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

SN/T 5151-2019

**Determination of seven benzophenones in sunscreen
cosmetics by high performance liquid chromatography**

防晒化妆品中7种二苯酮类物质的测定 高效液相色谱法

(English Translation)

Issued on: October 25, 2019

Implemented on: May 1, 2020

Issued by: General Administration of Customs

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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 elements of this document may involve patents. 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. China Academy of Inspection and Quarantine.

1 Scope

This standard specifies a method for the determination of seven benzophenones in sunscreen cosmetics by high performance liquid chromatography.

This standard applies to the determination of seven benzophenones in lotion and cream sunscreen cosmetics.

2 Normative references

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

The sample was extracted by solvent, filtered by a microporous membrane, and then determined by high performance liquid chromatography, quantified by external standard method, and confirmed by liquid chromatography-mass spectrometry.

4 Reagents and materials

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

4.1

Methanol. chromatographically pure.

4.2

Tetrahydrofuran. chromatographically pure.

4.3

Standards. 7 benzophenone standard standards Chinese name, English name, CAS number, molecular formula, structural formula, relative molecular weight See Table A in Appendix A. 1. Purity is not less than 95%.

4.4

Standard stock solutions of 7 benzophenones. Accurately weigh each 10mg of each benzophenone standard substance (4.3), accurate to 0.1mg, Put them in 10mL volumetric flasks, dissolve with methanol (4.1) and make up to the mark, shake well, prepare standard stock solutions with concentrations of 1,000µg/mL, store at 4 ° C, and can be used for three months.

4.5

Mixed standard stock solutions of 7 benzophenones. Accurately transfer each standard stock solution (4.4) 1mL to a 20mL volumetric flask in methanol, dilute to the mark with methanol (4.1). The concentration of benzophenones in the solution was 50 µg/mL. 4.6 Mixed standard working solution. Precisely transfer the mixed standard stock solution (4.5) of 7 benzophenones and dilute with methanol (4.1) it is a series of working solutions with a concentration of 0.5 µg/mL, 1 µg/mL, 2 µg/mL, 5 µg/mL, 10 µg/mL, 20 µg/mL, etc.

5 Instruments and equipment

5.1

High Performance Liquid Chromatography (HPLC) instrument. equipped with UV detector or diode array detector.

5.2

Liquid chromatography-mass spectrometry/mass spectrometry (LC-MS/MS) instrument. equipped with an electrospray ion source (ESI).

5.3

Analytical balance. Sensitivity is 0.0001g and 0.0001g.

5.4

Centrifuge. The rotation speed is not less than 10,000 rpm.

5.5

Ultrasonic water bath.

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5.6

Vortex mixer.

5.7

Nitrogen blowing instrument.

5.8

Colorimetric tube with stopper. 25mL.

5.9

Plastic centrifuge tube with stopper. 10mL.

5.10

Microporous membrane. 0.45 μm , organic phase.

6.1 Sample treatment

Weigh 0.1g (accurate to 0.01g) sample into a 25mL stopper colorimetric tube (5.8), and add 2mL of tetrahydrofuran to the colorimetric tube.

(4.2), vortex and mix well, add 80% methanol aqueous solution (??) and make up to 10mL, vortex for 1min, and ultrasonically extract 10min. take Part of the extraction solution was transferred to a 10 mL stoppered plastic centrifuge tube (5.9), centrifuged at 10,000 rpm for 10 min, and 1 mL of the supernatant was subjected to nitrogen After blowing to dryness,

redissolve in 1mL of methanol, and measure after passing through a 0.45µm microporous membrane (5.10).

6.2.1 Chromatographic conditions

Because the test results depend on the instrument used, it is not possible to give general parameters for instrumental analysis. Proven using Conditions are more appropriate.

a) Chromatographic column. C18 column, 5µm, 250mm x 4.6mm (inner diameter), or equivalent; b) Mobile phase. A (water solution containing 0.1% formic acid, ?/?), B (containing 0.1 % Formic acid in methanol, ?/?), see gradient elution procedure

Table 1;

c) Flow rate. 1.0mL/min;

d) Column temperature. 25 ° C;

e) Detection wavelength. 290nm;

f) Injection volume. 10 µL.

Table 1 Gradient elution procedure

Time/min A /% B /%

6.2.2 Drawing of standard working curve

According to the measurement conditions of 6.2.1, the concentration of the mixed standard working solution was sequentially measured from low to high, and the peak area of the chromatographic peak was used as the vertical sitting.

The corresponding solution concentration is plotted on the abscissa and a standard working curve is drawn. HPLC chromatograms of seven benzophenones see

Figure B in Appendix B. 1.

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6.2.3 Determination of samples

According to the measurement conditions of 6.2.1, the sample solution to be tested was measured and quantified by the external standard method. Response value of p-benzophenones in the test solution it should be within the linear range of the standard curve. If it exceeds the linear range, it should be diluted and then injected for analysis. If benzophenone is detected during sample measurement the retention time of the chromatographic peaks of the same substance is the same as that of the standard, and it can be initially confirmed that the benzophenone substance is detected in the sample. If necessary, positive Samples need to be confirmed by liquid chromatography-mass