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**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SN/T 5220-2019

**Determination of 3-chloropropanol esters and glycidyl esters in
exported foods by gas chromatography-mass spectrometry**

出口食品中3-氯丙醇酯及缩水甘油酯的测定 气相色谱-质谱法

(English Translation)

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Foreword

This standard is compiled in accordance with the rules given in GB/T 1.1-2009.

1 Scope

This standard specifies the gas chromatography-mass spectrometry method for the determination of 3-chloropropanol esters and glycidyl esters in food for export.

This standard is applicable to the qualitative inspection of 3-chloropropanol esters and glycidyl esters in edible oil, milk powder, fried puffed foods and baked foods Measurement and quantitative detection.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version is applicable to this file. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6682 Analyze laboratory water specifications and test methods.

3 Principles of the method

This standard uses hydrolysis reaction to remove all the 3-chloropropanol esters (3-MCPDE) and glycidyl esters (GE) in foods.

Converted into 3-chloropropanol (3-MCPD), after derivatization with phenylboronic acid (PBA), its content is determined by gas chromatography-mass spectrometry, and it represents the total amount of 3-MCPDE.

The method is to add sodium chloride-glacial acetic acid solution (a terminating reaction solution containing chlorine to provide chlorine element to shrink the end of the hydrolysis reaction).

Water glycerol is completely converted into 3-MCPD) and sodium sulfate-glacial acetic acid solution (chlorine-free terminating reaction solution, can not convert glycidol into 3-MCPD) Carry out a control experiment. After derivatization, the total amount of 3-MCPDE and GE and the actual content of 3-MCPDE are measured.

Subtract to get the content of GE.

4 Reagents and materials

Unless otherwise stated, all reagents used are of analytical grade. The water adopts the first grade water specified in GB/T 6682.

4.1

Methanol. chromatographically pure.

4.2

Glacial acetic acid. chromatographically pure.

4.3

Sodium methoxide.

4.4

Anhydrous sodium sulfate.

4.5

Sodium chloride.

4.6

Acetone. chromatographically pure.

4.7

Phenylboronic acid.

4.8

Tert-butyl methyl ether. chromatographically pure.

4.9

Ethyl acetate. chromatographically pure.

4.10

n-hexane. chromatographically pure.

4.11

Isooctane. chromatographically pure.

4.12 0.5 mol/L sodium methoxide-methanol solution. Weigh 2.701g of solid sodium methoxide (4.3) into a 100 mL volumetric flask, and use methanol (4.1) Dilute the volume to 100 mL and mix well.

4.13 10% sodium sulfate-acetic acid solution. Weigh 10 g of anhydrous sodium sulfate solid (4.4), dissolve it in 100 mL of water, and add to the solution 4 mL glacial acetic acid (4.2).

4.14 20% sodium chloride-acetic acid solution. Weigh 20 g of sodium chloride solid (4.5), dissolve it in 100 mL of water, and then add 4 mL to the solution Glacial acetic acid (4.2).

4.15

Acetone-aqueous solution (19.1, V/V). Measure 190 mL of acetone (4.6) and 10 mL of water, and mix well.

4.16

Phenylboronic acid (PBA) solution. Weigh 2.5 g of phenylboronic acid (4.7) and dissolve in 20 ml of acetone-water (19.1, V/V) (4.15), and mix well.

4.17

tert-butyl methyl ether-ethyl acetate solution (8.2, V/V). measure 80 mL tert-butyl methyl ether (4.8) and 20 mL ethyl acetate (4.9), Mix well.

4.18

Standard material. Palmitic acid-3-chloropropanediol ester (3-MCPDE-Palmitate, CAS number 51930-97-3, molecular weight 587.36), and Glycidyl Palmitate (CAS No. 7501-44-2, molecular weight 312.49), the purity is greater than 98%.

4.19

Internal standard. palmitic acid-d5-3-chloropropanediol ester (d5-3-MCPDE-Palmitate, CAS number 1185057-55-9, molecular weight 592.39), the purity is greater than 98%.

4.20

Standard stock solution. accurately weigh 25.0 mg of standard substances (accurate to 0.001g), and use tert-butyl methyl ether-ethyl acetate solution (4.17) After dissolving, dilute to the 50.0 mL mark, prepare a standard stock solution with a concentration of 500 mg/L, and store it in a refrigerator at 0 °C ~ 4 °C.

The validity period is 12 months.

4.21

Mixed standard stock solution. accurately draw 10.6 mL palmitate-3-chloropropanediol and 5.65 mL glycidyl palmitate respectively Standard stock solution (4.20) in a 50 mL volumetric

flask, dilute to the mark with tert-butyl methyl ether-ethyl acetate solution (4.17), and make a concentrated A mixed standard stock solution with a concentration of 20.0 mg/L (calculated as 3-chloropropanol), stored in a refrigerator at 0 °C ~ 4 °C, valid for 6 months.

4.22

Internal standard solution. accurately weigh 1.00 mg of internal standard substance (accurate to 0.001g), and use tert-butyl methyl ether-ethyl acetate solution (4.17) After dissolving, dilute to the 50.0 mL mark and prepare an internal standard solution with a concentration of 20.0 mg/L. Store in a refrigerator at 0 °C ~ 4 °C. The validity period is 12 Months.

4.23

Microporous membrane. 0.22 µm, organic phase type.

5 Apparatus and equipment

5.1

Gas chromatography-mass spectrometry (GC-MS) with electron bombardment source (EI source).

5.2

Vortex mixer.

5.3

Ultrasonic water bath.

5.4

Refrigerated centrifuge. 15°C, 10 000 r/min.

5.4

Nitrogen blowing concentrator.

5.5

Water bath constant temperature oscillator.

5.6

Analytical balance. Sensitivity is 0.001 g.

6.1.1.1 Milk powder sample

Weigh 2 g (accurate to 0.001 g) sample into a 50 mL stoppered PTFE centrifuge tube (each sample needs two copies for comparison), Add 2.0 mL of deionized water, accurately add 50 µL of 20.0 mg/L internal standard solution (4.22), and then add 5 mL of n-hexane (4.10), Ultrasonic extraction for 15 minutes, centrifugation at not less than 10,000 r/min for 10