

**SN**

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

**SN/T 1599-2005**

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**Determination for major elements in coal ash by ICP-AES**

煤灰中主要成分的测定 电感耦合等离子体原子发射光谱法

**(English Translation)**

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### 1 Scope

This Standard specifies determination of bisphenol A in polycarbonate resin and its products by gas chromatography-mass spectrometry.

This Standard is applicable to the determination of bisphenol A in polycarbonate resin and its products.

### 2 Method Summary

Acetone was used as the extraction solvent for the specimen. After ultrasonic extraction, derivatization was carried out by pentafluoropropionic anhydride. The solution was blown dry by nitrogen. Use n-hexane to make constant volume; and analyzed by gas chromatography-mass spectrometry.

### 3 Reagents and Materials

#### 3.1

##### Acetone

Chromatographically pure.

#### 3.2

##### n-hexane

chromatographically pure.

### 3.3

#### **Pentafluoropropionic anhydride**

Purity  $\geq 98\%$ .

### 3.4

#### **Bisphenol A standard product**

CAS: 80-05-7, purity  $\geq 99\%$ .

### **3.5 Bisphenol A standard stock solution: accurately weigh 10mg of bisphenol A**

standard (3.4); place it in a 100mL volumetric flask; dilute to the mark by acetone (3.1); and mix well. The concentration of this solution is 100g/L.

### **3.6 Preparation of standard working solution: respectively pipette appropriate volume**

of bisphenol A standard stock solution (3.5); dilute by acetone (3.1) to prepare standard working solution with required concentration.

at 4000 r/min for 15 min in a centrifuge (4.7). Absorb the supernatant by a syringe (4.8);

pass the injecting solution through a 0.20 $\mu$ m organic filter membrane (4.9); and collect the filtrate in a brown bottle. Pipette 1mL of the filtrate to a graduated centrifuge tube accurately; add 100 $\mu$ L of derivatization reagent pentafluoropropionic anhydride (3.3);

place in a vortex mixer (4.10) and vortex to mix; derivatize in a constant temperature water bath (4.11) at 60°C for 30min. After that, place it in a nitrogen blower (4.12) and slowly blow it to near dryness. Add 1mL of n-hexane (3.2); vortex to mix; and transfer the solution to the sample injecting bottle for gas chromatography-mass spectrometry detection.

## **6.3 Preparation of standard working solution**

Accurately transfer a certain volume of standard solution with the appropriate concentration to the graduated centrifuge tube (4.6); add 100 $\mu$ L of the derivatization reagent pentafluoropropionic anhydride (3.3); and follow the 6.2 derivatization procedures below.

**6.4.1 Chromatographic column: 30m x 0.25mm (inner diameter) x 0.25 $\mu$ m (film thickness);** HP-5MS quartz capillary column or equivalent.

**6.4.2 Chromatographic column temperature:** initial temperature is 70°C, maintain for 1min; then raise the temperature to 210°C at 30°C/min; and then raise the temperature to 260°C at 10°C/min, and maintain for 2min.

**6.4.3 Inlet temperature:** 260°C;

- 6.4.4 Mass spectrometer interface temperature: 280°C;
- 6.4.5 Carrier gas: helium, purity  $\geq 99.99\%$ ; flowrate of 1.0mL/min;
- 6.4.6 Sample injecting method: split-less injection; open the valve after 1.0min;
- 6.4.7 Sample injecting quantity: 1 $\mu$ L;
- 6.4.8 Electric ionization method: EI;
- 6.4.9 Electric ionization energy: 70eV;
- 6.4.10 Determination method: selective ion monitoring method;
- 6.4.11 Select monitoring ion (m/z): 265, 505, 520;
- 6.4.12 Solvent delay time: 5min.

## **6.5 Gas chromatography - mass spectrometry determination**

M - specimen mass, in g.

## **8 Lower Limit of Detection**

This method's lower limit of the detection is 0.05 mg/kg.

## **9 Precision**

Determine the reliability of the results (95% confidence level) as follows:

### **9.1 Repeatability**

In the same laboratory, the same person uses the same equipment, the same test method, and the absolute difference between the two parallel determination results of the same test object within the day is no greater than 10% of the arithmetic mean of the two measured values.

### **9.2 Reproducibility**

In different laboratories, the same person uses the same equipment and the same test method. The absolute difference between two parallel measurements of the same object is no greater than 15% of the arithmetic mean of the two measured values.

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