

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

SN/T 5149-2019

**Determination of cabergoline residues in foodstuffs of animal
origin for export LC-MS/MS method**

出口动物源食品中卡麦角林残留量的测定 液相色谱-质谱/质谱法

(English Translation)

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Foreword

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

Please note that some content of this document may involve patents, the publication of this document the agency is not responsible for identifying these patents.

This standard is proposed and managed by the General Administration of Customs of the People's Republic of China.

This standard was drafted. Shijiazhuang Customs, People's Republic of China.

1 Scope

This standard specifies the liquid chromatography-mass spectrometry/mass spectrometry method for cabergoline in foods of animal origin for export.

This standard applies to the detection of cabergoline residues in animal source foods such as chicken, pork, beef, beef fat, beef liver and milk.

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies to this document.

file. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle

Cabergoline in the sample was extracted with acetonitrile, degreased with acetonitrile-saturated n-hexane, purified on a neutral alumina column, and liquid chromatography-mass spectrometry /Determined by mass spectrometer and quantified by internal standard method.

4 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade, and water is the first-grade water specified in GB/T 6682.

4.1

Acetonitrile. HPLC grade.

4.2

n-hexane. HPLC grade.

4.3

Methanol. HPLC grade.

4.4

Formic acid. HPLC grade.

4.5

Anhydrous sodium sulfate.

4.6 0.1% formic acid. exactly 1.0mL of formic acid (4.4) was dissolved in 1L of water.

4.7 0.1% formic acid water-acetonitrile solution (9 + 1, v/v). Measure 90 mL of 0.1% formic acid aqueous solution (4.6) and 10 mL of acetonitrile, and mix.

4.8

Acetonitrile-saturated n-hexane solution. 100 mL of n-hexane was added to 20 mL of acetonitrile, and after shaking sufficiently, the layers were left to stand and the upper layer of n-hexane was taken.

The alkane layer is reserved.

4.9

Cabergoline (CAS. 81409-90-7, molecular formula. C₂₆H₃₇N₅O₂). purity is greater than 99.0%.

4.10

Cabergoline-D5. Purity greater than 99.0%.

4.11

Standard stock solution (100 µg/mL). Weigh accurately 10 mg of cabergoline standard (accurate to 0.1 mg), dissolve with methanol and make up to 100 mL, and prepare a standard stock solution of 100 µg/mL. This solution is stored at -18 ° C, protected from light, and has a validity of 3 months. 4.12 Standard intermediate solution (1µg/mL). Accurately transfer the standard stock solution from 1.0mL (4.11) to a 100mL volumetric flask, make up to volume with methanol, and prepare a concentration of 1µg/mL, below -18 °C Keep away from light, valid for one month. 4.13 Standard working solution. Dilute the standard intermediate solution to the required standard working solution step by step with a constant volume solution as required. The standard working solution requires Active use.

4.14

Standard internal standard stock solution (10 µg/mL). Accurately weigh 1 mg of cabergoline-D5 standard (accurate to 0.1 mg) and dissolve it in methanol.

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Dissolve and dilute to 100 mL, and prepare a standard internal standard stock solution of 10 µg/mL. This solution is stored at -18 ° C, protected from light, and has a validity of 3 months. 4.15 Standard internal standard working solution (100ng/mL). Accurately transfer the standard internal standard intermediate solution from 0.5mL (4.14) to a 50mL volumetric flask.

Make up the volume of methanol to the mark, prepare a concentration of 100ng/mL, and store at -18 ° C in the dark, and the solution is valid for one week.

4.16

Solid-phase extraction column. Neutral alumina column (Alumina-N), 1000 mg, 3 mL.

4.17

Microporous filter membrane. 0.22 µm, organic phase needle filter.

5 Instruments and equipment

5.1

Liquid chromatography-mass spectrometry/mass spectrometer. Power distribution spray ion source (ESI).

5.2

Analytical balance. Sensitivity is 0.01g and 0.1mg, respectively.

5.3

Homogenizer, rotation speed is not less than 1500r/min.

5.4

Centrifuge, the rotation speed is not less than 4,000r/min.

5.5

Vortex mixer.

5.6

Tissue masher.

5.7

Solid-phase extraction device.

5.8

Nitrogen blowing instrument.

6.1 Chicken, pork, beef, beef fat, beef liver and other animal tissues

Take a representative sample of about 500g, mash it with a pulverizer, mix and mix, place it in a clean container, seal and mark, and place the sample in Store at -18 ° C.

6.2 Milk

Take a representative sample of about 500g, put it into a clean container, seal and label it, and store the sample at 0 ° C to 4 ° C.

Note: During the sampling and sample preparation process, the sample should be prevented from being contaminated or changing the content of the target substance.

7.1 Extraction

Accurately weigh 5g (accurate to 0.01g) sample into a 50mL centrifuge tube, accurately add 25μL of internal standard working solution (4.115), and then add 5g of anhydrous sodium sulfate and 25mL of acetonitrile (4.1), 10000r/Homogenize 2 min, centrifuge at 5000 r/min for 10 min, take 6 mL 10 mL of acetonitrile-saturated n-hexane (4.8) was added to the supernatant, vortexed for 2 min, and centrifuged. The upper solution was discarded,