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# Determination of prohibited captan in cosmetics - High performance liquid chromatography

化妆品中禁用物质克霉丹的测定 高效液相色谱法

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**Standardization Administration of the People's Republic of China.**

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# Determination of prohibited captan in cosmetics - High performance liquid chromatography

## 1 Scope

This Standard specifies determination method of high performance liquid chromatography for prohibited captan in cosmetics.

This Standard is applicable to determination of captan in creams, liquids, loose powder cosmetics.

The detection limit of this Standard for captan is 2 mg/kg, and the limit of quantification is 5 mg/kg.

## 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

After the specimen is ultrasonically extracted by solvent and is centrifuged, use high performance liquid chromatography to determine, use external standard method to quantify, use liquid chromatography-mass spectrometry to confirm.

## 4 Reagents and materials

Unless otherwise stated, the reagents used are analytically pure, the water is of grade one specified in GB/T 6682.

**4.1** Acetonitrile: chromatographically pure.

**4.2** Methanol: chromatographically pure.

**4.3** Tetrahydrofuran.

### 6.1.2 Liquid and loose powder samples

Weigh 1g (to the nearest of 0.001g) of sample. Place in a 10mL plugged plastic centrifuge tube (5.6). Shake well. Add into 10mL of methanol (4.2). After performing ultrasonic extraction for 20min, centrifugation at 5000r/min for 15min, the supernatant is filtered through a 0.45μm microporous membrane for determination of high performance liquid chromatography.

## 6.2 Determination conditions

The reference conditions for high performance liquid chromatography are as follows:

a) Chromatographic column: $C_{18}$, 5μm, 250mm × 4.6mm (inner diameter), or the equivalent;

b) Flow rate: 1.0mL/min;

c) Mobile phase: acetonitrile + water = 65 + 35 (volume ratio);

d) Detection wavelength: 206nm;

e) Column temperature: 30°C;

f) Injection volume: 10μL.

## 6.3 Drawing of standard curve

The captan standard working solution (4.7) was determined according to the determination conditions in 6.2 and the concentration from low to high injection. Draw in peak area-concentration. Make standard working curve.

See Figure A.2 in Annex A for a high performance liquid chromatogram of the captan standard solution. The UV absorption spectrum of the captan standard product is shown in Figure A.3 of Annex A.

## 6.4 Determination

Conduct determination for the testing sample according to the determination conditions in 6.2. If the retention time of the chromatographic peak detected is consistent with the standard product, and the chromatogram of the sample after subtracting the background, the UV absorption spectrum of the substance is consistent with the standard product, it can be preliminarily determined that captan exists in the sample. Use external standard method to quantify. The content of captan shall be within the standard curve. If it is outside the linear range, it shall be diluted before analysis. If necessary, positive sample shall be confirmed by liquid chromatography-mass spectrometry. See Annex B for confirmation test.

# Annex B

(informative)

## Test of confirmation

### B.1 Liquid chromatography conditions

The reference conditions for liquid chromatography determination are as follows:

a) Chromatographic column: XBridge $C_{18}$, 3.5μm, 150mm × 2.1mm (inner diameter);

b) Mobile phase: 0.1% formic acid solution + acetonitrile = 10 + 90 (volume ratio);

c) Flow rate: 0.3mL/min;

d) Column temperature: 30°C;

e) Injection volume: 5μL.

### B.2 Mass spectrometry conditions

The reference conditions for mass spectrometry are as follows:

a) Ion source: electrospray ion source;

b) Ionization mode: positive ion mode;

c) Capillary voltage: 3.5kV;

d) Extraction voltage: 3.0V;

e) Ion source temperature: 150°C;

f) Desolvation gas temperature: 500°C;

g) Data collection method: multiple reaction monitoring.

### B.3 Qualitative determination

When the specimen is measured, the sample solution is appropriately diluted, and the sample solution and the standard working solution are determined according to liquid chromatography-mass spectrometry conditions. If the selected ions are present, and the selected ion ratio is consistent with the

**------ The End ------**