

ICS 67.050

SN

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

SN/T 1588.3-2013

**Rules for the inspection of luminaires for import and export -
Part 3: Particular requirements of luminaries for swimming
pools and similar applications
(English Translation)**

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of China**

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1 Scope

This document specifies the determination method for hexavalent chromium in foods for export.

This document is applicable to the determination of hexavalent chromium in health foods, cereals, vegetables, meat, aquatic products, candy and other foods.

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 Terms and definitions

There are no terms or definitions to be defined in this document.

Method One -- High performance liquid chromatography-inductively coupled plasma mass spectrometry

4 Method summary

The chromate ions (CrO_4^{2-}) in the sample are extracted using dipotassium hydrogen phosphate buffer solution (0.1 mol/L) with a pH of 7.9~8.1. After high-speed centrifugation, it is passed through a 0.45 μm filter membrane for high-performance liquid chromatography-inductively coupled plasma mass spectrometry measurement.

Quantify by external standard method.

5 Reagents and materials

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

5.1

Nitric acid ($\rho=1.42 \text{ g/mL}$): guaranteed reagent.

5.2

Dipotassium hydrogen phosphate.

5.3

Ammonia.

5.4

n-Hexane.

5.5 Dipotassium hydrogen phosphate buffered extraction solution (0.1 mol/L): Dissolve

22.822 g of dipotassium hydrogen phosphate in water. Add nearly 1000 mL of water.

Mix well. Use phosphoric acid solution or potassium hydroxide solution to adjust the pH to pH 7.9 ~8.1. Set the volume to 1000 mL.

5.6 Hexavalent chromium standard solution: 100 mg/L hexavalent chromium standard

solution. It is certified reference material.

5.7 Hexavalent chromium standard stock solution: Dilute the hexavalent chromium

standard stock solution with water step by step to a 100 µg/L solution. Prepare when needed.

5.8 Hexavalent chromium standard curve solution: Pipette 0 mL, 0.125 mL, 0.5 mL, 1.25 mL, 2.5 mL, and 5 mL of standard stock solution (5.7) into 25 mL volumetric flasks. Set the volume up to the mark with water. Mix well. The standards of this standard series are: 0 µg/L, 0.5 µg/L, 2 µg/L, 5 µg/L, 10 µg/L, and 20 µg/L. Prepare when needed.

5.9 Mobile phase (56 mmol/L nitric acid-67 mmol/L ammonia, pH 9.0): Accurately

pipet 2.00 mL of nitric acid and 2.50 mL of ammonia solution into a beaker. Add nearly 500 mL of water. Mix well. Adjust the pH to 9.0 with ammonia or nitric acid solution, and transfer it to a 500 mL volumetric flask. Set the volume to the mark with water.

Conduct ultrasonic degassing for 10 min.

6.1 High performance liquid chromatography-inductively coupled plasma mass spectrometer.

6.2 Ultrasonic generator.

6.3 Vortex mixer.

6.4 Oscillator.

6.5 Refrigerated centrifuge: the rotation speed is greater than 10000 r/min; the minimum temperature is less than 4°C.

6.6 Analytical balance: sensitivity is 1 mg.

7.1 Specimen preparation

Take about 500 g of representative sample from all the samples taken. Grind solid samples to powders. Those with high moisture content are beaten into a paste. Mix well.

Divide into two equal portions. Put them into clean containers separately. Seal them as specimens. Indicate the mark.

For health care capsule samples, discard the capsule shell and remove the entire contents.

7.2 Specimen storage

Specimens with high moisture content shall be frozen and stored below -18°C.

8.1 Sample processing

Weigh 0.5 g of powder sample and 0.5 g~1.0 g (accurate to 0.001 g) of the sample with high moisture content in a 50 mL centrifuge tube. Add dipotassium hydrogen phosphate extraction solution (5.5). Dilute to 25 mL. The sample is vortexed for 2 min, ultrasonic extracted for 20 min, extracted on a shaking table for 30 min, and centrifuged at 10000 r/min for 5 min. Take 4 mL of the supernatant into a 15 mL centrifuge tube. Add 1 mL of n-hexane. Then conduct high-speed centrifugation at 10000 r/min for 5 min. Remove 2 mL of supernatant. Pass through a 0.45 µm aqueous phase filter membrane and wait for measurement on the machine. Complete the measurement within 2 h.

8.2.1 Liquid chromatography reference conditions

Liquid chromatography reference conditions are listed below:

- a) Chromatographic column: anion exchange column, 250 mm x 4.1 mm (inner diameter), 10 µm, or one with equivalent performance.
- b) Mobile phase: 56 mmol/L nitric acid-67 mmol/L ammonia, pH 9.0 (5.9).
- c) Flow rate: 1.0 mL/min.
- d) Injection volume: 50 µL.

10.1 Limit of quantification (LOQ)

The limit of quantitation of this method is 0.05 mg/kg.

10.2 Recovery rate and precision

The recovery and precision results of this method at the added concentration are shown in Table C.1.

Method Two -- Ion chromatography post-column derivatization

11 Method summary

Use dipotassium hydrogen phosphate buffer solution (0.1 mol/L) with pH 7.9~8.1 to extract chromate ions (CrO_4^{2-}) in the specimen. After high-speed centrifugation, the extract is filtered through a 0.45 µm filter. Interfering substances are separated by ion chromatography. Mix with derivatization reagent (diphenylcarbazide). Utilizing the strong oxidizing property of chromate, diphenylcarbazide is oxidized in an acidic environment and