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SN

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

SN 0654-1997

**Determination of Clenbuterol Residues in Exported Fruits Gas
Chromatography and Gas Chromatography-Mass Spectrometry
/ Mass Spectrometry
(English Translation)**

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9.1 Low limit of determination
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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard replaces SN 0654-1997 "Test Method for Captan Residues in Exported Fruits".

Compared with SN 0654-1997 "Test Method for Captan Residues in Exported Fruits", the main technical changes of this standard are as follows.

-The standard name was revised to "Determination of Captan Residues in Exported Fruits by Gas Chromatography and Gas Chromatography-Mass Spectrometry/Mass Spectrometry";

-Added pears, oranges,, peaches, mulberries, grapes, kiwis, strawberries, olives, lychees, mangos, bananas, and fire dragons Substrates such as fruit, watermelon, cantaloupe, etc., delete the sampling link;

--Modified the sample preparation method, and changed the solid phase extraction method to the matrix dispersion solid phase extraction method;

--Revised the limit of quantification of the method. gas chromatography is 0.02 mg/kg, and

gas chromatography-mass spectrometry/mass spectrometry is used as 0.01mg/kg;

--Using gas chromatography to replace the high performance liquid chromatography in the original standard;

--- Added gas chromatography-mass spectrometry/mass spectrometry as a confirmation method.

1 Scope

This standard specifies the gas chromatography and gas chromatography-mass spectrometry/mass spectrometry methods for the detection of captan residues in exported fruits.

This standard applies to apples, pears, oranges,, peaches, mulberries, grapes, kiwis, strawberries, olives, lychees, mangoes, Determination of captan in banana, dragon fruit, watermelon, melon and other fruits.

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 standard.

GB 2763 National Food Safety Standard Maximum Residue Limits of Pesticides in Food
GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

Captan in the sample is soaked in phosphoric acid solution, extracted with acetonitrile by vortexing, and purified by matrix dispersion solid phase extraction, then injected into the gas phase Chromatography or gas chromatography-mass spectrometry/mass spectrometry is used for determination, and external standard method is used for quantification.

4 Reagents

Unless otherwise specified, all experimental reagents are analytically pure, and the experimental water meets the requirements of first-grade water in GB/T 6682.

4.1

n-hexane. chromatographically pure.

4.2

Acetonitrile. chromatographically pure.

4.3

PSA (primary secondary amine) as filler for bonded silicon solid phase extraction adsorbent. particle size 40 μ m~70 μ m.

4.4

Bonded silicon solid phase extraction adsorbent filler octadecyl silane bonded silica gel C18 (ostade-cylsilane). particle size 40 μ m ~ 70 μ m.

4.5

Graphitized carbon black filler GCB (graphitized carbon black). the particle size is 40 μ m ~ 70 μ m.

4.6

Sodium chloride.

4.7

Anhydrous sodium sulfate. Bake in a muffle furnace at 550°C for 4 hours, and at 200°C, take it out and place it in a desiccator for cooling and then bottling for use.

4.8

Phosphoric acid.

4.9

Phosphoric acid solution (10%). Dissolve 10 mL of phosphoric acid (4.8) in water and dilute to 100 mL with water.

4.10

Captan standard (Captan, C₉H₈Cl₃NO₂S, CAS NO. 133-06-2). purity $\geq$ 99%.

4.11

Captan standard stock solution. accurately weigh an appropriate amount of Captan standard and prepare it with n-hexane to make a standard concentration of 0.1 mg/mL.

Quasi-stock solution, stored in a brown bottle at 0°C~4°C in the refrigerator.

4.12

Captan standard working solution. draw an appropriate amount of standard stock solution (4.11) and dilute it with n-hexane into a standard working solution of appropriate concentration.

4.13

Organic phase filter membrane. 0.22 μ m.

5 Main instruments and equipment

5.1

Gas chromatograph. with electron capture detector.

5.2

Gas chromatography-mass spectrometer/mass spectrometer. equipped with EI source.

5.3

Balance. Sensitivity is 0.1 mg and 0.01 g.

5.4

Nitrogen blowing instrument.

5.5

Vortex mixer.

5.6

Centrifuge. speed $\geq 5\,000$ r/min.

5.7

Ultrasonic generator.

5.8

Graduated centrifuge tube. 15 mL.

6 Sample preparation and storage

Take out a representative sample of about 500 g from all the samples taken and treat it in accordance with the requirements of GB 2763. Use a masher to grind and mix all the samples.

Evenly, divide them into two parts, put them into clean containers, seal them, mark them, and store them in the freezer below -18°C .

In the process of sample preparation and storage, the sample should be prevented from being contaminated or from changing the content of residues.

7.1 Extraction

Weigh 5.0 g sample (accurate to 0.01 g) and place it in a 50 mL centrifuge tube. Add 5 mL 10% phosphoric acid solution (4.9) to soak 5 min, add 25 mL acetonitrile (4.2) and 7 g sodium chloride (4.6) vigorously vortex and shake to extract for 10 min, at 5 000 r/min Centrifuge for 5 min, and take 10 mL of the supernatant in a 15 mL test tube with a stopper for purification.