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**GB**

NATIONAL STANDARD OF THE  
PEOPLE'S REPUBLIC OF CHINA

**GB 5009.246-2016**

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**National food safety standard -  
Determination of titanium dioxide in foods**

食品安全国家标准

食品中二氧化钛的测定

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## Table of Contents

|                                                                                              |    |
|----------------------------------------------------------------------------------------------|----|
| Foreword.....                                                                                | 3  |
| 1 Scope.....                                                                                 | 4  |
| Method I – Inductively coupled plasma-atomic emission spectrometry (ICP-AES) .....           | 4  |
| 2 Principle .....                                                                            | 4  |
| 3 Reagents and materials .....                                                               | 4  |
| 4 Apparatus.....                                                                             | 5  |
| 5 Sample preparation.....                                                                    | 5  |
| 5 Analytical procedures.....                                                                 | 6  |
| 6 Expression of analytical results.....                                                      | 7  |
| 7 Precision .....                                                                            | 8  |
| 8 Others .....                                                                               | 8  |
| Method II – Diantipyryl methane colourimetry .....                                           | 8  |
| 9 Principle .....                                                                            | 8  |
| 10 Reagents and materials .....                                                              | 8  |
| 11 Apparatus .....                                                                           | 9  |
| 12 Sample preparation.....                                                                   | 9  |
| 13 Analytical procedures.....                                                                | 10 |
| 14 Expression of analytical results.....                                                     | 11 |
| 15 Precision .....                                                                           | 12 |
| 16 Limit of detection and limit of quantitation .....                                        | 12 |
| Annex A Reference operating conditions for microwave digestion and ICP-AES instruments ..... | 13 |

# **National food safety standard - Determination of titanium dioxide in foods**

## **1 Scope**

This Standard specifies the Inductively coupled plasma-atomic emission spectrometry and diantipyryl methane colourimetry for the determination of titanium dioxide in foods.

This Standard applies to the determination of titanium dioxide in flours, jams, preserved fruits, dehydrated potatoes, deep-fried nuts and seeds, candies (including gam base candies), cocoa products, chocolates and chocolate products (including cocoa butter replacer chocolates and products), and coatings of candies, products of candy and chocolate, decorative candies, toppings, sweet juices, flavored syrups, mayonnaises, salad dressings, solid beverages, jellies, puffed foods, beverage turbidity agents and konjac gel products.

## **Method I – Inductively coupled plasma-atomic emission spectrometry (ICP-AES)**

## **2 Principle**

After acid digestion of sample, use an inductively coupled plasma-atomic emission spectrometer for analysis and use standard curve external standard method for quantitation.

## **3 Reagents and materials**

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

### **3.1 Reagents**

**3.1.1** Perchloric acid ( $\text{HClO}_4$ ): guaranteed reagent.

**3.1.2** Sulfuric acid ( $\text{H}_2\text{SO}_4$ ): guaranteed reagent.

**3.1.3** Nitric acid ( $\text{HNO}_3$ ): guaranteed reagent.

**3.1.4** Ammonium sulfate  $[(\text{NH}_4)_2\text{SO}_4]$ .

### **3.2 Preparation of reagents**

**3.2.1** Sulfuric acid solution (5 + 95): measure 50 mL of sulfuric acid; add slowly into 950 mL of water; mix up.

**3.2.2** Mixed acid [perchloric acid + nitric acid (1 + 9)]: measure 100 mL of perchloric acid; add slowly into 900 mL of nitric acid; mix up.

### **3.3 Standard substance**

Titanium dioxide ( $\text{TiO}_2$ ): primary reagent or spectrographically pure.

### **3.4 Preparation of standard solutions**

**3.4.1** Titanium standard stock solution (1,000  $\mu\text{g/mL}$ ): weigh 0.167 g of titanium dioxide; add 5 g of ammonium sulfate; add 10 mL of sulfuric acid; heat to dissolve; allow to cool; transfer to a volumetric flask of 100 mL; dilute to scale; mix up. Or use a standard solution which has passed the national certification and awarded the certificate of a standard substance.

**3.4.2** Titanium standard using solution (10.0  $\mu\text{g/mL}$ ): absorb 1.00 mL of titanium standard stock solution to pour into a volumetric flask of 100 mL; use sulfuric acid solution (5 + 95) to dilute to scale.

**3.4.3** Titanium standard serial working solutions: absorb 0.000 mL, 0.500 mL, 2.00 mL, 5.00 mL, 10.0 mL and 20.0 mL of titanium standard using solution; pour respectively into a volumetric flask of 100 mL; use sulfuric acid solution (5 + 95) to dilute to scale; prepare the titanium serial standard working solutions of 0.000  $\mu\text{g/mL}$ , 0.050  $\mu\text{g/mL}$ , 0.200  $\mu\text{g/mL}$ , 0.500  $\mu\text{g/mL}$ , 1.00  $\mu\text{g/mL}$  and 2.00  $\mu\text{g/mL}$ .

## **4 Apparatus**

**4.1** Inductively coupled plasma-atomic emission spectrometer.

**4.2** Microwave digestion system.

**4.3** Analytical balance: sensitivity 0.1 mg.

## **5 Sample preparation**

### **5.1 Solid and semisolid sample**

**10.2.1** Mixed solution [perchloric acid + nitric acid (1 + 9)]: measure 100 mL of perchloric acid; slowly add into 900 mL of nitric acid; mix up.

**10.2.2** Hydrochloric acid solution (1 + 1): measure 100 mL of hydrochloric acid; slowly add into 100 mL of water; mix up.

**10.2.3** Hydrochloric acid solution (1 + 23): measure 10 mL of hydrochloric acid; slowly add into 230 mL of water; mix up.

**10.2.4** Sulfuric acid solution (2 + 98): measure 20 mL of sulfuric acid; slowly add into 980 mL of water; mix up.

**10.2.5** Ascorbic acid solution (2%): weigh 2 g of ascorbic acid; use water to dissolve before diluting to 100 mL; prepare immediately prior to use.

**10.2.6** Diantipryl methane solution (5%): weigh 5 g of diantipryl methane; use hydrochloric acid solution (1 + 23) (10.2.3) to dissolve and dilute to 100 mL.

### **10.3 Standard substance**

Titanium dioxide ( $\text{TiO}_2$ ): primary reagent or spectrographically pure.

### **10.4 Preparation of standard solutions**

**10.4.1** Titanium standard stock solution (1,000  $\mu\text{g/mL}$ ): weigh 0.167 g of titanium dioxide; add 5 g of ammonium sulfate; add 10 mL of sulfuric acid; heat to dissolve; allow to cool; transfer to a volumetric flask of 100 mL; dilute to scale; mix up. Or use a standard solution which has passed the national certification and awarded the certificate of a standard substance.

**10.4.2** Titanium standard using solution (10  $\mu\text{g/mL}$ ): absorb 1.00 mL of titanium standard stock solution (10.4.1) to pour into a volumetric flask of 100 mL; use sulfuric acid (2 + 98) to dilute to scale.

## **11 Apparatus**

**11.1** Ultraviolet spectrophotometer.

**11.2** Microwave digestion system.

**11.3** Analytical balance: sensitivity 1 mg.

## **12 Sample preparation**

### **12.1 Solid and semisolid sample**

use water to dilute to scale; shake up; place aside for 40 min. The concentrations of titanium in standard serial working solutions are 0.000 µg/mL, 0.100 µg/mL, 0.200 µg/mL, 0.500 µg/mL, 1.00 µg/mL and 2.00 µg/mL.

### 13.5 Plotting of standard curve

Use the standard blank solution after developing as the reference; use a cuvette of 1 cm; at the wavelength of 420 nm; use the ultraviolet spectrophotometer to determine the absorbance of standard serial working solutions after developing. Plot the standard curve using the concentrations of standard serial working solutions as the abscissa and the corresponding absorbances as the ordinate.

### 13.6 Determination

Under the same experimental conditions as those for the determination of standard solutions, determine the absorbances of sample solution and blank solution after developing. Obtain the concentrations of sample solution and blank solution based on the standard curve (13.5) and the absorbances of blank solution and sample solution.

## 14 Expression of analytical results

The content of titanium dioxide in sample is calculated in accordance with Equation (2):

$$X = \frac{(c - c_0) \times V_1 \times 50 \times 1\,000}{m \times V_2 \times 1\,000} \times 1.668\,1 \dots\dots\dots (2)$$

where:

$X$  – the content of titanium dioxide in sample, in mg/kg;

$c$  – the concentration of titanium in sample solution after developing which is obtained from the standard curve, in µg/mL;

$c_0$  – the concentration of titanium in blank solution after developing which is obtained from the standard curve, in µg/mL;

$V_1$  – the volume made up for the first time after sample digestion, in mL;

50 – the volume made up of sample solution after developing, in mL;

$m$  – the mass of sample, in g;

$V_2$  – the volume of sample solution transferred for developing, in mL;

1.668 1 – 1 g of titanium is equivalent to 1.668 1 g of titanium dioxide.

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Contact: Wayne Zheng, [Sales@ChineseStandard.net](mailto:Sales@ChineseStandard.net)

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