

ICS 67.220.20
CCS C 54;X40



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

GB 26403-2011

National food safety standards of food additives TBHQ

食品添加剂 特丁基对苯二酚

(English Translation)

Issued on: March 15, 2011

Implemented on: May 15, 2011

Issued by: Ministry of Health of the People's Republic of China

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1 Scope

This standard is applicable to the use of hydroquinone as raw material, the alkylation reaction of the food additive t-butyl hydroquinone (referred to as TBHQ).

2.1 Molecular formula

C₁₀H₁₄O₂

2.3 Relative molecular mass

166.22 (according to the 2007 International Relative Atomic Quality)

3 Technical requirements

3.1

sensory requirements. should be consistent with the provisions of Table 1.

Table 1 sensory requirements

The project requires a test method Color white Odor has a special smell Organizational state Crystalline powder Take the appropriate amount of sample placed in a clean, dry white porcelain dish, in the natural light, Observe its color and state, and smell its taste

3.2

Physical and chemical indicators. should be consistent with the provisions of Table 2.

Table 2 Physical and chemical indicators

Item Index Test Method Tert-butyl hydroquinone (calculated as C₁₀H₁₄O₂), w /% ≥ 99.0 Appendix A, A.3 Tert-butyl p-benzoquinone, w /% ≤ 0.2 Appendix A, A.3 2,5-di-tert-butyl hydroquinone, w /% ≤ 0.2 A.3 in Appendix A Hydroquinone, w /% ≤ 0.1 Appendix A, A.3 Toluene/(mg/kg) ≤ 25 Appendix A A.4 Lead (Pb)/(mg/kg) ≤ 2 GB 5009.12 Melting

point/°C 126.5 ~ 128.5 GB/T 617 Appendix A Testing method A.1 General provisions the reagents and water used in this standard, when not specified in other requirements, refer to the analysis of pure reagents and GB/T 6682-2008 in the provisions of the three Water level. Standard titration solution used in the test, the standard solution for the determination of impurities, preparations and products, without any other requirements, GB/T 601, GB/T 602, GB/T 603. The solution used in the test refers to water when it is not specified with the formulation of the solvent Solution.

A.2 Identification test 4 mg to 8 mg of the sample was dissolved in 1 mL of methanol, and then a few drops of 25% aqueous dimethylamine solution was added and the solution changed from light red to red Color without being restored.

A.3 Determination of tert-butyl hydroquinone, p-butyl p-benzoquinone, 2,5-dibutyl hydroquinone and hydroquinone A.3.1 Reagents and materials A.3.1.1 Acetone.

A.3.1.2 Hydroquinone standards. known purity.

A.3.1.3 Tertin hydroquinone standard. Purity $\geq 99\%$.

A.3.1.4 tert-butyl p-benzoquinone standard. purity $\geq 99\%$.

A.3.1.5 2,5-di-tert-butyl hydroquinone standard. purity $\geq 99\%$.

A.3.2 Instruments and equipment Gas chromatograph. equipped with hydrogen flame ionization detector and automatic integrator.

A.3.3 Reference chromatographic conditions A.3.3.1 Column. HP-5 Elastic Quartz Capillary Column with 30m in length, 0.32mm in inner diameter, 0.25 μ m in coating thickness, or others the efficiency of the column.

A.3.3.2 Air flow rate. The carrier gas is high purity nitrogen and the wire speed is 30cm/s.

A.3.3.3 Temperature. column temperature 220 °C, inlet 250 °C, detector 300 °C.

A.3.3.4 Split ratio. 20. 1.

A.3.3.5 Injection volume. 1 μ L.

A.3.4 Analysis steps A.3.4.1 Preparation of standard solutions Respectively, weighing 10mg hydroquinone, tert-butyl hydroquinone, p-butyl p-benzoquinone and 2,5-di-tert-butyl hydroquinone standard, dissolved with acetone, Were transferred to 10mL volumetric flask, dilute the volume to the scale, shake.

A.3.4.2 Preparation of sample solution Weigh 0.2g sample, dissolved with acetone, transferred to 10mL volumetric flask, dilute the volume to the scale, shake.

A.3.4.3 Determination Under the conditions of A.3.3 reference chromatography, the standard solution was analyzed by gas chromatography to determine the retention time of each standard.

1 µL of sample solution was subjected to chromatographic analysis.

A.3.5 Calculation of results the contents of t-butyl hydroquinone, p-butyl-p-benzoquinone, 2,5-dibutyl hydroquinone and hydroquinone were calculated by area normalization.

The results show that the relative deviation of the results of the determination of tert-butyl hydroquinone is less than 0.2% the relative deviation of other substances is less than 2%.

A.4 Determination of Toluene A.4.1 Reagents and materials A.4.1.1 Octanol.

A.4.1.2 Toluene.

A.4.2 Instruments and equipment Gas chromatograph. equipped with hydrogen flame ionization detector.

A.4.3 Reference chromatographic conditions A.4.3.1 Column. packed column made of stainless steel, 3.66m in length, 3.18mm in outer diameter, silicone in the form of SE-30, Diatoport S (DiatoportS) (180 µm to 250 µm), by weight, is 10. 100; or other equivalent color Column.

A.4.3.2 Carrier gas is nitrogen, air flow rate. 25mL/min.

A.4.3.3 Temperature. column temperature 70 °C, inlet 275 °C, detector 300 °C.

A.4.3.4 Split ratio. 50. 1.

A.4.3.5 The injection volume is 1 µL.

A.4.4 Analysis steps A.4.4.1 Preparation of sample solution Weigh about 2g sample, accurate to 0.0002g, transferred to 10mL volumetric flask, dissolved with octanol, and diluted volume to the volume, shake Uniform, accurate concentration of cS.

A.4.4.2 Preparation of standard solutions Prepare an octanol solution containing 50 µg of toluene per milliliter, with an exact concentration of cR.

A.4.4.3 Determination the standard solution and sample solution were chromatographed under A.4.3 reference chromatography. Measured on the chromatogram of the standard solution the peak height (HR) of toluene and the other peaks did not affect the analysis. Likewise, the peak height (HS) of toluene was measured on the chromatogram of the sample solution.

A.4.5 Calculation of results Toluene content in toluene by the mass fraction w, the value in milligrams per kilogram (mg/kg), according to formula (A.1).

$w = (HS/HR) \times (cR/cS)$ (A.1) where.

HS --- the value of the toluene peak in the chromatogram of the sample liquid;

HR --- standard solution chromatogram toluene high peak value;

cR --- the concentration of toluene in the standard solution, in micrograms per milliliter

($\mu\text{g/mL}$);

cS - the concentration of the sample solution in grams per milliliter (g/mL).

The experimental results are based on the arithmetic mean of the parallel measurement results, and the relative deviation of the parallel measurement results is less than 10%.