线性回归曲线,计算相关系数。

7.3.3.2 根据样液中被测物含量情况,对标准工作溶液和样液等体积穿插进样测定,以峰面积为纵坐标,标准工作溶液浓度为横坐标绘制标准工作曲线,用标准工作曲线对样品进行定量。标准工作液和样液中待测物的响应值均应在仪器检测的线性范围内。如果含量超过标准曲线范围,应稀释到适合浓度后分析,标准品的色谱图参见附录 A。

7.3.4 气相色谱-质谱确证

根据样液中被测物含量情况,对标准基质工作溶液和样液等体积穿插进样测定,以峰面积为纵坐标,标准基质工作溶液浓度为横坐标绘制标准工作曲线,用标准基质工作曲线对样品进行定量。标准工作液和样液中待测物的响应值均应在仪器检测的线性范围内。如果含量超过标准曲线范围,应稀释到适合浓度后分析。在相同试验条件下,试样中待测物质的保留时间与标准工作溶液中对应的保留时间偏差在 $\pm 2.5\%$ 之内;并且被测样品与标准品的质谱图相似,所选择的全部监测离子均出现且丰度比也

相一致,其允许偏差不超过表 2 规定的范围时,则可确定为样品中存在这种药物残留。溴螨酯的选择离子监测总离子流图 and 全扫描质谱图参见附录 B。

表 2 定性确证时相对离子丰度的最大允许偏差

相对离子丰度	>50%	>20%至 50%	>10%至 20%	≤10%
允许的相对偏差	±10%	±15%	±20%	±50%

7.4 空白试验

除不加试样外,均按上述操作步骤进行。

8 结果计算和表述

用色谱处理软件中的外标法绘制标准曲线,试样中溴螨酯残留量分别按式(1)计算。计算结果需扣除空白值。

$$X = \frac{c \times 1\,000}{m \times 1\,000} \times V$$

.....(1)

式中:

- X ——样品中溴螨酯残留量,单位为毫克每千克(mg/kg);
- c ——从标准曲线中得到溴螨酯标准工作溶液的浓度,单位为微克每毫升(μg/mL);
- V ——样品定容体积,单位为毫升(mL);
- m ——样品称样量,单位为克(g)。

9 测定低限、回收率

9.1 测定低限

气相色谱和气相色谱-质谱法对溴螨酯残留量的测定低限均为:0.01 mg/kg。

9.2 回收率

详见表 3 和表 4。

表 3 水果中溴螨酯的添加回收率(气相色谱法)

添加水平 /(mg/kg)	回收率范围/%						
	苹果	柠檬	菠萝	葡萄	樱桃	草莓	西瓜
0.01	82.1~106	82.1~105	80.2~104	81.2~108	80.6~103	82.5~108	86.1~108
0.50	91.2~101	89.0~100	89.8~101	89.4~106	87.2~99.4	92.4~107	88.8~105
2.00	92.0~105	90.5~101	88.5~98.5	91.0~103	85.5~97.0	87.0~101	92.0~104
5.00	91.4~100	92.2~101	93.0~101	91.6~101	90.8~100	92.4~100	91.8~101

表 4 水果中溴螨酯的添加回收率(气相色谱-质谱法)

添加水平 (mg/kg)	回收率范围/%						
	苹果	柠檬	菠萝	葡萄	樱桃	草莓	西瓜
0.01	83.2~103	82.3~113	80.2~96.3	81.3~103	81.8~104	82.5~113	82.1~106
0.50	89.4~103	93.4~105	93.2~108	90.2~103	94.6~110	93.6~105	95.4~104
2.00	87.0~101	89.5~102	87.0~97.5	89.0~101	92.0~104	86.0~98.0	86.5~96.5
5.00	90.4~100	92.6~102	91.6~101	96.6~106	96.4~105	92.6~107	96.4~107

附录 A
(资料性附录)
溴螨酯标准品的气相色谱图

溴螨酯标准品的气相色谱图见图 A.1。

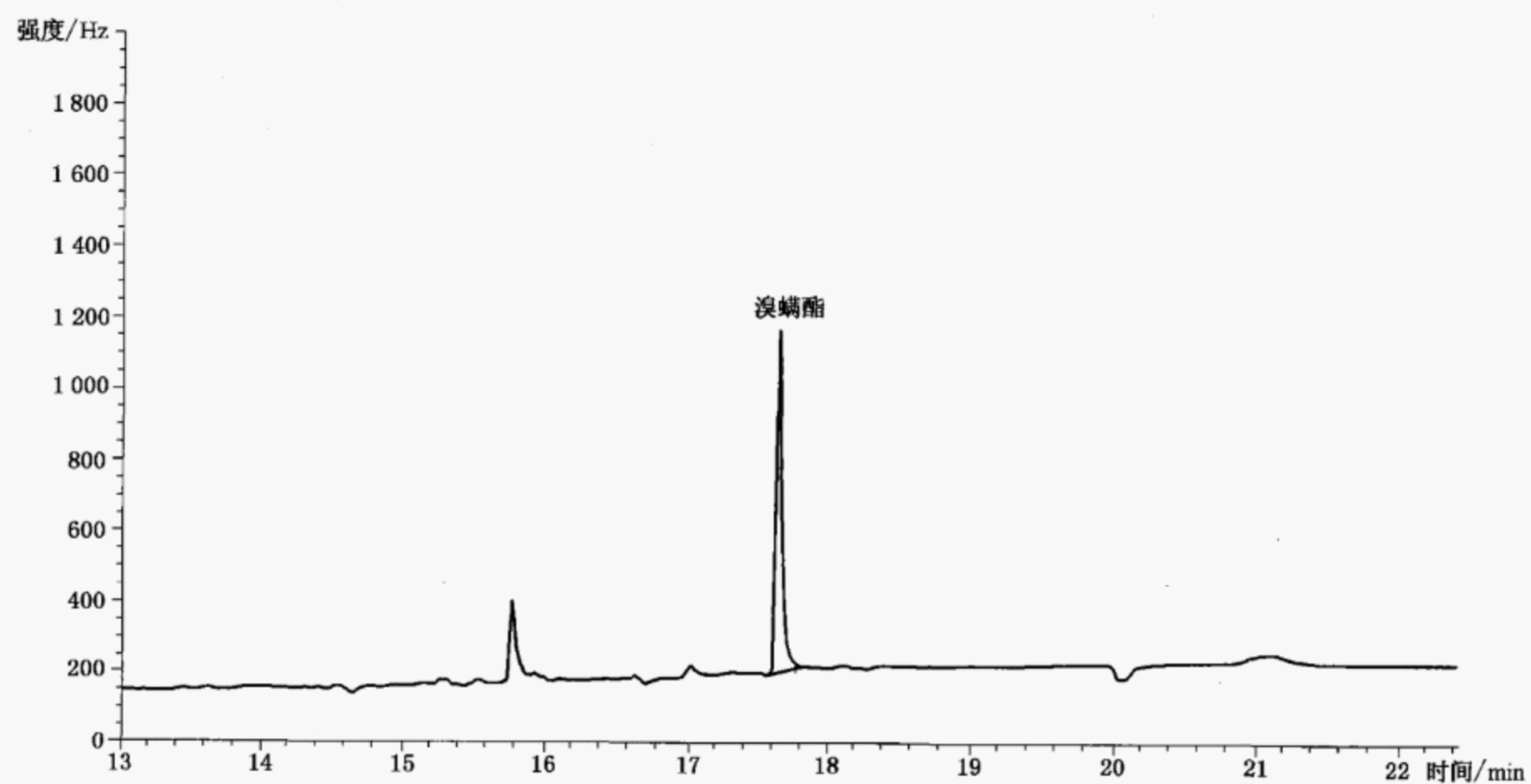


图 A.1 溴螨酯标准品的气相色谱图(0.01 $\mu\text{g/mL}$)

附录 B (资料性附录)

溴螨酯标准品的气相色谱-质谱图

溴螨酯总离子流图 and 全扫描质谱图分别见图 B.1 和图 B.2。

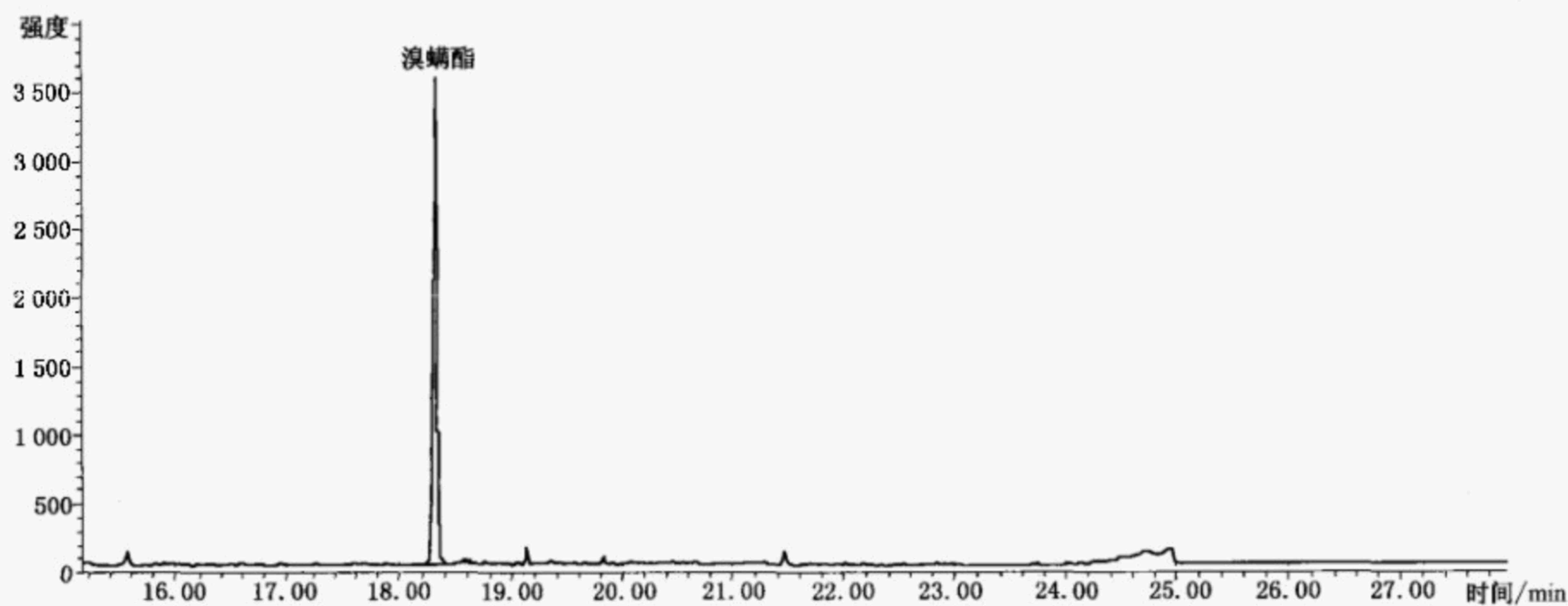


图 B.1 溴螨酯标准品选择离子监测总离子流图(0.01 $\mu\text{g/mL}$)

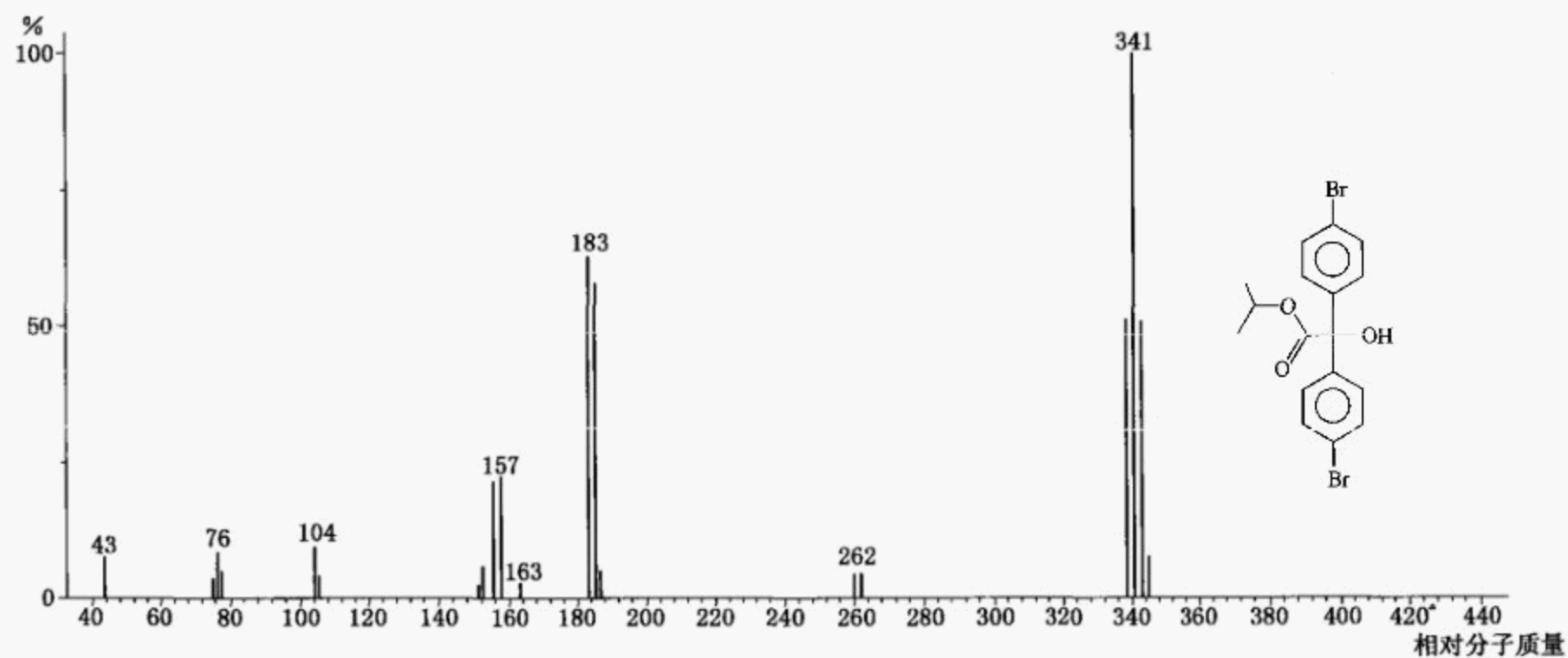


图 B.2 溴螨酯全扫描质谱图

Foreword

This standard is drafted in accordance with the rules given in the GB/T 1.1—2009.

SN 0192—1993 (Method for determination of bromopropylate residues in fruits for export) is replaced with this standard.

Compared with SN 0192—1993, the modifications are below:

- Added more fruit matrix;
- Modified method for the sample pretreatments;
- Modified method for the cleanup procedure;
- Changed the packing column to capillary column;
- Added the ascertainment experiments by GC-MS;
- Skipped the sampling procedure.

Attention is drawn to the possibility that some of the elements of this standard may be the subject of patent rights. The issuing body of this document shall not be held responsible for identifying any or all such patent rights.

This standard was proposed by and is under the charge of the Certification and Accreditation Administration of the People's Republic of China.

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

This standard was mainly drafted by Shi Yiyin, Yang Huiqin, Cheng Jia, Fan Yanli, Zhou Yao, Yi Xionghai, Deng Xiaojun, Zhu Jian.

This standard replaced the following standards:

- SN 0192—1993.

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

Determination of bromopropylate residue in fruits for export

1 Scope

The standard specifies the method of determination bromopropylate residue in fruits.

This standard is applicable to the determination of bromopropylate residue in apple, cherry, lemon, strawberry, pineapple, grape and watermelon.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682, *Water for analytical laboratory use—Specification and test methods*

3 Principle

Bromopropylate residue is extracted with acetonitrile, after salt out and centrifuge, then cleaned up by SPE column. The residual content is determined by GC with ECD detector and quantified by external standard, if necessary GC-MS can be used for confirmation.

4 Reagents and materials

Unless otherwise specified, all the reagents used should be analytical grade.

4.1 Acetonitrile: HPLC grade.

4.2 Toluene: HPLC grade.

4.3 Hexane: HPLC grade.

4.4 Sodium chloride.

4.5 Anhydrous sodium sulfate: Ignite at 650 °C for 4 h, then cool to room temperature and keep in a tightly closed container.

4.6 Acetonitrile-toluene (3+1, V/V): Combine 200 mL acetonitrile and 600 mL toluene.

4.7 Standard substance: Bromopropylate, $C_{17}H_{16}Br_2O_3$, CAS # 8181-80-1, purity $\geq 98\%$.

4.8 Stock standard solution: Accurately weigh 0.01g \pm 0.000 1 g of bromopropylate standard into a 10 mL volumetric flask and dilute to the volume with hexane. Mix well. This solution is stock standard solution of 1.0 mg/mL in hexane, and can be used for three months when stored under 4 °C.

4.9 Intermediate standard solution: Accurately measure 1 mL of stock standard solution into a 10 mL volumetric flask and dilute to the volume with hexane. Mix well. This solution is intermediate standard solution of 100 μ g/mL in hexane, prepare it when necessary.

4.10 Matrix standard solution: Measure certain volume of bromopropylate intermediate standard solution to blank matrix solution. Mix well. This solution is matrix standard solution, prepare it when necessary.

4.11 Carb-NH₂ SPE column: Contain 0.5 g of graphite carbon and ammonia, 6 mL, or equivalent.

4.12 Anhydrous sodium sulfate column: 7.5 cm \times 1.5 cm (i.d.) glass column, with 5 cm high anhydrous sodium sulfate.

4.13 Organic filter: 0.45 μ m.

5 Apparatus and equipment

5.1 GC with ECD.

5.2 GC-MS with EI.

5.3 Balance: Scale 0.000 1 g and 0.01 g.

5.4 Food grinder.

5.5 Homogeneous device.

5.6 Low speed centrifuge: 4 000 r/min.

5.7 Rotary evaporator.

5.8 SPE set.

5.9 Nitrogen evaporator.

5.10 Vortex mixer.

6 Preparation and storage of test sample

6.1 Preparation of test samples

About 500 g representative sample should be taken from all samples. Collect the edible parts and cut minces. Cut by a chopper into pulp. Mix thoroughly and divided into two equal portions. Each portion is placed in clean containers. Seal and label them.

6.2 Storage of test samples

Samples shall be stored below $-18\text{ }^{\circ}\text{C}$. While sampling and preparing sample, please avoid contamination or any factors that may change residue content.

7 Procedure

7.1 Extraction

Weigh 10 g ($\pm 0.01\text{ g}$) of the sample into a 50 mL polypropylene centrifuge tube, add 10.0 mL of acetonitrile and homogeneous extraction for 1 min, centrifuge for 5 min at 4 000 r/min. Collect the supernatant, repeat the extraction procedure once more and combine the organic supernatant. Add 2 g sodium chloride, shake for 10 min, and then centrifuge for 5 min at 4 000 r/min. Transfer the supernatant through the anhydrous sodium sulfate column, and collect the extraction solution in the round bottom flask. Evaporate the solution to dryness through rotatory evaporator, add 2 mL of acetonitrile-toluene solution to dissolve the residue and it is ready for cleanup.

7.2 Clean up

Activate the graphitized carbon-amino column by using 10 mL acetonitrile-toluene solution and discard the effluent solution. Transfer the extract solution (7.1) into the column then use 6 mL acetonitrile-toluene solution to wash the round bottom flask, and collect all the elute. (Avoid dryness during the whole solid phase extraction procedure) Control the flow rate near 1 mL/min during the whole procedure. Collect all the elution into a 15 mL glass tube and evaporate the extract to dryness under $40\text{ }^{\circ}\text{C}$. Add exactly 1.0 mL hexane to dissolve the residue and for GC or GC-MS determination.

7.3 Determination

7.3.1 GC condition

GC condition is as follows:

- a) Colum (analysis): DB-5 30 m \times 0.32 mm(i.d.) \times 0.25 μ m (film thickness), or equivalent;
- b) Injection port temperature: 280 $^{\circ}$ C ;
- c) Injection volume: 1.0 μ L, splitless, open the valve at 0.6 min;
- d) Column temperature: 100 $^{\circ}$ C (1 min)—10 $^{\circ}$ C/min—230 $^{\circ}$ C (5 min)—30 $^{\circ}$ C/min—280 $^{\circ}$ C (10 min);
- e) Carrier gas: Nitrogen, purity \geq 99.999%, constant flow, flow rate: 1.0 mL/min;
- f) Detector temperature: 300 $^{\circ}$ C ;
- g) Make up gas: Nitrogen, 60 mL/min.

7.3.2 GC-MS condition

7.3.2.1 GC condition is as follows:

- a) Colum (analysis): DB-5MS 30 m \times 0.25 mm(i.d.) \times 0.25 μ m (film thickness), or equivalent;
- b) Injection port temperature: 280 $^{\circ}$ C ;
- c) Injection volume: 1.0 μ L, splitless, open the valve at 0.6 min;
- d) Column temperature: 100 $^{\circ}$ C (1 min)—10 $^{\circ}$ C/min—230 $^{\circ}$ C (5 min)—30 $^{\circ}$ C/min—280 $^{\circ}$ C (10 min);
- e) Carrier gas: Helium, purity \geq 99.999%, constant flow, flow rate: 1.0 mL/min;

7.3.2.2 MS condition is as follows:

- a) Interface temperature: 280 $^{\circ}$ C ;
- b) Electron ionization mode: EI;

- c) Ion source temperature: 150 °C ;
- d) Solvent delay: 10 min;
- e) Selected monitoring ion (m/z): Table 1.

Table 1—Monitor ions and relative intensity for bromopropylate

Name of pesticides	Monitor ion(m/z) amu	Relative intensity/%
Bromopropylate	341 * ,339,343,183,157	100 : 50 : 50 : 60 : 30
NOTE: * stands for quantitation ion.		

7.3.3 GC determination

7.3.3.1 Standard curves: Prepare mixed standard solution, whose concentrations are 0.00 µg/mL, 0.01 µg/mL, 0.05 µg/mL, 0.10 µg/mL, 0.20 µg/mL, 0.50 µg/mL, 1.00 µg/mL. Establishing the standard curves by peak areas on GC responses against the concentrations, and then calculating the correlation coefficients.

7.3.3.2 Use the standard working solutions based on estimating the concentration of analyses in the sample. The responses of analyses in the standard working solution and sample solutions 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 operation condition, the chromatograms of bromopropylate is in Figure A.1 in Annex A.

7.3.4 GC-MS determination

Determination the standard working solutions and sample solutions based on condition on above. If the retention time of the analyses are the same to that of the external standards when subtraction the background, in the same time the ion relative abundance of the compounds from samples are similar to that of the standards' at the deviation as Table 2, it can be inferred that is positive sample. The TIC and SCAN chromatograms of bromopropylate is in Annex B.

Table 2—Maximum permitted tolerances for relative ion intensities while confirmation

Relative intensity	>50%	>20% to 50%	>10% to 20%	≤10%
Permitted tolerances	± 10%	± 15%	± 20%	± 50%

7.4 Blank test

The operation of the blank test is the same as described in the method of determination, but with the

omission of sample addition.

8 Calculation and expression of result

Calculate the content of bromopropylate residue in the test sample according to the formula (1). The blank value should be subtracted from the result of calculation above.

$$X = \frac{c \times 1\,000}{m \times 1\,000} \times V \quad \dots\dots\dots(1)$$

where

X is the residue content of bromopropylate in the test sample, mg/kg;

c is the concentration of bromopropylate in the standard working solution, $\mu\text{g/mL}$;

V is the final volume of the sample solution, mL;

m is the corresponding mass of test sample in the final sample solution, g.

9 Limit of quantitation(LOQ) and recovery

9.1 Limit of quantitation

The limit of quantitation (LOQ) of both GC and GC-MS method is 0.01 mg/kg.

9.2 Recovery

Listed in Table 3 and Table 4.

Table 3—Recovery range of bromopropylate in fruits by GC method

Spike level /(mg/kg)	Recovery range/%						
	Apple	Lemon	Pineapple	Grape	Cherry	Strawberry	Watermelon
0.01	82.1~106	82.1~105	80.2~104	81.2~108	80.6~103	82.5~108	86.1~108
0.50	91.2~101	89.0~100	89.8~101	89.4~106	87.2~99.4	92.4~107	88.8~105
2.00	92.0~105	90.5~101	88.5~98.5	91.0~103	85.5~97.0	87.0~101	92.0~104
5.00	91.4~100	92.2~101	93.0~101	91.6~101	90.8~100	92.4~100	91.8~101

Table 4—Recovery range of bromopropylate in fruits by GC-MS method

Spike level /(mg/kg)	Recovery range/%						
	Apple	Lemon	Pineapple	Grape	Cherry	Strawberry	Watermelon
0.01	83.2~103	82.3~113	80.2~96.3	81.3~103	81.8~104	82.5~113	82.1~106
0.50	89.4~103	93.4~105	93.2~108	90.2~103	94.6~110	93.6~105	95.4~104
2.00	87.0~101	89.5~102	87.0~97.5	89.0~101	92.0~104	86.0~98.0	86.5~96.5
5.00	90.4~100	92.6~102	91.6~101	96.6~106	96.4~105	92.6~107	96.4~107

Annex A
(informative)

GC chromatogram of bromopropylate standard

GC chromatogram of bromopropylate standard see Figure A.1.

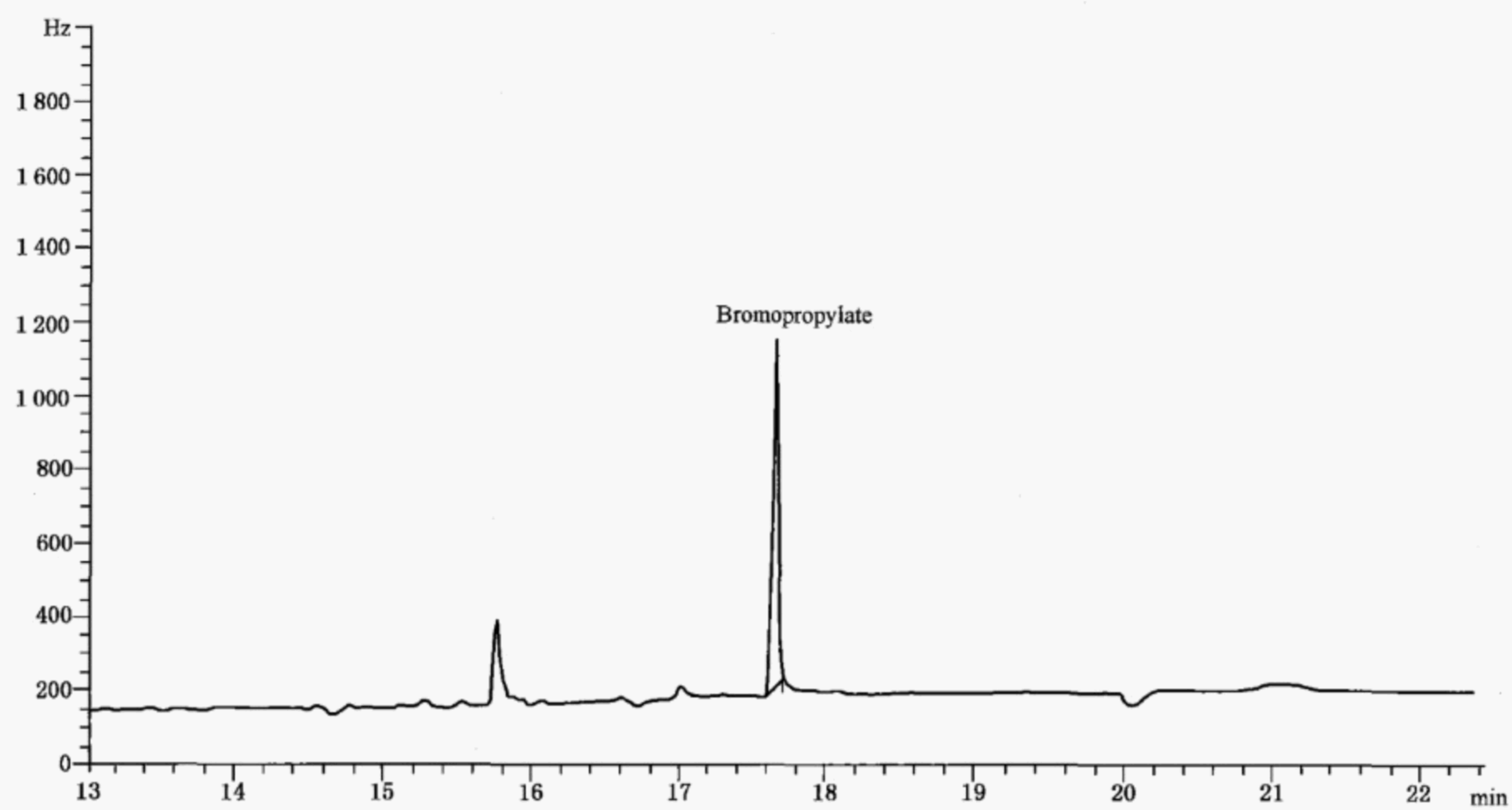


Figure A.1—GC chromatogram of bromopropylate standard(0.01 $\mu\text{g/mL}$)

Annex B
(informative)

TIC and SCAN chromatograms of bromopropylate

TIC and SCAN chromatograms of bromopropylate see Figure B.1 and Figure B.2.

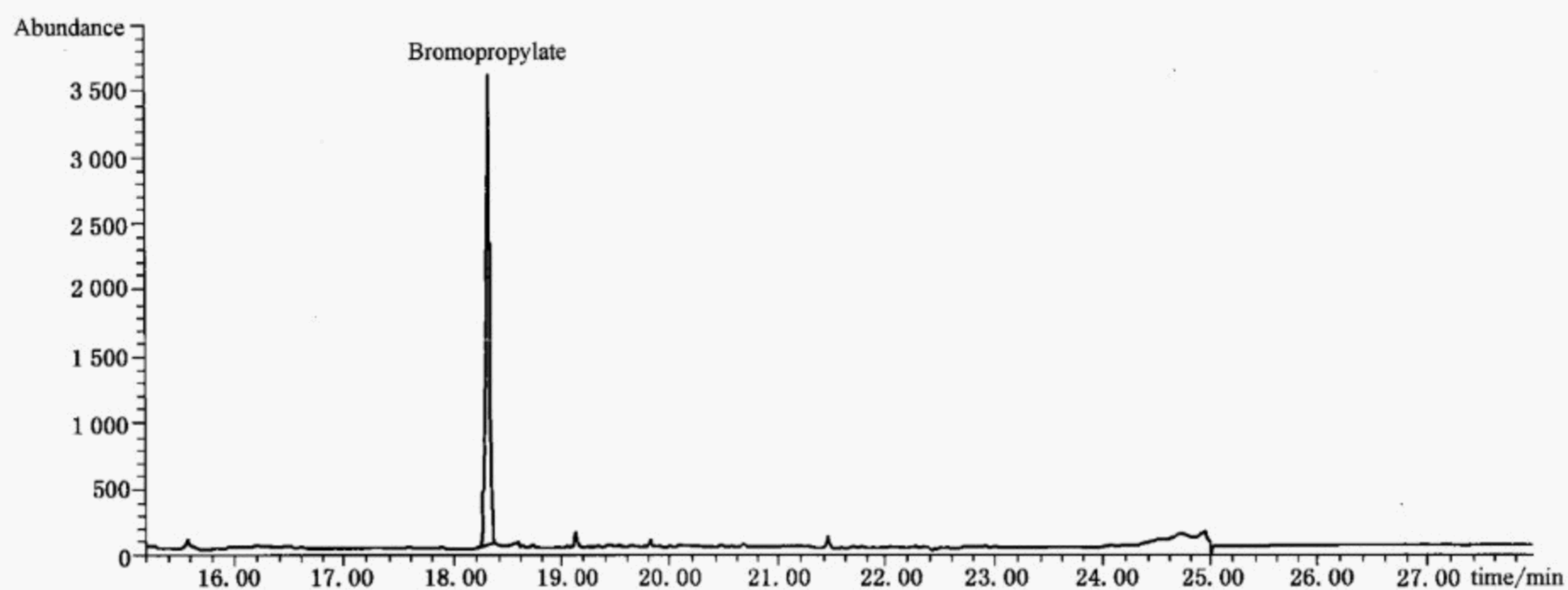


Figure B.1—TIC of bromopropylate standard on SIM mode (0.01 $\mu\text{g/mL}$)

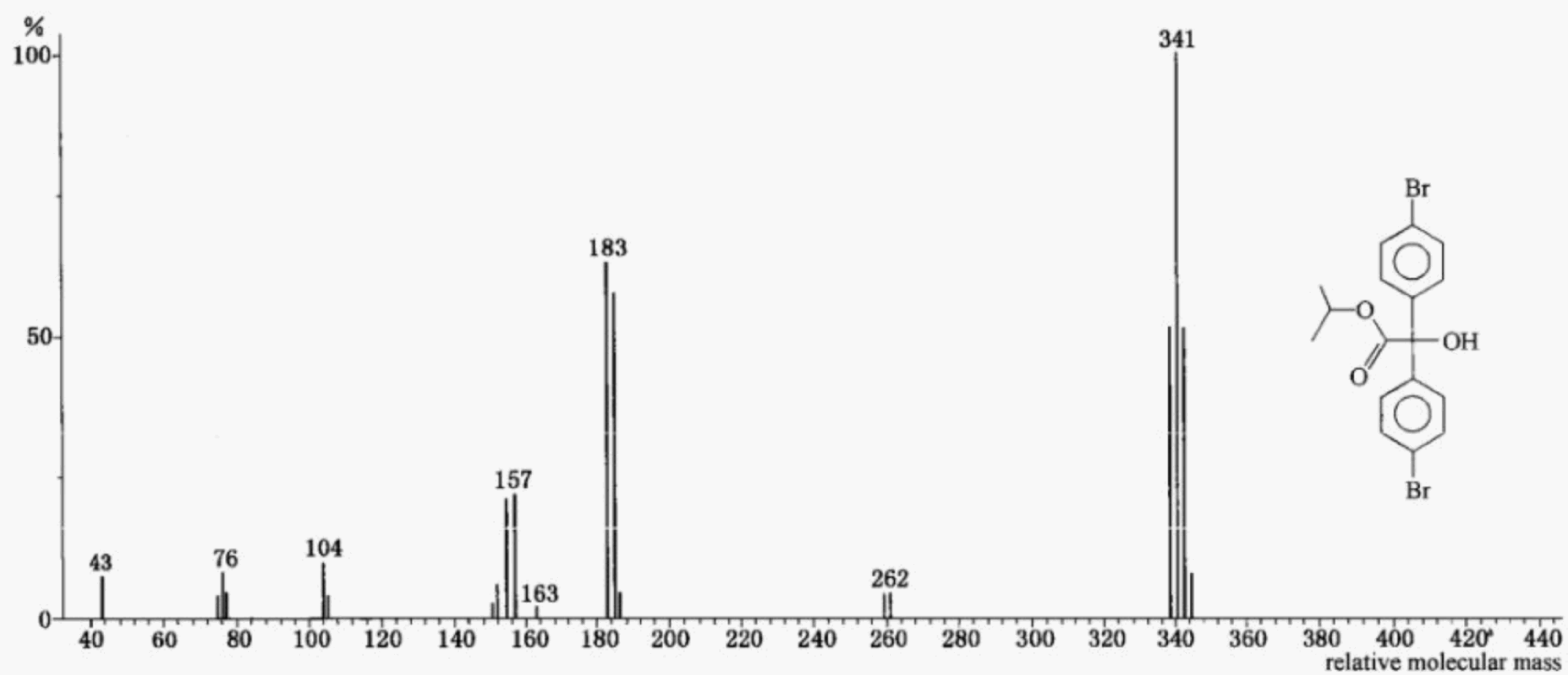


Figure B.2—SCAN chromatogram of bromopropylate

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